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RPPR Final Report
as of 19-Jun-2020

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Proposal Number: 72746MSII **Agreement Number: W911NF-18-1-0298**
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STEM Degrees: 0 **STEM Participants:**

Major Goals: The purpose of the Short-Term Innovative Research (STIR) Program is to provide short-term funding to help launch high-risk, innovative research and allow researchers to obtain preliminary results that can steer further explorations of new concepts. To put our proposed work in a larger context, we first describe our overarching goals. In Section II, we describe the specific short-term studies we will undertake with this seed funding to help realize our long-term vision.
Our long-term goal is to revolutionize the design of soft robots, enabling these machines to exhibit the dynamic and adaptive functionality of biological systems. Current robots require microprocessors and microelectronics to direct and power the devices to perform specific tasks. For the robot to alter its function, the programmer must alter the code. Hence, such robotic systems are not particularly adaptive; they cannot spontaneously modify their functionality in response to environmental changes. In contrast, polymer gels offer a significant range of stimuli-responsive properties, allowing the material to dynamically and autonomously change its behavior in response to external cues. We aim to merge fundamental concepts from robotics and polymer science to design systems where the machine and the material are one and the same entity, thus creating soft robots that can autonomously sense, communicate, move and perform collaborative activities.
To carry out these studies, we must establish new design rules for creating systems that operate out of equilibrium and dissipate energy to function. To date, there are few guidelines for designing dissipative, dynamic materials, which are vital for fabricating the next generation of adaptive robots. To address this challenge, we will develop new multi-physics models that capture the range of dynamic mechanical, chemical, thermal and opto-electronic processes—and the inter-conversion of energy among these domains—that are needed to formulate the fundamental principles that will ultimately enable the fabrication of highly responsive, self-regulating, and cooperative soft robotic systems. Without such models, we are constrained to design “static materials” that are governed by thermodynamic equilibrium and thus, cannot make vital predictions needed to facilitate the fabrication of dynamic, adaptive material systems.

Accomplishments: Please see uploaded file.

In the future, we will build on these studies to:

• Harness more than one stimulus—including responsive surfaces To promote greater mobility and “programmable” structural reorganization, we will focus on gels that are responsive to more than one stimulus. The different stimuli can allow us to effectively “program” the assembly to exhibit complex, interactive behavior. For example, the thermo-responsive, loop-containing gels will be placed on top of a thin piezoelectric (PZ) film. The temperature induced distortion of the gels will impose a force on the PZ material, which generates a voltage under

deformation. If the gels are electro-active, the generated voltage will in turn affect the behavior of each unit and can be harnessed to direct the organization of the units. If the gels are also photo-responsive, then light can also be used to direct their interactions. Importantly, none of these interactions require microprocessors to direct the systems' behavior. Rather, this behavior evolves from the dynamic response of the system to external stimuli. These stimuli can be applied simultaneously or serially, providing additional means of "programming" the dynamic behavior of the system.

- Utilize chemical gradients in solution With the gels in solution, we can introduce another component into the system that will provide chemical stimuli and a "fuel" to direct the movement of the gels. In previous studies, we utilized the exothermic reaction between reagents in the solution and catalytic sites in the gels to drive the thermo-chemo-mechanical transduction in the system. Gradients of chemical reagents in the solution can also be used to direct the movement of the gel-bots; this is particularly intriguing when the gels themselves produce4 or release the chemical gradients.

Training Opportunities: The postdoctoral fellow associated with the project, Dr. Santi Biswas, participated in weekly meetings of the research groups, where he had his oral presentations critiqued both for substance and delivery. Presenting talks at national meetings is a key to the future success of postdocs. The PI encouraged and funded Dr. Santi to present his findings at the American Physical Society Meeting. The weekly group meetings provided him with a forum to hone his presentation skills and his material, and thus, prepare him for these conferences.

