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A study of mechanoelectrical transduction behavior in polyvinyl chloride (PVC) gel as smart sensors

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Abstract

Polyvinyl chloride (PVC) gels are soft electroactive polymers (EAPs) being researched for soft robotic applications. Sensing properties of these EAPs have not been investigated in detail in regard to fundamental mechanoelectrical transduction behavior, but this smart material has been shown to exhibit a detectable response to external stimuli. This study shows PVC gels to be an extremely sensitive material when undergoing mechanoelectrical transduction and explores some response dependencies and proposes a theoretical framework for mechanoelectrical transduction within the gel. The work presented here also uncovers a very interesting phenomena under extremely low compressive loads during the initial contact with the gel. This phenomenon is attributed to a surface tension creeping motion onto the loading surface with an accompanying polarity inversion in the sensing signal relative to fully loaded gels in compression. Experimental work on hysteresis was also completed showing very little memory in steady state mechanoelectrical response to repeated stepped loading cycles. This study demonstrates the mechanoelectric ability of PVC gels to perform in sensing experiments and acts as a fundamental framework to further broaden the applications of PVC gel sensors.

Keywords: polyvinyl chloride (PVC) gels, electroactive polymers, mechanoelectrical transduction

(Some figures may appear in color only in the online journal)

1. Introduction

Polyvinyl chloride (PVC) gels are electroactive polymers comprised of a PVC matrix with varying composition and amounts of plasticizer [1, 2]. Adding plasticizer to the PVC matrix results in a decreased modulus of the material, and is tunable for desired stiffness [3, 4]. An abundance of plasticizer content causes fluid plasticizer to situate interstitially between the PVC matrix and allows for free plasticizer motion [2]. A few plasticizers have been experimentally tested on PVC gels for actuation properties, in which dibutyl adipate (DBA) has proven to provide desirable actuation properties [5, 6]. DBA has a polar molecular structure that will polarize under an applied electric field [2, 7]. Like many dipolar materials, once an electric field is dissipated DBA molecules will exhibit hysteresis potentially resulting in a steady state voltage response due to remnant polarization [8]. Common modeling methods for explaining dipolar polarization include the use of the Langevin function to describe the polarization of dipolar insulators [9, 10]. This may be useful in modeling the free interstitial plasticizer polarization within the PVC matrix, however the Langevin model does not capture relaxation of dipoles.

The general theory of actuation for PVC gels is as follows: under an applied electric field DBA migrates towards the anode due to a change in space charge density, causing

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a significant decrease in the modulus of the material in this solvent rich layer, Maxwell and electrostatic forces then cause a creeping of the solvent rich layer around the anode known as anodophilic creeping [8, 11–13]. This theory of actuation is not wholly understood and is an area of active research including describing the fundamental driving force through mathematical modeling [14, 15].

There have been very few studies on mechanoelectrical transduction properties of PVC gels. One study conducted by Tanaka and Hirai investigated PVC gel mechanoelectrical transduction under impulse loading, which found some potential for PVC gels to undergo mechanoelectrical transduction under compressive impulse loading [16]. The outcome of this study showed a sensing potential including a voltage output dependent on plasticizer content and magnitude of loading input. Tanaka and Hirai show the mechanoelectrical potential of PVC gels under impulse loading but do not address fundamental framework of mechanoelectrical transduction. The study also only observes transient response and attempts to show magnitude of mechanoelectrical response is related to magnitude of impulse with limited experimental results. Ko *et al* tested PVC gel sensors under dynamic loading and were compared to common piezoelectric materials in terms of sensing behavior [17]. This study alludes to a piezoelectric-like response of PVC gel sensors under dynamic strain sensing with very low phase shift. These PVC gels showed desirable sensing properties in comparison to common piezoelectric sensing materials. PVC has also been used in conjunction with piezoelectric films and have been found to increase mechanoelectrical transduction of these composite materials in comparison to the pure piezoelectric films [18]. Under these mechanoelectrical studies, there is no study on the fundamental mechanoelectrical response including signal dependencies or steady state responses of PVC gel sensors. These limited studies strictly show dynamic oscillatory loading applications and compares mechanoelectrical response to commonly used piezoelectric materials. These studies address the underlying mode of mechanoelectric transduction as piezoelectric with limited experimental results and no static loading applications.

In this study, we aim to investigate fundamental mechanoelectrical transduction characteristics of PVC gel sensors. This study is comprised of four major experiments to investigate the characteristics of PVC gels in compression. In the first experiment the strain rate application is varied to determine the effect of strain rate on steady state response of the gel sensors. In the second experiment DBA content is varied to determine the effect of plasticizer content on PVC gel mechanoelectrical transduction. In the third experiment differing thicknesses of gel samples are tested to determine if the mechanoelectrical transduction is a volumetric effect within the gel. In the final experiment varying cross-sections of gel samples are tested to determine any edge effects as well as aid in the determination of the fundamental relationship of the mechanoelectrical transduction within the gel samples. These results with the results from varying thickness can help determine any volumetric or surface effects while the samples undergo mechanoelectrical transduction. Additional results are presented highlighting the surface properties of PVC gels including the creeping effect of this material onto the loading plunger with the associated polarity inversion of electrical response in the gel samples.

Table 1. Fabricated samples showing composite weight ratios of experimental PVC gel samples and associated naming convention.

