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14. ABSTRACT

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Final Report for Period Beginning 01-May-2018 and Ending 01-May-2022

Title: W911NF-17-S-0002_LCE-LM Composites for Soft-Matter Embodied Intelligence

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STEM Participants: 5

Major Goals: The goal of this project is to introduce a class of soft elastomer composites that demonstrate “embodied intelligence” by sensing, processing signals, and dynamically responding to external conditions in an autonomous manner. In contrast to current methods that utilize distributed motors and electronics, this work is focused on achieving autonomy through intrinsic material functionality. Rather than rely on microelectronic or mechatronic hardware, the material system that we are developing achieves sensing, signal processing, and actuation through internal electro-thermo-mechanical coupling of the constituent material phases.

To achieve embodied intelligence, we are developing soft material architectures composed of liquid crystal elastomers (LCEs) and liquid metal (LM) alloy. One embodiment of this is a percolating network of microscale LM droplets suspended within an LCE matrix. These LCE-LM composites exhibit a unique combination of electrical, thermal, and dynamic properties that have never before been demonstrated in other soft material systems. We are also exploring a second embodiment in which strips of LCE are coated with a thin film of biphasic LM-silver electrodes. Heating causes the LCE-LM-Ag strips to form helical coils that entangle and create semisolid networks. For both of these material systems, reversible shape change and actuation is achieved through the application of either thermal or electrical stimulation. Moreover, the materials are capable of sensing deformation or damage through changes in electrical resistivity of embedded conductive pathways. In this way, active response is accomplished through electro-thermo-mechanical coupling that arises from the interplay of thermally-induced LCE phase change, flow of electrical current, and changes to the shape or structural connectivity of the material.

Our research efforts have been focused on the following tasks, with the ultimate goal of enabling a new approach to multi-input multi-output (MIMO) “algorithmic” materials that dynamically respond to external cues:

[Task 1] Synthesis of LCE+LM Material Systems: Creation of LCE-LM composites, LCEs with LM microfluidics, and LM-coated LCE microstrips in which actuation and sensing is directly encoded in the composite microstructure. This effort includes liquid crystal monomer systems are formulated that can simultaneously disperse liquid metal droplets, align on the molecular scale, and polymerize into electrically or thermally responsive LCEs.

[Task 2] Algorithmic Materials Design & Testing: Design and characterization of LCE+LM-based material architectures that can detect mechanical/thermal loading or damage and autonomously reconfigure themselves to control electrically and thermally conductive pathways for embedded sensing, circuitry, LCE actuation. This task also involves development of methods for direct-write “4D” printed materials for more complex LCE-LM structures. Such structure has the potential to allow for next-generation systems capable of programmable sensing, signal processing, and dynamical response.

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These research tasks have resulted in a framework for engineering soft materials capable of intrinsic sensing, actuation, and processing. Such “algorithmic materials” can be programmed for a variety of functions and autonomously adapt their shape or internal material properties in response to damage or changes in external conditions or functional demands.

Accomplishments: In this project we achieved the following key accomplishments: (i) introduction of a novel class of liquid metal (LM) embedded liquid crystal elastomer (LCE) composites capable of coupled actuation and sensing; (ii) development of a new processing approach to create “4D printed actuators” composed of a 3D printed LM-LCE composites; (iii) demonstration of embodied intelligence with coupled actuation and energy harvesting with a novel material architecture composed of LCE films coating a soft thermoelectric interface; (iv) introduction and continued development of LM-coated LCE capable of reversible, temperature controlled helical coiling and mechanical connectivity. The key results from each effort are summarized in the subsections below.

LM-LCE Composites – Intelligent soft materials composed of liquid metal microdroplets embedded in a shape memory LCE matrix represents a key breakthrough of this project. These LM-LCE composites were discovered early in the project and much of the first two years was dedicated to (i) characterizing its mechanical, shape memory, electrical, and thermal properties; (ii) controlling the formulation and synthesis method to tailor the properties of the composite; (iii) exploring various applications in engineered soft systems and use cases for soft robotics, including demonstrations of embodied intelligence. These project outcomes are reported in the following journal publications:

Ford et al. PNAS (2019) – Introduces the LM-LCE composite architecture and presents its properties and operation for various soft engineering applications

Ford et al. Soft Matter (2020) – Examines influence of synthesis methods on shape memory properties of LM-LCE composite

Ford et al. Advanced Materials (2020) – Presents further developments in the synthesis method to expand the variety of polymer matrix materials that can be used for LM-based multifunctional composites.