The PI afforded Dr. Biswas the opportunity to develop, prepare and present lectures in an undergraduate class. The PI mentored Dr. Biswas in the development of lesson plans, lectures, and interactive activities that are especially attractive and comprehensible to the undergraduates. Dr. Biswas also had opportunities to interact with students in the class, which required the students to develop their own computer code. Finally, Dr. Biswas was also given opportunities to improve upon this component of the course, and in particular, develop new assignments for this course.

The PI also engaged Dr. Biswas in writing research proposals and thereby, teaching him the critical skill of formulating logical arguments and writing in the most effective manner to convey his ideas. As a starting point, we used an approach articulated by George Heilmeier, who was a former director of DARPA and developed a set of questions to help formulate thoughts for a new project. They are (taken from Wikipedia's article):

1) What are you trying to do? Articulate your objectives using absolutely no jargon. 2) How is it done today, and what are the limits of current practice? 3) What's new in your approach and why do you think it will be successful? 4) Who cares? 5) If you're successful, what difference will it make? 6) What are the risks and the payoffs? 7) How much will it cost? 8) How long will it take? 9) What are the midterm and final "exams" to check for success?

This set of questions provides is a good starting place to teach effective proposal writing and, consequently, preparing Dr. Biswas for the highly competitive research environment that he will enter in the next stage of his career.

Results Dissemination: Dr. Santi Biswas presented his findings at the APS meetings.

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Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: PD/PI
Participant: Anna C. Balazs
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)

RPPR Final Report

as of 19-Jun-2020

Participant: Santi Biswas
Person Months Worked: 12.00 **Funding Support:**
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National Academy Member: N
Other Collaborators:

FINAL REPORT: DESIGNING "GEL BOTS" THAT COMMUNICATE THROUGH SELF-GENERATED MECHANICAL FORCES

I. RESEARCH NARRATIVE

Our long-term goal is to revolutionize the design of soft robots, enabling these machines to exhibit the dynamic and adaptive functionality of biological systems. Current robots require microprocessors and microelectronics to direct and power the devices to perform specific tasks. For the robot to alter its function, the programmer must alter the code. Hence, such robotic systems are not particularly adaptive; they cannot spontaneously modify their functionality in response to environmental changes. In contrast, polymer gels offer a significant range of stimuli-responsive properties, allowing the material to dynamically and autonomously change its behavior in response to external cues. We aim to merge fundamental concepts from robotics and polymer science to *design systems where the machine and the material are one and the same entity*, thus creating soft robots that can autonomously sense, communicate, move and perform collaborative activities.

To drive the gel bots to undergo dramatic changes in volume, we developed a theoretical and computational model for thermo-responsive gels that encompass polymeric loops. *The inclusion of loops allows the material to undergo dramatic, reversible changes in volume and shape*. The gel in **Fig. 1** exhibits a lower critical solution temperature (LSCT) and thus expands when the temperature, T, is decreased; the expansion of the gel causes the loops to unfold. (The loops and gel return to their original structure when temperature is increased.) *Via* our model, we designed a new class of reconfigurable, patterned gels that encompass sections with and without loops. The sample in **Fig. 1** contains eight fundamental sections where alternate sections in the sample contain loops. (The length of the smallest cubic element outlined in back lines in Fig. 1 corresponds to 40 μm; our systems can range from the μm to *cm* length scale.) These gels formed the versatile modular units in our exemplar system. By alternating the arrangement of loop-containing sections, we can now design networks that swell into distinct architectures as T is varied (**Fig. 1b**). Furthermore, by causing the expansion of just the loop-containing domains, we can achieve asymmetric actuation, which can facilitate locomotion of the gel bots. Below, we provide a brief description of our recent studies.

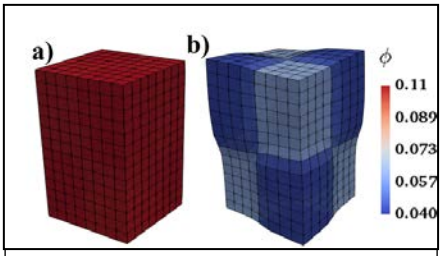

Fig. 1. Results of our simulations on patterned gels where only alternating units contain loops.