Name	P4	P6	P8
PVC:DBA ratio	1:4	1:6	1:8

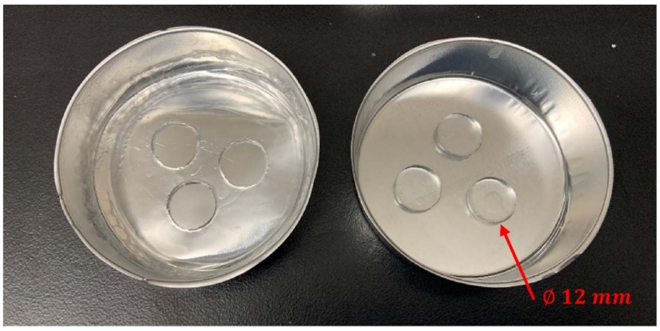

Figure 1. Sample preparation for mechanoelectrical experimentation by punching circular samples from a cured dish of PVC gel material (*left*) to help reduce edge effects and defects in the experimental samples (*right*).

2. Methodology

Fabrication was conducted using the casting method with tetrahydrofuran (THF) as the solvent. A mixture of commercially available PVC (Mw = 80 000 g mol^{-1}) and DBA (Mw = 258.38 g mol^{-1}) were dissolved in a bath of THF, which were all purchased from Sigma-Aldrich Co. The ratios of PVC to DBA were composed of 1:4, 1:6, and 1:8 by weight as seen in table 1. This mixture was stirred at 50 °C for 4 h. This mixture was then poured into a casting dish and allowed to cure at room temperature for four days under a fume hood where the THF was evaporated out of the mixture leaving the prepared PVC gel. The experimental samples were then cut using a circular punch to reduce any edge effects incurred during the curing process (figure 1).

Experimentation on the PVC gel samples was conducted using a modified 3D printer controlled by an Arduino microcontroller and attached motor driver (figure 2). This allowed for high control of compressive force application to the PVC gel samples using standard 3D printer software. A stepped compressive force input allowed observations to be made on the transient and steady state mechanoelectrical responses of the tested PVC gels. This force application mechanism contained an integrated Transducer Technologies GS0-100 load cell that was powered by a GW Instek GPS-2303 power supply with a copper electrode placed on the plunger. A copper electrode was also placed on the base of the mechanism. The PVC gel sample was then placed onto the copper electrode on the base of the mechanism, and the plunger was lowered onto

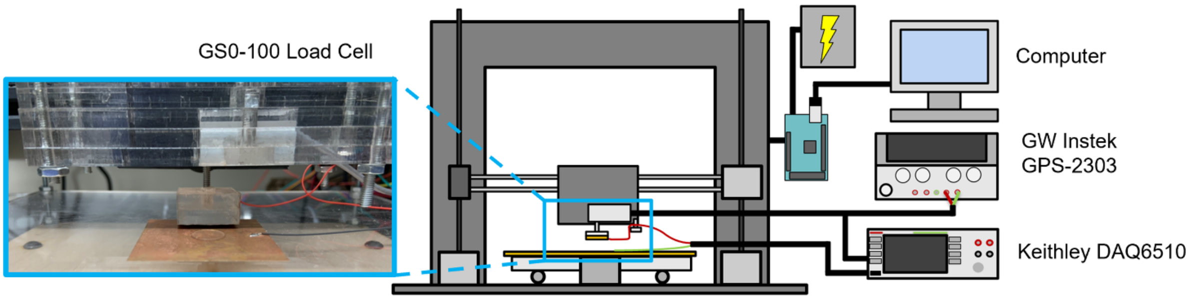

Figure 2. Experimental setup with force application mechanism and integrated force sensor for mechanoelectrical experimentation with magnified testing region (*left*).