Kent et al. Multifunctional Materials (2020) – Alternative material architecture in which LM is incorporated into LCE in the form of laser-patterned microfluidic channels.

Key demonstrations of “embodied intelligence” with these material systems is presented in Figures 1 & 2. Fig. 1 shows that the LM-LCE composite can sense and respond to damage through the interplay of mechanical forces, LCE contraction, and the activation of internal conducting pathways (Ford 2019). Conductive traces used to power an LED were induced in the composite. Mechanical damage can cause an electrical short around the LED, and the electrical current is rerouted through the composite. Referring to the figure, the rerouted current initiates Joule-heated actuation of the LCE and cause the composite to contract. Fig. 2 presents another approach to embodied intelligence, in which self-regulated actuation can be achieved by coupling changes in the electrical resistance of embedded liquid metal channels with the voltage-controlled deformation of the shape memory actuator (Kent 2020). The interplay of LCE phase change, shape memory induced deformation, electrical resistance, and Joule heating represents an opportunity to explore new concepts in material embodied intelligence

4D printed LM-LCE Composites – Building on the efforts described above, we succeeded in demonstrating 3D printing of LM-LCE composites. These 4D printed structures (the fourth dimension relates to actuation and shape reconfiguration over time) are described in more detail in Ambulo et al. ACS Applied Materials & Interfaces (2020).

In addition to introducing the additive manufacturing technique and performing materials characterization, we demonstrated how the multifunctionality of these shape memory systems can be used for embodied intelligence. Referring to Fig. 3, we fabricated a light-responsive actuator capable of reporting its state by coprinting near infrared (NIR)-responsive and electrically conductive LM-LCE composites. The LM-LCE was configured into a simple circuit that controls an indicator LED (Fig. 3A). When NIR light is turned off, the sample remains static, provides a closed circuit, and lights up the indicator LED. Upon irradiation and activation through NIR light, the LM-LCE bends, thus breaking the electrical connection and turning off the indicator LED (Fig. 3B). When the NIR light stimulus is removed, the LM-LCE switch reverts back to its original shape and reforms the electrical connection turning the LED on. This feedback control can be utilized to allow for real-time monitoring of the composite.

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LCE Actuators with Integrated Thermoelectrics – We also explored the incorporation of thermoelectric materials in order to allow for better control of LCE temperature and shape memory actuation. The layout of the device is shown in Fig. 4, which shows how two films of LCE are coated on the top and bottom surface of a soft thermoelectric array composed of Bi₂Te₃ chips embedded in a 3D printed elastomer and wired together using eutectic gallium-indium alloy. Details of the LCE-thermoelectric architecture are reported in the Zadan et al. *Advanced Materials* (2022).

The thermoelectric layer serves two purposes – it can either be used for energy harvesting (i.e. convert residual heat in the LCE into electricity through the Seebeck effect) or for actuation (i.e. control shape memory actuation by actively heating and cooling the LCE through the Peltier effect). Moreover, energy harvesting and actuation can be combined to achieve embodied intelligence by which the LCE-thermoelectric system will autonomously move toward a thermal energy source and harvest even more energy. This effect, which loosely resembles phototropism and heliotracking shape memory polymers is demonstrated in Fig. 5. By placing the device vertically near a heat source, we see the soft transducer autonomously sense and bend toward the heat source. Bending decreases the distance between the thermoelectric layer and the heat source, which can cause an increase in the amount of electricity that is generated. When the heat source is turned off, the voltage and angle decrease as the device moves away from the heat source and returns to its naturally straight configuration. Comparison tests conducted with and without the LCE coatings show that the added effect of actuation and bending towards the heat source leads to a significant increase in the amount of energy that is harvested.