II. RESULTS AND DISCUSSION

A. Development of Model: Achieving Large Volume Changes with Loops

1. *Theoretical Modeling of Gels Encompassing Thermo-responsive Loops*

To perform these studies, we veloped the necessary theoretical models that characterize the internal structure of the polymer network, which encompasses thermo-responsive loops that can unfold and refold. Using this information, we then derived the constitutive equation for this system. Below, we describe each step of this derivation.

a. Modeling the folding and unfolding of chains in a polymer gel We consider a permanently cross-linked, swollen polymer gel. The cross-links in the network are marked by the X's in **Fig. 2** and the chains between these cross-links are drawn in gray. These chains contain reactive, labile bonds—the blue and red dots in the figure. In the undeformed state, these reactive sites can bind to form a loop. When the thermo-responsive gels expand and thus the network is mechanically deformed, the labile bonds break and

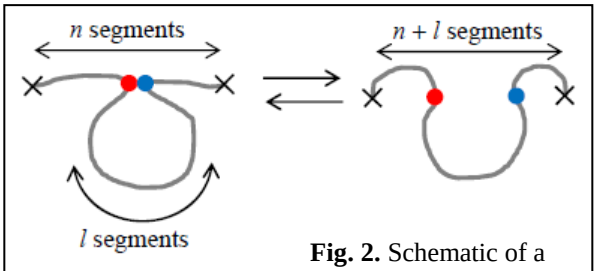

Fig. 2. Schematic of a network chain undergoing folding and unfolding. Reactive units that can form a labile bond are shown as red- and blue-colored disks.

cause the chain to unfold. If the deformation is released, the labile bonds can come together to reform the loop, so that the network chains reassemble the initial folded conformation.

b. Unfolding at finite extensibility of network chains A chain in the network is considered to contain $n+l$ Kuhn's segments, with l being the number of segments that form the loop (see **Fig. 2**). We assume that the time scale for the breaking and reforming of the bond between the reactive groups is longer than the time scale associated with conformational changes of the chain. Hence, the probability of finding the chain in the unfolded conformation, p_U, as a function of time t is governed by the equation for the kinetics of bond rupture and formation

$$dp_U / dt = k_r(R)(1 - p_U) - k_f(R)\, p_U \quad . \tag{1}$$

Here, $k_r(R)$ and $k_f(R)$ are the respective rate constants of the bond rupture and formation that depend on the distance between the chain ends, R. The rate of bond rupture depends on R because the stretching of the chain increases the force acting on the bond, and the force facilitates bond breakage. We utilize the Bell model to take the latter effect into account, and hence the rupture rate is calculated as

$$k_r(R) = k_r^{(0)} \exp[\gamma_0 F_n(R) / k_B T] \quad , \tag{2}$$

where $k_r^{(0)}$ is the rupture rate at zero force, $F_n(R)$ is the force created by separating the ends of a chain of n segments to the distance R, and γ_0 is the parameter that characterizes the sensitivity of the bond to applied force. The loop does not contribute to $F_n(R)$, which depends only on the number of segments in the unlooped part of the chain, n (**Fig. 2**).

The force acting on the bond is calculated according to the freely-jointed chain model (FJC), which accounts for the finite extensibility of the chain and yields the following expression:

$$F_n(R) = \frac{k_B T}{b} \mathcal{L}^{-1}[R(nb)^{-1}] \quad (3), \qquad \text{where } \mathcal{L}(x) = \coth(x) - x^{-1} \quad (4)$$

Here, $\mathcal{L}(x)$ is the Langevin function. In eq. (3), k_B and T are the respective Boltzmann's constant and temperature, and b is the length of the Kuhn's segment. It is convenient to re-write the dimensionless variable $x = R(nb)^{-1}$ in eq. (4) in terms of the chain extension λ as $x = \lambda n^{-1/2}$.