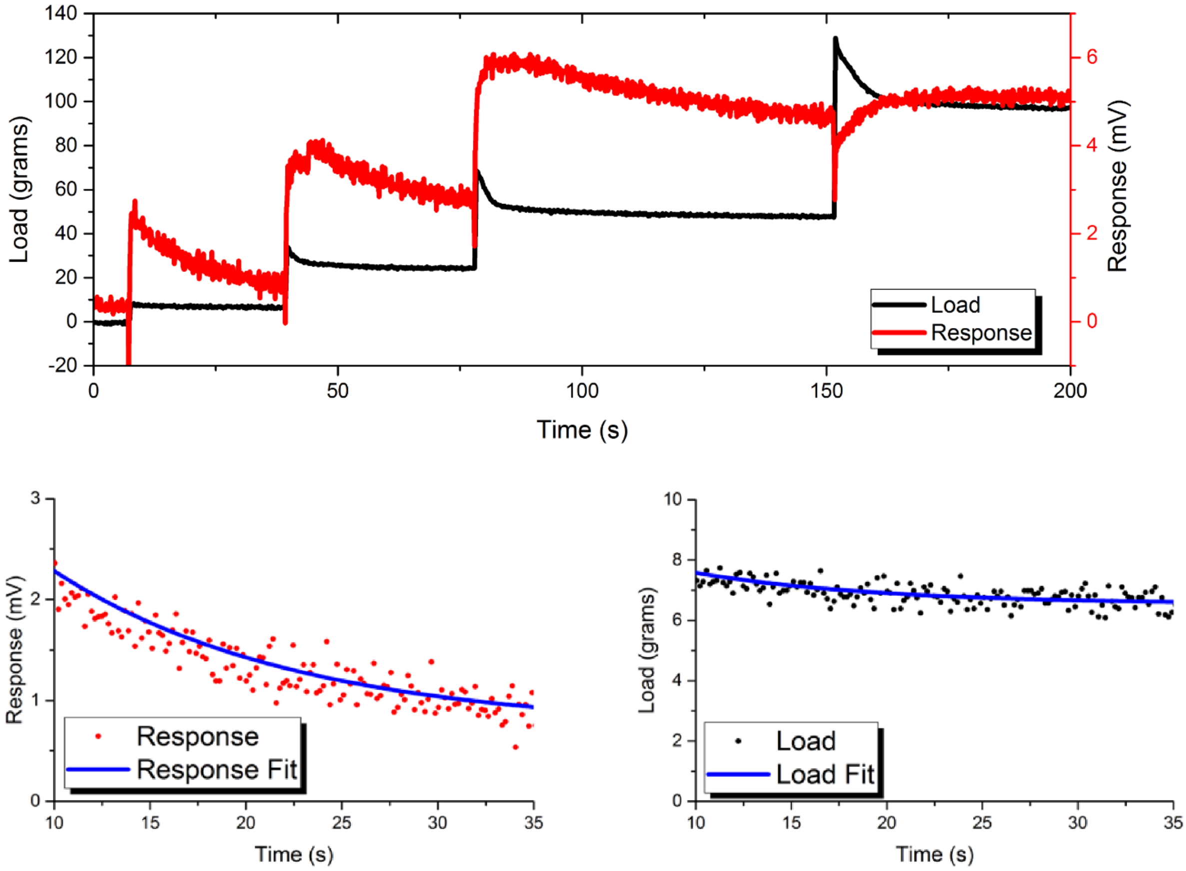

Figure 3. Raw data collected from mechanoelectrical testing (*top*) and fitting single exponential curves for steady state approximations of response (*bottom left*) and load (*bottom right*) to determine steady state values.

the PVC gel sample. The plunger was incrementally lowered onto the gel sample, and data was recorded using the Keithley DAQ6510 data acquisition and multimeter system. This instrument recorded both the force application from the integrated load cell as well as the mechanoelectrical response of the gel samples through the copper electrodes.

Each experiment consisted of three test samples of the same material characteristics. Three test samples were used due to the fabrication method where three samples could be cut from a single cured dish of PVC gel material. These samples underwent mechanoelectrical transduction under a single stepped compressive loading cycle. The raw data was then processed into individual step loads (figure 3). These individual data sets were then fit using a single exponential function for both the load and voltage response of the gel sample. This simple model agrees well with the collected data

$$y(t) = a\mathrm{e}^{-bt} + c \tag{1}$$

where constant a, b, and c will differ for the load and electrical response of each data set. The c parameter in the resulting fit functions gave the estimated steady state values for load and electrical response of the gel sample due to the decay of the exponential term as time increases. This function was fit to both the response and the load using a nonlinear least squares regression. The transient response, specifically at higher loading inputs shows a phase lag in response where the peak response may not coincide with the peak time of the load. This may be due to steric effects within the gel near the saturation point of the mechanoelectrical response. While fitting the response curve, data was taken from the peak of the response to the end of the loading cycle. The steady state estimated value, or c parameter, for the transient response shown (figure 3 *bottom left*) is 0.7197 mV with a 95% confidence level between 0.5781 mV and 0.8613 mV. The steady state estimated value for the load shown (figure 3 *bottom left*) is 6.777 g with a 95% confidence level between 6.709 g and 6.846 g.

The observed decay in the load signal is likely caused by the viscoelastic properties of the gel samples. The Kelvin–Voigt viscoelastic material model assumes an exponentially decaying stress relaxation behavior upon stepped strain inputs, which is experimentally observed. The response is more phenomenalistic in fitting with this simple model but may be directly related to surface stress within the material. The observed decay in the mechanoelectrical response of the PVC gel sample is potentially due to the relaxation of the interstitial plasticizer. The initial field may relate to polarization of plasticizer, then when the load is held constant the dipolar plasticizer molecules may relax causing further decay in mechanoelectrical response. Another interesting component of the transient response is the initial response seems to be a very fast negative electrical output to each stepped loading application. This may be due to the initial disturbance of the polar polymeric lattice structure within the gel samples.

2.1. Experiment 1—strain rate

The strain rate was varied using 1, 2, 5, and 10 mm s^{-1} loading application on the gel samples. This was completed by editing the 3D printer software to apply compressive stepped loads at these rates, which in turn applied varying strain rates to the gel samples. This range covers one order of magnitude within the operating range of the force application mechanism. Three test samples were fabricated for each of the incoming strain applications resulting in a total of 12 samples. Each of these P4 samples were punched to a 12 mm diameter with an approximate 1 mm thickness as seen in figure 1. Each sample was loaded once using the stepped loading input.

2.2. Experiment 2—plasticizer content

The plasticizer content was varied by using 1:4, 1:6, and 1:8 PVC to plasticizer ratios by weight and are named P4, P6, and P8 respectively. This range was chosen to display the high sensitivity of these sensors under low force applications. Three samples were fabricated for each plasticizer ratio and tested using a 1 mm s^{-1} load rate application with a 12 mm diameter and 1 mm approximate thickness.