LM-coated LCE Microstrips – The last component of this project involved studies of LM-coated LCE strips can be induced to aggregate in response to multiple stimuli. Such systems are capable of dramatic, triggered changes in material properties (e.g., form, elastic modulus, electrical conductivity). We explored this by performing studies on how planar ribbons of LM-coated LCE dispersed in a fluid aggregate to form solids on heating and then reversibly disassociate and flow on cooling. Referring to Fig. 6, we coat the LCE strips with silver particles alloyed with liquid metal to allow for both stretchable conductive pathways and a NIR-light triggered photothermal response. By shining NIR light at an intensity of 800 mW/cm², the LCE films aggregate (Figure 6a-e). This development enables the self-healing of aggregate structures through the use of light (Fig. 6c). Furthermore, due to the conductive nature of each individual strip and the mechanical interlocking achieved through aggregation, conductive aggregates are realized (Fig. 6f,g). By allowing the liquid metal coated LCE strips to actuate in a well with leads on opposite ends, they begin to mechanically interlock and form a conductive pathway (Fig. 6h,i). This development may be used to enable multi-stimulus responsive materials with tunable electrical and mechanical properties. A manuscript is anticipated to be submitted in 2022 on this work.

Training Opportunities: This project created training opportunities for the following researchers:

- Dr. Michael Ford (CMU); postdoc leading efforts on LCE-LM synthesis and characterization
- Cedric Ambulo (UT Dallas); PhD researcher assisting in characterization and novel synthesis technique
- Hyun Kim (UT Dallas); PhD researcher developing methods for materials synthesis and patterning
- Teresa Kent (CMU); MS researcher assisting in material synthesis and actuator characterization
- Mason Zadan (CMU); PhD researcher leading efforts on LCE-thermoelectric devices and assisting with LM-coated LCE microstrips
- Mustafa Abdelrahman (Texas A&M); PhD researcher leading efforts on LM-coated LCE microstrips

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Results Dissemination: Results of this project have been reported in the following journal publications:

M Zadan, DK Patel, AP Sabelhaus, J Liao, A Wertz, L Yao, C Majidi, "Liquid Crystal Elastomer with Integrated Soft Thermoelectrics for Shape Memory Actuation and Energy Harvesting," *Advanced Materials* 2200857 (2022).

CP Ambulo, MJ Ford, K Searles, C Majidi, TH Ware, "4D-Printable Liquid Metal-Liquid Crystal Elastomer Composites," *ACS Applied Materials & Interfaces* 13 12805-12813 (2020).

MJ Ford, DK Patel, C Pan, S Bergbreiter, C Majidi, "Controlled Assembly of Liquid Metal Inclusions as a General Approach for Multifunctional Composites," *Advanced Materials* 32 2002929 (2020).

TA Kent, MJ Ford, EJ Markvicka, C Majidi, "Soft actuators using liquid crystal elastomers with encapsulated liquid metal Joule heaters," *Multifunctional Materials* 3 025003 (2020).

MJ Ford, M Palaniswamy, CP Ambulo, TH Ware, C Majidi, "Size of liquid metal particles influences actuation properties of a liquid crystal elastomer composite," *Soft Matter* 16 5878-5885 (2020).

MJ Ford, CP Ambulo, TA Kent, EJ Markvicka, C Pan, J Malen, TH Ware, C Majidi, "A multifunctional shape-morphing elastomer with liquid metal inclusions," *Proceedings of the National Academy of Sciences* 116 21438-21444 (2019).

Honors and Awards: During the course of this project, PI Majidi received tenure and promotion to Full Professor and was awarded the Carnegie Science Award and CMU David P. Casasent Outstanding Research Award

Protocol Activity Status:

Technology Transfer: Invention disclosures and provisional patent applications have been submitted for the original work presented in the following publications:

M Zadan, DK Patel, AP Sabelhaus, J Liao, A Wertz, L Yao, C Majidi, "Liquid Crystal Elastomer with Integrated Soft Thermoelectrics for Shape Memory Actuation and Energy Harvesting," *Advanced Materials* 2200857 (2022).

MJ Ford, CP Ambulo, TA Kent, EJ Markvicka, C Pan, J Malen, TH Ware, C Majidi, "A multifunctional shape-morphing elastomer with liquid metal inclusions," *Proceedings of the National Academy of Sciences* 116 21438-21444 (2019).