The rate constant for forming a labile bond, $k_f(R)$, depends on the chain end-to-end distance R because in order to form a bond, the reactive units in the unfolded chain of $n+l$ segments must first come in contact, and the probability of contact, P_c, depends on R. When in contact, the reactive units form a labile bond with the rate constant $k_f^{(0)}$, and hence $k_f(R) = P_c(R) k_f^{(0)}$. As in previous studies, [13-18] it is assumed that $k_f^{(0)}$ does not depend on the force acting on the bond. The probability of contact, $P_c(R)$, is calculated using the conformational statistics of the polymer chain: $P_c(R) = P_n(R) P_l(0) / P_{n+1}(R)$, where $P_n(R)$ is the probability distribution function for finding the ends of a chain of n segments at distance R apart. For the FJC model, this distribution function is [12]

$$P_n(R) = \frac{[\mathcal{L}^{-1}(x)]^2}{(2\pi n b^2)^{3/2} x \{1 - [\mathcal{L}^{-1}(x)\,\mathrm{csch}(x)]^2\}^{1/2}} \left[\frac{\sinh \mathcal{L}^{-1}(x)}{\mathcal{L}^{-1}(x)} \right]^n \exp[-nx\mathcal{L}^{-1}(x)] \tag{5}$$

where $x = R(nb)^{-1} = \lambda\, n^{-1/2}$. Note that through the equations for $k_f^{(0)}$ and $P_c(R)$, the rate of chain folding depends on both the total length of the chain $(n+l)$ and the length of the loop (l).

Figure 3 shows how the probability of unfolding in the steady-state depends on the chain extension λ at various values of γ_0, the parameter characterizing the sensitivity of the bond to the applied force. At steady-state, $dp_U/dt = 0$ so that

$$p_U(R) = \{1 + k_f^{(0)}/k_r^{(0)}\, P_c(R) \exp[-\gamma_0 F_n(R)/k_B T]\}^{-1} \quad , \tag{6}$$

The figure reveals that as the dimensionless parameter γ_0/b is increased, the unfolding occurs at progressively lower chain extensions.

c. Constitutive equation for gel containing folded and unfolded network chains By deriving eq. (11) below, we obtain the necessary constitutive equation of the system, as discussed here. The stress-strain relationship for the polymer gel is described by the following equation

$$\hat{\boldsymbol{\sigma}} = \hat{\boldsymbol{\sigma}}_{\text{el}} - \pi_{\text{FH}}(\phi)\hat{\mathbf{I}} \quad , \tag{7}$$

where $\hat{\boldsymbol{\sigma}}_{\text{el}}$ is the contribution from the elasticity of the polymer chains, and $\pi_{\text{FH}}(\phi)$ is the osmotic pressure in the system according to the Flory-Huggins model

$$\pi_{\text{FH}}(\phi) = -[\phi + \log(1-\phi) + \chi(\phi)\phi^2] \quad , \tag{8}$$

where ϕ is the volume fraction of polymer, and the function $\chi(\phi)$ describes the polymer-solvent interactions. The polymer elasticity contribution is usually introduced through the neo-Hookean model

$$\hat{\boldsymbol{\sigma}}_{\text{el}} = c_0 \phi_0^{-1} \phi \hat{\mathbf{B}} - c_0 \phi (2\phi_0)^{-1} \hat{\mathbf{I}} \quad , \tag{9}$$

where c_0 is the cross-link density, $\hat{\mathbf{B}}$ is the Finger strain tensor, and ϕ_0 is the volume fraction of the polymer in the as-prepared gel.

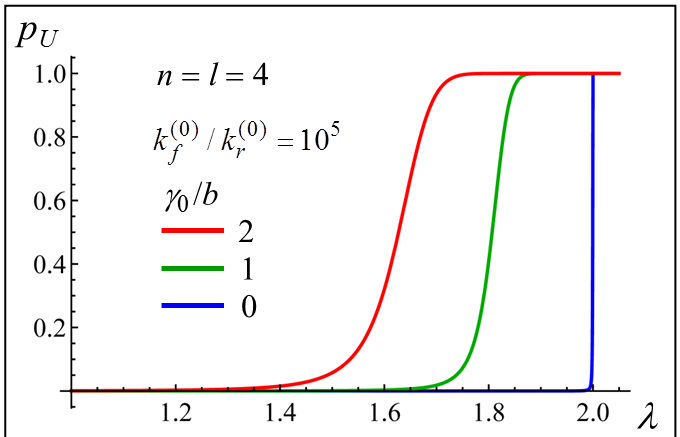

Fig. 3. Probability of unfolding the loop as a function of the chain extension λ at different values of γ_0/b.