2.3. Experiment 3—thickness

The thickness of gel samples was varied using 0.5, 1, 2.5, and 3.5 mm thick samples. These thicknesses were used due to limited ability to control thickness of samples. Using the current fabrication method, the thickness of samples is very difficult to control due to changes in solution strength when pouring solution in curing dishes and convective evaporation effects. Three samples were fabricated for each thickness resulting in a total of 12 samples. A loading rate of 1 mm s^{-1} load rate application was used for this experiment with 12 mm diameter samples.

2.4. Experiment 4—cross-section

The cross-section of gel samples was varied using 8, 12, and 16 mm diameters. These diameters were used to show the effects of changing the cross-section of the gel samples while still being able to cut the samples from the same respective curing dishes. Only two gel samples could be punched from a casting dish using the 16 mm diameter due to the physical size of the casting dish, but three sample were used for the remaining diameters. A 1 mm s^{-1} load rate application was used in this experiment with a 1 mm approximate thickness.

2.5. Additional work

Raw data was captured for a P4 gel sample with a smaller step input to the load application plunger for finer control of the loading application. When in close proximity to the surface of the gel, a very small tensile strain was induced in the gel causing partial contact with the surface of the gel. Very fine steps were used to capture the motion of the sample onto the plunger causing a tensile loading within the sample. The standard size step used in experiments was then applied to the gel to reach a neutral and compressive loading state. Hysteresis was also tested by increasing the stepped load on a gel sample, then decreasing the stepped load to observe any memory in the mechanoelectrical response. Multiple loading cycles were applied to the same gel sample to observe any memory effects.

3. Results and discussion

As seen in figure 3, these experiments aim to study the steady state response of the gel samples undergoing mechanoelectrical transduction while varying parameters to determine electrical response dependencies. The steady state values for both mechanoelectrical response and load were separately obtained using equation (1) to fit the transient data. Because of the decaying nature of this equation the c parameter was used to obtain these steady state estimations. The transient response was not studied directly, but qualitatively has a very fast response time and a slow decay to a steady state value. This response can be described by two separate characteristics. The *first* portion of the response is very fast, the phase lag is currently indistinguishable using the experimental sampling rate. This response is piezoelectric-like in that it is a quick response and may potentially decay under a stepped loading input. This

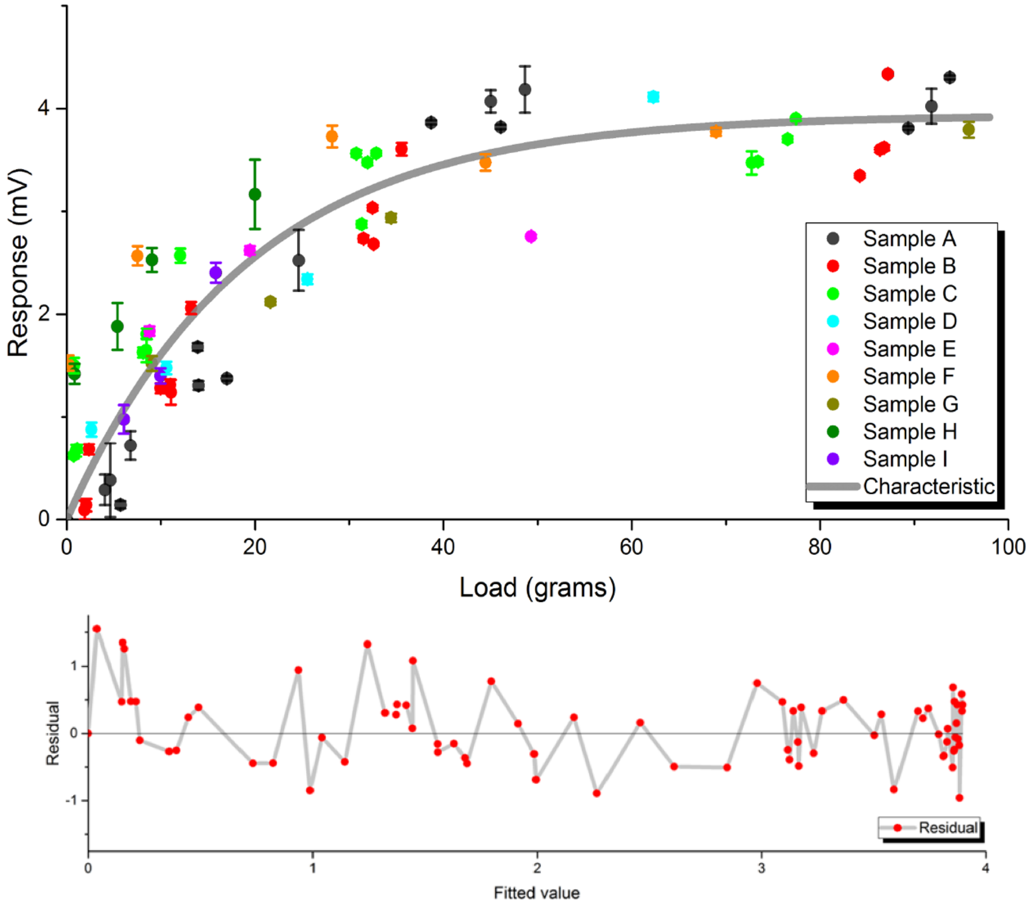

Figure 4. Experimental results from all P4 samples with 12 mm diameter and 1 mm thickness in mechanoelectrical experimentation with steady state estimations and fit characteristic curve (*top*) and residual plot showing goodness of fit of the characteristic curve (*bottom*).

may be due to the PVC lattice structure within the gel deforming causing electrostatic interactions. The *second* portion of the response is a slow relaxation, which also is shown in the load cell decay. This decay in load cell could be attributed to the viscoelasticity within the gel. The electrical response decay may be attributed to the relaxation of dipolar plasticizer molecules. The dipolar plasticizer molecules may polarize under the initial piezoelectric-like response, and once this initial field is dissipated the dipolar molecules will begin to relax but remain somewhat polarized due to the hysteresis of dipolar insulators.