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Person Months Worked: 1.00

Project Contribution:

National Academy Member: N

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Project Contribution:

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Funding Support:

Project Contribution:

National Academy Member: N

Participant Type: Graduate Student (research assistant)

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Person Months Worked: 6.00

Funding Support:

Project Contribution:

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Participant Type: Graduate Student (research assistant)

Participant: Mustafa Abdelrahman

Person Months Worked: 12.00

Funding Support:

Project Contribution:

National Academy Member: N

Participant Type: Graduate Student (research assistant)

Participant: Teresa Kent

Person Months Worked: 6.00

Funding Support:

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Volume: 116

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Article Title: A multifunctional shape-morphing elastomer with liquid metal inclusions

Authors: Michael J. Ford, Cedric P. Ambulo, Teresa A. Kent, Eric J. Markvicka, Chengfeng Pan, Jonathan Malen,

Keywords: Soft actuator, soft conductor, liquid crystal elastomer, liquid metal

Abstract: We present a soft actuator composed of fluidic channels of liquid metal alloy embedded in a liquid crystal elastomer (LCE). The LM channels function as stretchable Joule heating elements that deliver heat to the LCE to induce a shape memory phase transition. Because the heater is fluidic, it can deform with the surrounding LCE as the actuator extends and contracts during actuation. In addition to contractile actuation, the LCE can be programmed to perform in-plane or out-of-plane flexural actuation, which exhibit deformations predictable using a simple finite element analysis model. By combining a liquid metal heater with a shape memory polymer, we achieve a soft actuator that does not require an external heat source and can instead be directly activated with electrical current. Finally, we show that the liquid metal channels can also function as a sensor during the actuation cycle, allowing for closed-loop control of the soft actuator.

Distribution Statement: 2-Distribution Limited to U.S. Government agencies only; report contains proprietary info

Acknowledged Federal Support: Y

Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published

Journal: Multifunctional Materials

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Article Title: Soft actuators using liquid crystal elastomers with encapsulated liquid metal joule heaters

Authors: Teresa A Kent, Michael J Ford, Eric J Markvicka, Carmel Majidi

Keywords: Soft actuator, soft sensor, liquid crystal elastomer, liquid metal

Abstract: We present a soft actuator composed of fluidic channels of liquid metal alloy embedded in a liquid crystal elastomer (LCE). The LM channels function as stretchable Joule heating elements that deliver heat to the LCE to induce a shape memory phase transition. Because the heater is fluidic, it can deform with the surrounding LCE as the actuator extends and contracts during actuation. In addition to contractile actuation, the LCE can be programmed to perform in-plane or out-of-plane flexural actuation, which exhibit deformations predictable using a simple finite element analysis model. By combining a liquid metal heater with a shape memory polymer, we achieve a soft actuator that does not require an external heat source and can instead be directly activated with electrical current. Finally, we show that the liquid metal channels can also function as a sensor during the actuation cycle, allowing for closed-loop control of the soft actuator.

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Article Title: 4D-Printable Liquid Metal–Liquid Crystal Elastomer Composites

Authors: Cedric P. Ambulo, Michael J. Ford, Kyle Searles, Carmel Majidi, Taylor H. Ware

Keywords: soft actuator, liquid crystal elastomer, liquid metal, 4D printing, direct ink write printing

Abstract: Soft composites that use droplets of gallium-based liquid metal (LM) as the dispersion phase have the potential for transformative impact in multifunctional material engineering. However, it is unclear whether percolation pathways of LM can support high electrical conductivity in a wide range of matrix materials. This issue is addressed through an approach to LM composite synthesis that focuses on the interrelated effects of matrix curing/solidification and droplet formation. The combined influence of LM concentration, particle size, and sedimentation is explored. By developing this approach, the functionalities that have been demonstrated with LM composites can be generalized to other matrix materials that impart additional functionality. Specifically, composites are synthesized using a biodegradable/reprocessable plastic (polycaprolactone), a hydrogel (poly(vinyl alcohol)), and a processable rubber (a styrene–ethylene–butylene–styrene derivative) to demonstrate wide applicability.

Distribution Statement: 1-Approved for public release; distribution is unlimited.

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Article Title: Controlled Assembly of Liquid Metal Inclusions as a General Approach for Multifunctional Composites

Authors: Michael J. Ford, Dinesh K. Patel, Chengfeng Pan, Sarah Bergbreiter, Carmel Majidi

Keywords: Soft multifunctional materials, soft electronics, liquid metal

Abstract: Soft composites that use droplets of gallium-based liquid metal (LM) as the dispersion phase have the potential for transformative impact in multifunctional material engineering. However, it is unclear whether percolation pathways of LM can support high electrical conductivity in a wide range of matrix materials. This issue is addressed through an approach to LM composite synthesis that focuses on the interrelated effects of matrix curing/solidification and droplet formation. The combined influence of LM concentration, particle size, and sedimentation is explored. By developing this approach, the functionalities that have been demonstrated with LM composites can be generalized to other matrix materials that impart additional functionality. Specifically, composites are synthesized using a biodegradable/reprocessable plastic (polycaprolactone), a hydrogel (poly(vinyl alcohol)), and a processable rubber (a styrene–ethylene–butylene–styrene derivative) to demonstrate wide applicability.