Equation (9) for $\hat{\boldsymbol{\sigma}}_{\text{el}}$ does not capture the finite chain extensibility effects; since the latter effects are crucial in our system, we must derive a new expression. To do so, we use a generalization of eq. (9) based on the FJC model to obtain

$$\hat{\boldsymbol{\sigma}}_{\text{el}}(n) = c_0 \phi_0^{-1} \phi\, \zeta(\bar{\lambda}\, n^{-1/2}) \hat{\mathbf{B}} - c_0 \phi (2\phi_0)^{-1} \zeta_0 \hat{\mathbf{I}} \quad . \tag{10}$$

Here, $\zeta(x) = (3x)^{-1}\mathcal{L}^{-1}(x)$, where $\mathcal{L}^{-1}(x)$ is the inverse Langevin function, $\zeta_0 = \zeta(n^{-1/2})$, and $\bar{\lambda}$ is the average strain calculated as $\bar{\lambda} = \sqrt{(\lambda_1^2 + \lambda_2^2 + \lambda_3^2)/3}$ where λ_1, λ_2, and λ_3 are the principal strains. In contrast to eq. (9), the stress tensor in eq. (10) explicitly depends on the number of Kunh segments in a network chain, n.

In the systems considered here, some of the chains are in the folded state and some chains are in the open configuration. We assume that the deformation of the gel is affine, so the contributions of the folded and unfolded chains to the network elasticity are additive. The resulting constitutive equation is

$$\hat{\boldsymbol{\sigma}} = p_U(\bar{\lambda})\hat{\boldsymbol{\sigma}}_{\rm el}(n+l) + [1 - p_U(\bar{\lambda})]\hat{\boldsymbol{\sigma}}_{\rm el}(n) - \pi_{\rm FH}(\phi)\hat{\mathbf{I}} \quad , \tag{11}$$

where $p_U(\bar{\lambda})$ is the probability of chain unfolding at the average degree of stretching $\bar{\lambda}$ and is found from the equation for the bond kinetics, eq. (1), or from eq. (6) for the steady-state scenario. In eq. (11), it is assumed implicitly that all network chains in the gel contain the reactive groups. We relax this restriction in the section below; this restriction can be easily modified so that only a given fraction of the chains contain such sites.

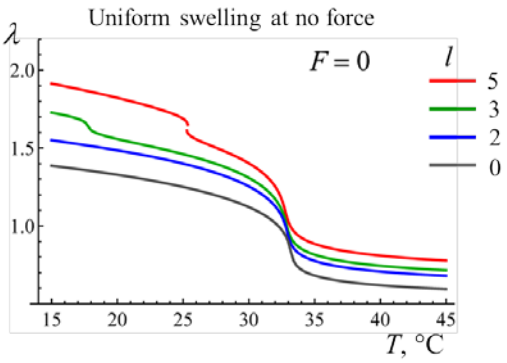

Fig. 4. Gel size at various numbers of segments in the loops, l. Here, n = 4.

Using this newly developed model, we considered the equilibrium swelling of a poly(N-isopropylacrylamide) (pNIPAAm) gel as a function of temperature (**Fig. 4**). For affine deformations, the degree of swelling (λ in the above plot) is equal to the chain extension, so the same notation is used for the both values. This gel has a lower critical solution temperature (LCST) and thus it swells as temperature is decreased. The parameters we used to describe pNIPAAm gels are taken from the relevant experimental values (as detailed in ref. 21). **Figure 4** shows the gel size obtained from our calculations in the absence of an applied external force. If the loops are sufficiently long, then the degree of swelling exhibits distinctive "jumps" when the chains unfold due to the internal stresses developed in the course of swelling.