First, a plot of all P4 samples with a 12 mm diameter were plotted using the estimated steady state response and load. The 95% confidence error bars are shown for the electrical response of the gels, which the majority of these errors are less than the height of the marker. All 95% confidence levels for the load were below an error of 0.5 g which is essentially the width of the markers used, so these error bars are not shown. To show the characteristic curve of the gel sensors with these properties where the characteristic function is a fit single exponential function that is forced through the origin (figure 4)

$$y = \alpha\left(1 - \mathrm{e}^{-\beta t}\right) \tag{2}$$

where model parameter α determines the total saturated output and β determines the rate of saturation from the mechanoelectrical response of the gel samples. Equation (2) was used in a nonlinear least squares regression to fit a curve with a coefficient of determination of 0.865, a plot of the residuals is also shown (figure 4). The coefficient of determination with the residual plot show a good fit to the data but is slightly skewed at very low fitted values. This may be due to initial stress within the gel when placed on the base electrode without applying a compressive load. This subtle initial stress within the material may cause a nonzero response before the plunger is lowered onto the gel sample. The gel samples show high sensitivity under very low load application and the response can potentially be linearized in this low force region (<20 g). The fit characteristic curve also captures saturation of the mechanoelectrical transduction within the gel samples at higher force inputs.

3.1. Experiment 1—strain rate

The strain rate application experiment shows very little change in mechanoelectrical transduction in the gel samples in terms of the steady state response (figure 5). This means that the steady state response is independent of strain rate in the experimented region, but the transient response will show

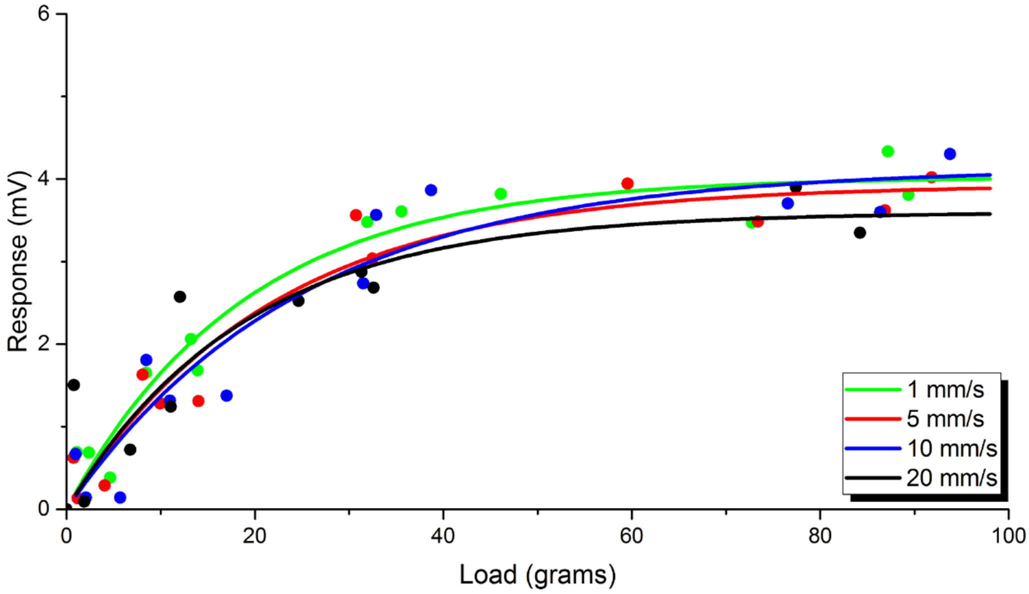

Figure 5. Experimental results for steady state signal dependencies when varying sample strain rate application on P4 samples with 12 mm diameter and 1 mm thickness.