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Partners

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I certify that the information in the report is complete and accurate:

Signature: Carmel Shamim Majidi

Signature Date: 8/29/22 12:50PM

Figures

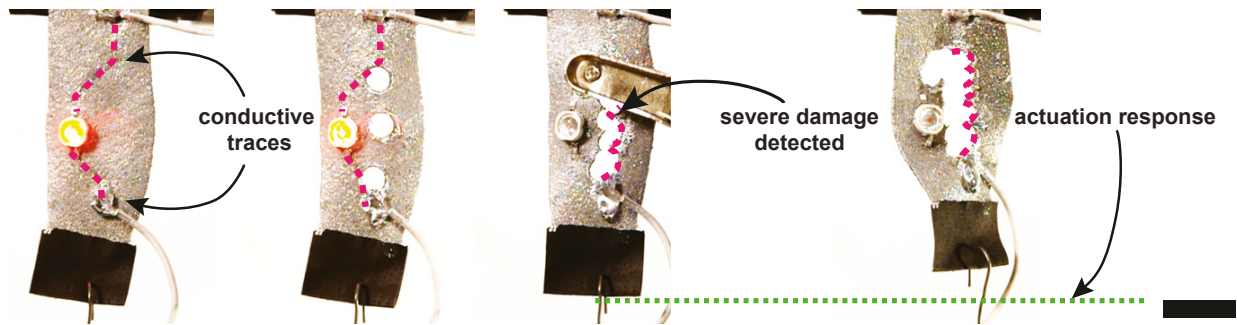


Figure 1 Damage detection and response of an LM-LCE composite. In the first panel, a conductive trace powers an LED. As the sample is damaged (second panel), the LED remains on. After severe damage, Joule heating lifts the weight hanging from the composite (third and fourth panels). (Scale bar, 20 mm.)