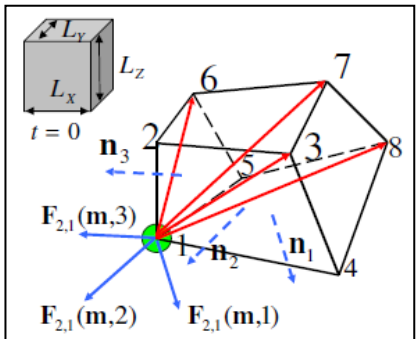

Fig. 5. Schematic of the 3D element $\mathbf{m} = (i, j, k)$.

The above evolution equations are discretized and solved numerically using our three dimensional gel Lattice Spring Model (gLSM). Within the framework of this model, a gel layer is represented by a set of general lineal hexahedral elements (see **Fig. 5**). Initially, the sample is undeformed and consists of $(L_x - 1)\times(L_y - 1)\times(L_z - 1)$ identical cubic elements; here L_i is the number of nodes in the i-direction, $i = x, y, z$. In the undeformed state, each element is characterized by the same volume fraction ϕ_0 and cross-link density c_0. Upon deformation, the elements move together with the polymer network so that the amount of polymer and number of cross-links within each hexahedral element remain equal to their initial values.

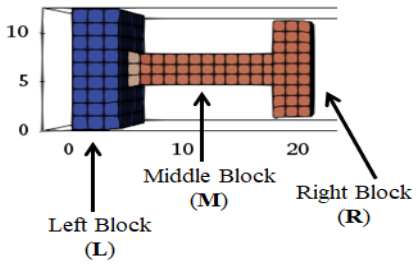

Fig. 6. Schematic of the gel bot

Through the gLSM, we will introduce an underlying substrate and introduce a friction coefficient between the gels and this surface. We have already modeled multiple chemo-responsive gels that interact through a steric repulsion. Hence, with these different components of the model, we are ready to simulate the interaction of the gel bots on the surface. A significant challenge is to determine how to "program" the units to undergo concerted motion.

B. Directing the Concerted Motion of the Gel Bots

For our initial studies that provide "proof-of-concept", we designed a gel bot that comprises three spatially separated domains (**Fig. 6**). Such structures allow us to produce coordinated movement by: 1)

varying the temperature, 2) patterning the arrangement loops in the gel and 3) tailoring the shape of the individual domains. For the basic structure in **Fig. 6**, we established key design rules that gave rise to novel forms of actuation (**Fig. 7**). The design rules are as follows:

Step 1. The gel bot is initially equilibrated at 30 °*C*. The left Block (**L**) is then cooled to T = 15 °*C*, leading to the swelling of the block. Consequently, the block becomes anchored to the top & bottom plane.

Step 2. The left block (**L**) acts as an anchor. The middle block (**M**) is then cooled to T = 15 °C, leading to the swelling and lateral extension of this middle portion. This portion take a "stride" (as in a walk), and thus moves the right block (**R**) forward.

Step 3. The right block (**R**) is now cooled to 15 °*C*. Once the **R** block becomes swollen, it is anchored in place between the top and bottom planes.

Step 4. Now that the gel bot has taken a stride forward, the temperature is increased to T = 30 °*C* at **L.** Consequently**, L** de-swells and releases the anchor at the left end.

Step 5. M is now heated to T = 30 °C, resulting in the contraction of the length of this domain. Subsequently, **L** is cooled back to T = 15 °*C* to act as an anchor.

Step 6. The right block (**R**) is then heated to T =30 °*C* to shrink this portion and free the right-end anchor.

We first applied these design rules to a gel bot composed of elements that do not contain any loops. To create successive strides forward, we repeated steps 2 through 6 and thus achieved directed movement of the gel bot (**Fig. 7**). In one cycle, the gel traverses ~ 3.2 units (128 μm) the in x-direction.