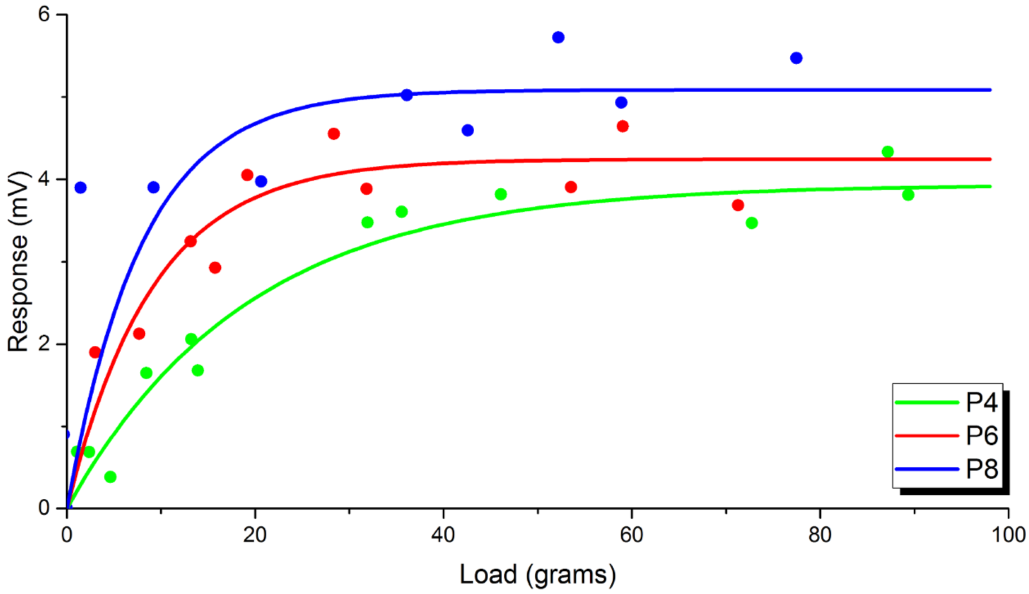

Figure 6. Experimental results for steady state signal dependencies when varying sample plasticizer content on samples with 12 mm diameter and 1 mm thickness.

noticeable differences. This characteristic curve shows the strain rate independence and strain dependence of the steady state mechanoelectrical response in the gel samples within the tested region.

3.2. Experiment 2—plasticizer content

As the plasticizer content increases, the overall magnitude of electrical response is increased including increased sensitivity in the low force region (figure 6). This could be attributed to the amount of polar plasticizer molecules having some dipolar interactions within the material. In the low force region, the P8 samples have approximately twice the response of the P4 samples, which directly relates the amount of plasticizer to the mechanoelectrical response. The P8 samples saturate at a lower force input, which may be due to the decreased in modulus and stiffness of the material. This shows the potential of tuning the sensitivity of the gel samples to a desired load input to allow for an optimal mechanoelectrical response over some range of force inputs. Gels with higher plasticizer content are more sensitive and are more suited for lower force inputs, gels with lower plasticizer content have a higher modulus and are more suited towards higher force inputs.

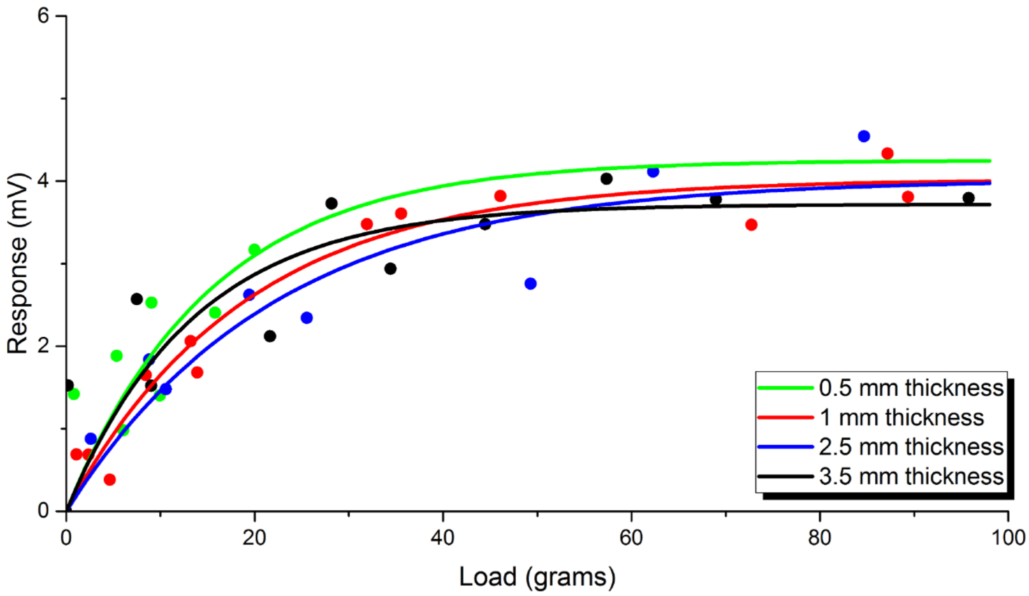

Figure 7. Experimental results for steady state signal dependencies when varying sample thickness on P4 samples with 12 mm diameter.

3.3. Experiment 3—thickness

The experimental results from the variation in thickness shows a minimal effect of thickness of the gel sample under the tested loading conditions (figure 7). This insinuates the mechanoelectrical response may be a surface property of the gel. By increasing the thickness, no apparent effect is seen in the steady state response of the gel samples in this experimental test region. The initial load application may cause an initial response that is concentrated near the surface of the gel. Due to the insulation properties of the gel, only plasticizer in a small region near the surface of the gel may polarize under the surface-dominated initial response.

3.4. Experiment 4—cross-section

As the cross-section of the gel samples increased, the total response of the gel also increased (figure 8). This result, in conjunction with Experiment 3 results, support the theory of mechanoelectrical transduction being a surface property of PVC gels. The volume of the gel sample is changing in both experiments, while the mechanoelectrical response seems only dependent on the cross-section of the sample and not the thickness. The results from the variation in cross-section were also normalized to give the total output per area. This normalization was done with respect to the undeformed cross-section. Experimentation was completed showing no significant change in cross-section at the fully loaded state. The ratio of area to circumference of each sample is changing, while the normalized results show relatively close output per area meaning the results show that edge effects are minimal in the gel samples. The small cross-sectional area gel samples show saturation of response at a lower loading input.