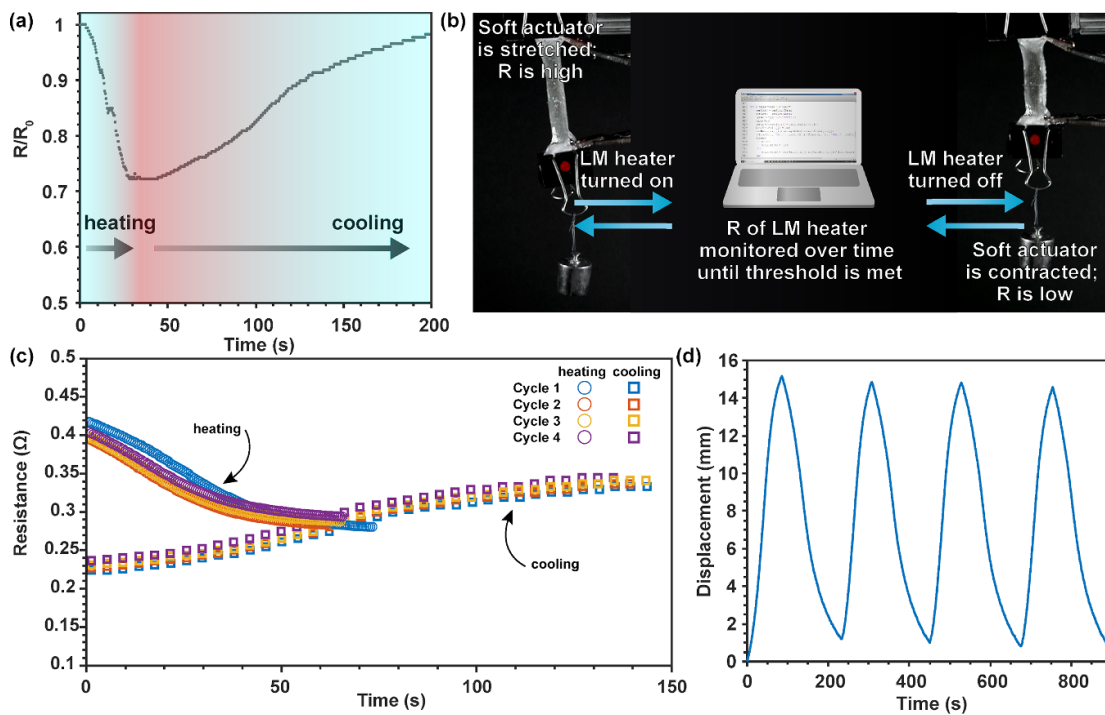


Figure 2: Closed-loop control of the soft actuator. (a) Normalized resistance as a function of time while heating the ambient environment with a heat gun, where the normalized resistance is the measured resistance (R) divided by the initial resistance (R_0). (b) Outline of closed-loop control where the resistance (R) of the soft actuator is measured and used to determine the electrical current delivered to the LM joule heating element. (c) Resistance as a function of time during heating and cooling demonstrated closed-loop control by coupling electrical power input with resistance output. (d) Actuator displacement as a function of time during actuation using closed-loop control.

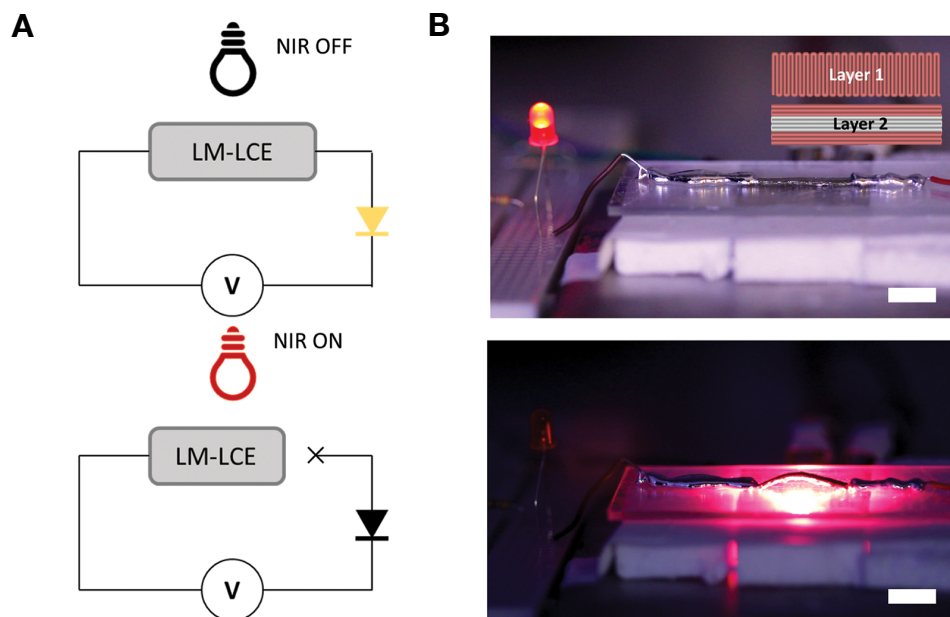


Figure 3. (A) Schematic of circuit for the LM-LCE actuator “switch” activating an indicator LED where the LCE bends, which opens the circuit and turns off the LED. (B) Sequential images of the “switch” turning on/off the indicator LED. Top: The sample is not irradiated and therefore the circuit is closed and the LED remains on. Bottom: The sample responds to NIR light and opens the circuit, turning off the LED. (inset) Top-view schematic of the printed patterns of the multimaterial printed actuator “switch” where the red material is 71 wt % LM and the gray material is 88 wt % LM. Scale bars are 5 mm.