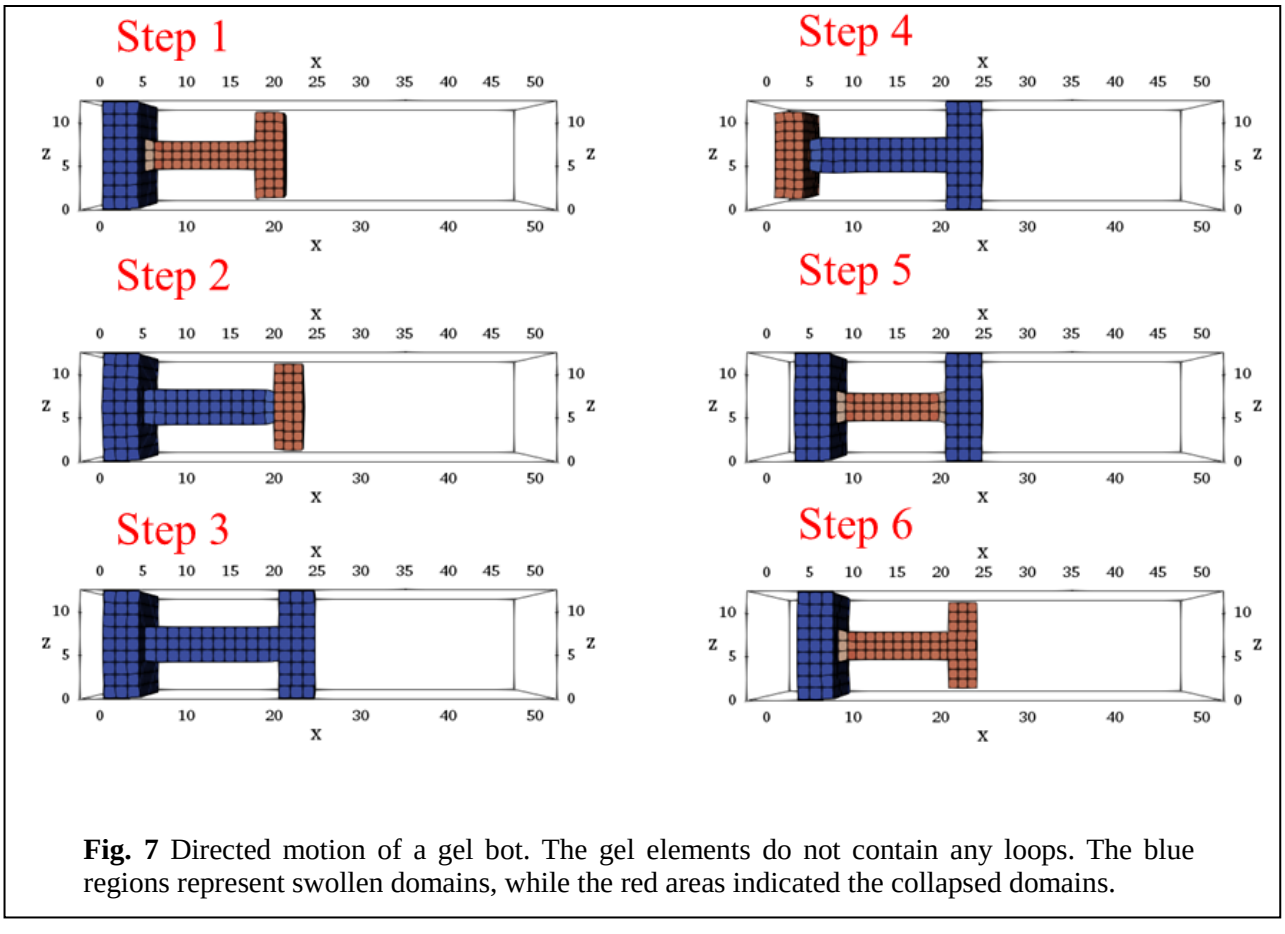

Fig. 7 Directed motion of a gel bot. The gel elements do not contain any loops. The blue regions represent swollen domains, while the red areas indicated the collapsed domains.