3.5. Additional work

When the plunger of the force application mechanism is moved incrementally onto to surface of the gel in very fine steps, the gel shows a creeping motion up onto the plunger. This is attributed to a combination of surface tension effects and imperfection in the gel surface. When the plunger first contacts the surface of the sample it is only partially contacting at the peaks of the uneven gel surface, and the adhesive forces pull more gel material onto the plunger. These adhesive forces could be attributed to the surface tension of the gel, so while the plunger is partially contacting the gel there is a creeping motion of the gel onto the plunger causing a tensile load within the gel. This creeping deformation onto the plunger induces tension in the gel and hence a tensile load is registered on the load cell. This is seen as a polarity inversion, relative to a gel experiencing compressive loads, in the mechanoelectrical response (figure 9). At a critical point, the gel suddenly makes full contact with the plunger causing a large tensile load in the gel. This large transient response seems to decay to a similar voltage than the comparable compressive load, except with opposing polarity. This may suggest that there is a memory effect in the transient response, but the steady state response may be independent. The plunger is then incrementally lowered eventually reaching a neutral point assumingly with no tensile or compressive load. At this neutral point there is no mechanoelectrical response because the material is neither in tension nor compression. The plunger is continually lowered reaching the compressive loading regime. In this compressive loading regime, the mechanoelectrical response of the gel is the opposite polarity of the tensile loading regime.

The plunger was incrementally lowered onto the sample to apply increasingly stepped loading as in the prior experiments, then was incrementally raised to observe any hysteresis or memory effects in the steady state mechanoelectrical response.

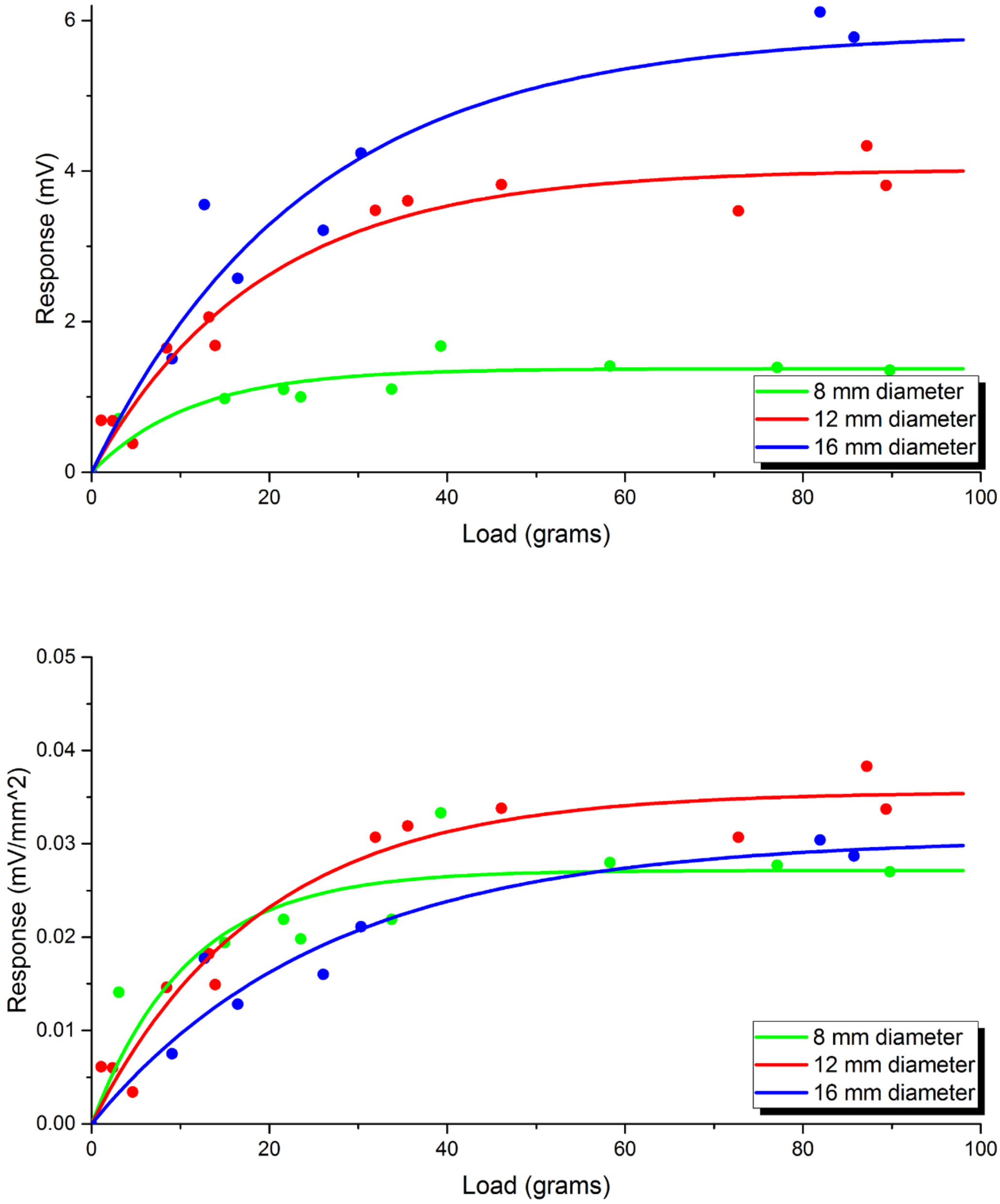

Figure 8. Experimental results for steady state signal dependencies when varying sample cross-section (*top*) and normalized output per cross-sectional area (*bottom*) on P4 samples with 1 mm thickness.