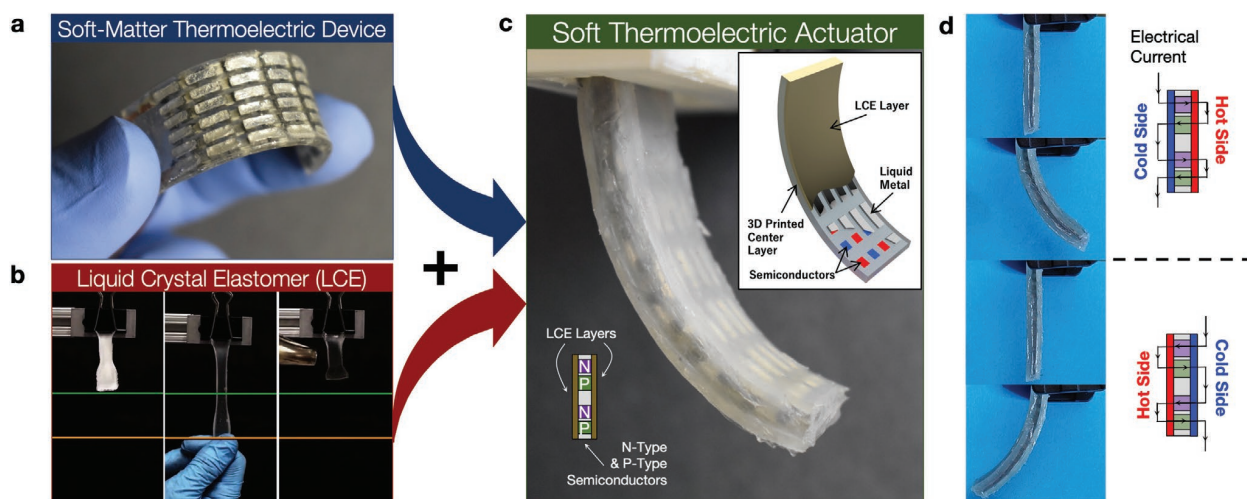


Figure 4. (a) A stretchable 90 semiconductor soft-matter TED under deformation highlighting conformity of LM traces and 3D printed center layer. (b) Images showing the responsiveness of the LCE shape memory polymer to heat. (c) LCE-TED soft limb during actuation with right side heating and left side cooling. The inset represents a schematic diagram showing LM traces and semiconductors beneath the LCE layer. (d) Images highlighting how the change in the current direction across semiconductors reverses the direction of actuation using only one input source.

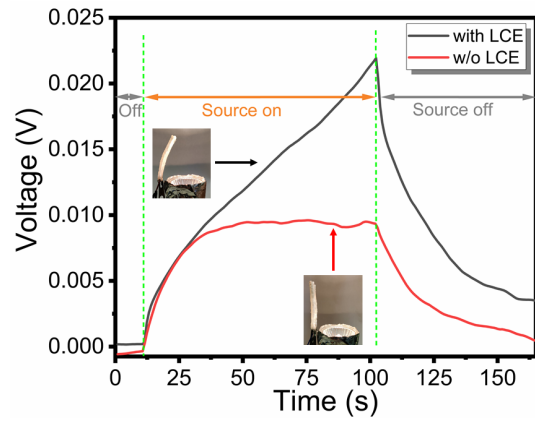
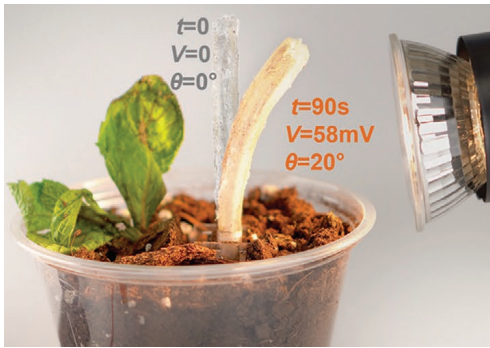


Figure 5. Results of phototropism-inspired energy harvesting test. The transducer was placed above and parallel to a heat source with voltage collected with and without the LCE layer, highlighting the physical intelligence of this system track towards a heat source and increase voltage output.