In the following study (shown in **Fig. 8**), we introduced loops of Kuhn length l = 4 (**Fig. 2**) solely within the elements of the middle block (**M**), i.e., the left and right blocks do not contain loops. As above, decreases in temperature cause the LCST gel to swell. In addition, the swelling now causes the loops to unfold and release the hidden length, which contributes to the stride made by this central domain. The sample in **Fig. 8** traverses ~ 7.0 units (128 μm) in the x-direction.

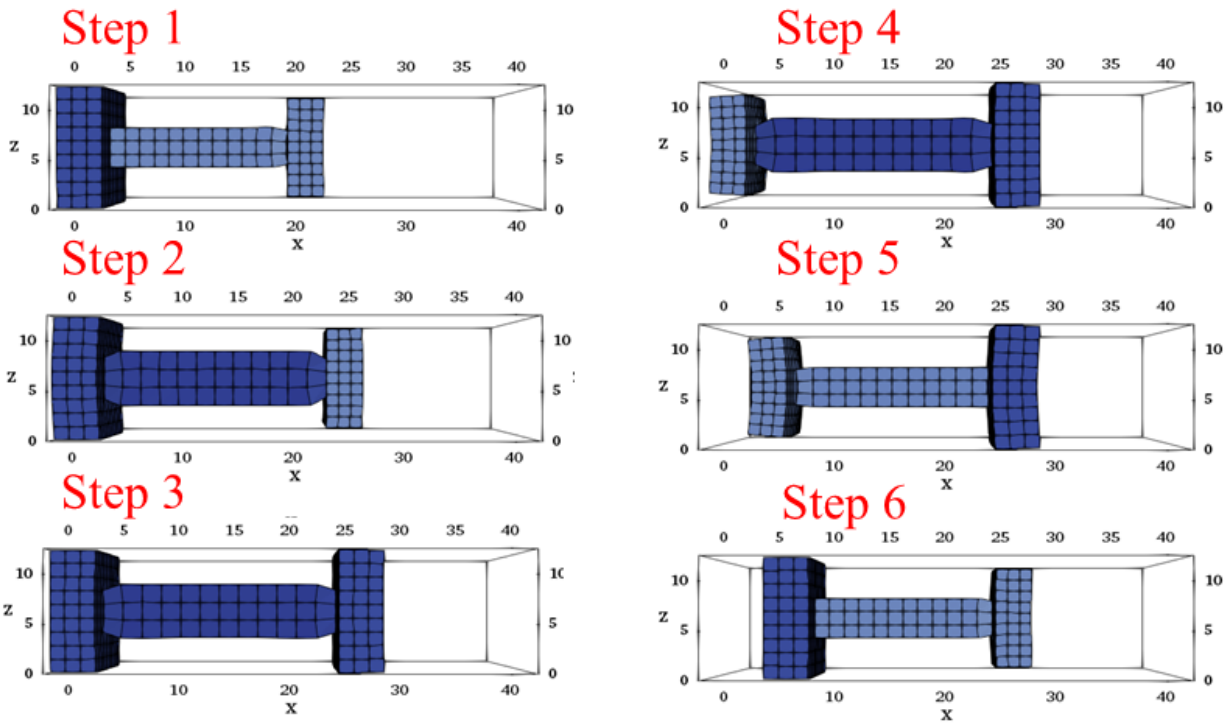

Fig. 8 Directed motion of a gel bot with elements in the middle block containing loops with Kuhn length l=4.

III. SUMMARY

We modified our gLSM approach to simulate materials that transduce and harvest energy from the local environment (rather than external electrical power) to perform directed motion. By co-designing the steps to actuate the gel and the shape of the gel, we showed that simple LCST gel structures can exhibit coordinated movement. In effect, the gel bots take a forward stride with each cycle of directions (steps 1-6). This new gLSM allowed to model polymer gels that encompass loops; the temperature-induced unfolding of the loops releases the stored hidden length. Utilizing controlled variations in temperature and the placement of loops, we showed the stride length can be increased by roughly a factor of two in each cycle. Our modeling studies can help launch the development of self-regulating, interactive soft robots that rely on the innate response of the materials—rather than the coding of microprocessors—to autonomously perform a range of useful functions. Such developments would revolutionize the entire field of soft robotics.