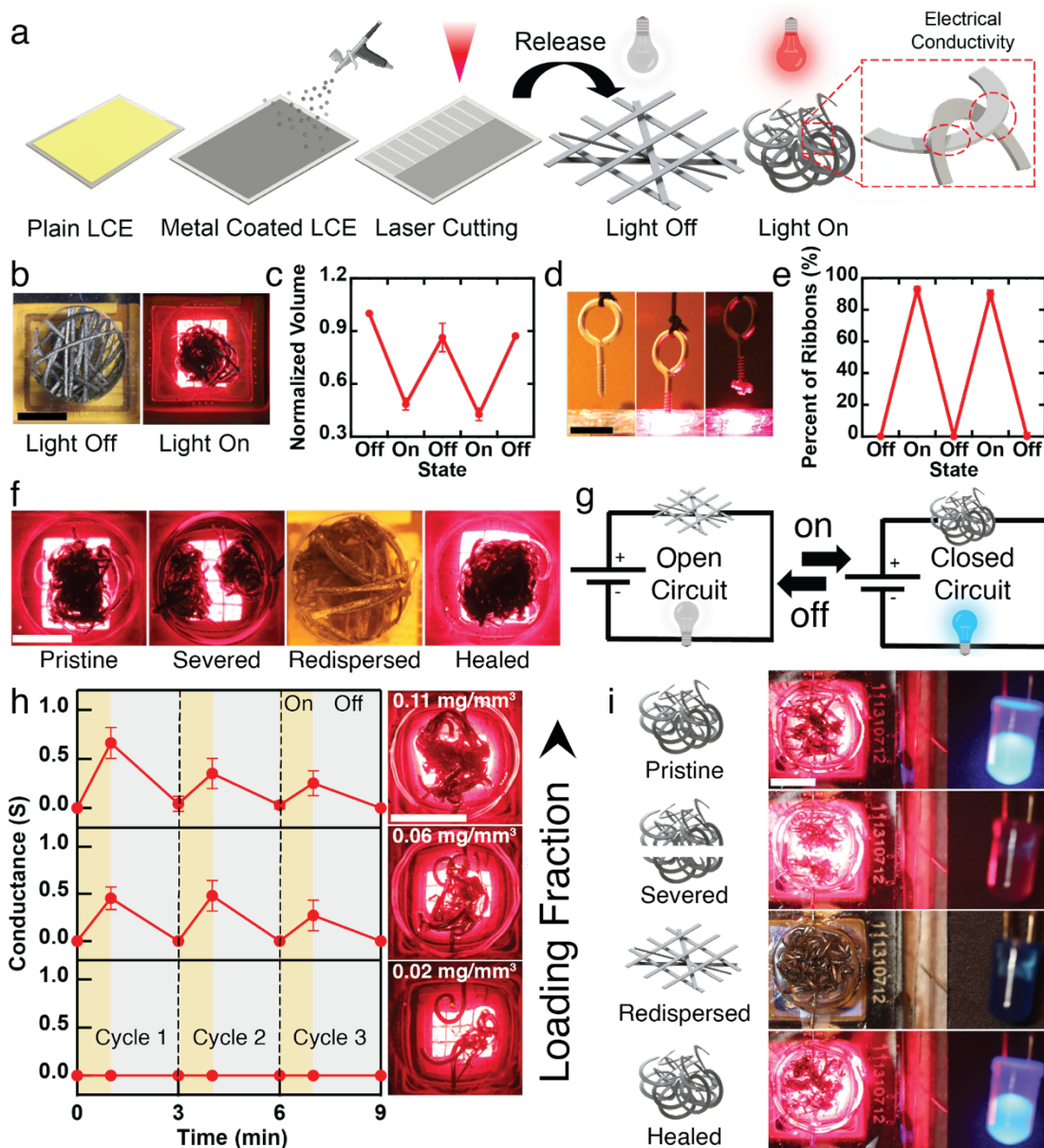


Fig. 6: Metal coating enables emergent functionalities **a**, Schematic illustrating the process of coating ribbons with liquid metal, enabling NIR responsive aggregates and conductive aggregates. **b**, Dispersion of coated ribbons with an offset angle of 10° and a length of 6 mm at a loading fraction of 0.075 mg/mm³ aggregating in the presence of NIR light (scale bar = 3 mm). **c**, Normalized volume plotted as a function of NIR light state of coated ribbons with an offset angle of 10° and a length of 6 mm at a loading fraction of 0.075 mg/mm³ for 3 cycles. **d**, Aggregation can be used to form a solid around a foreign object and latch on (scale bar = 5 mm). **e**, Percent of ribbons attached to the foreign object plotted as a function of the NIR light state. **f**, Aggregates are capable of healing damage through aggregating and redispersing using NIR light (scale bar = 3 mm). **g**, Schematic illustrating ribbons can transition from an insulating state to a conductive state depending on the state of the NIR light. **h**, Conductance measured as a function of time for 3 cycles of turning the NIR light from off to on for 3 different loading fractions (scale bar = 8 mm). **i**, Conductive aggregates are capable of healing damage (scale bar = 5 mm). For all plots lines between points are added to guide the eye. Error bars represent the standard deviation. N = 3.