There seems to be very little hysteresis in the mechanoelectrical response in the tested loading region (figure 10). This may be related to the mobility of the dipolar plasticizer within the material, because of the high mobility of plasticizer within the material there is little to no memory effect in mechanoelectrical response.

The transient response seen in raw data shows a combination of two responses, one being a very fast response that is currently indistinguishable in terms of phase delay, and a secondary response being a slow relaxation. The proposed mechanoelectrical framework (figure 11) includes a combination of two responses. The initial response, which is surface dominated, may stem from electrostatic interactions at the surface of the gel when undergoing mechanoelectrical transduction. As a result of variation of thickness and cross-section of the samples, the mechanoelectrical response seems to be a surface dominated property under these loading conditions. The gels contain a PVC lattice structure that are made of polar polymer chains that may generate an electric field upon initial deformation near the surface due to electrostatic interactions of these polar polymeric chains. This initial field may be piezoelectric-like and decay with a stepped input. This initial response may also stem from the adsorption and polarization of free plasticizer within the gel near the contacting electrode surface. The secondary response may stem from the dipolar interactions of the free plasticizer content. The DBA in the gel sits interstitially and will polarize in the presence of an electric field. Once the initial electric field has decayed and applied

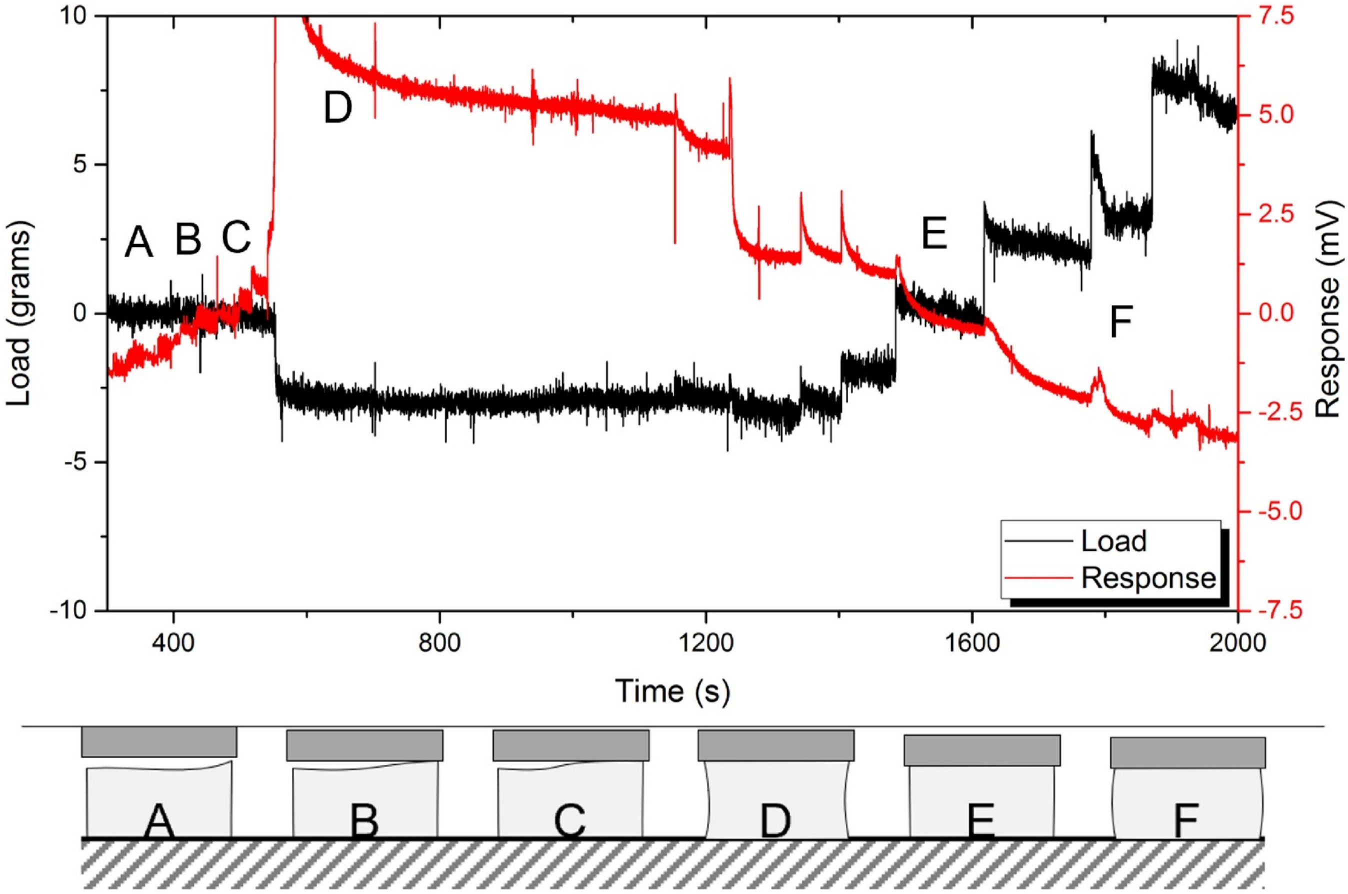

Figure 9. Experimental results exhibiting uneven surface phenomena when partially contacting surface (*A, B*, and *C*), gel rapidly creeping onto plunger resulting in overall tension (*D*), reaching a neutral position (*E*), and reaching overall compressive loading.

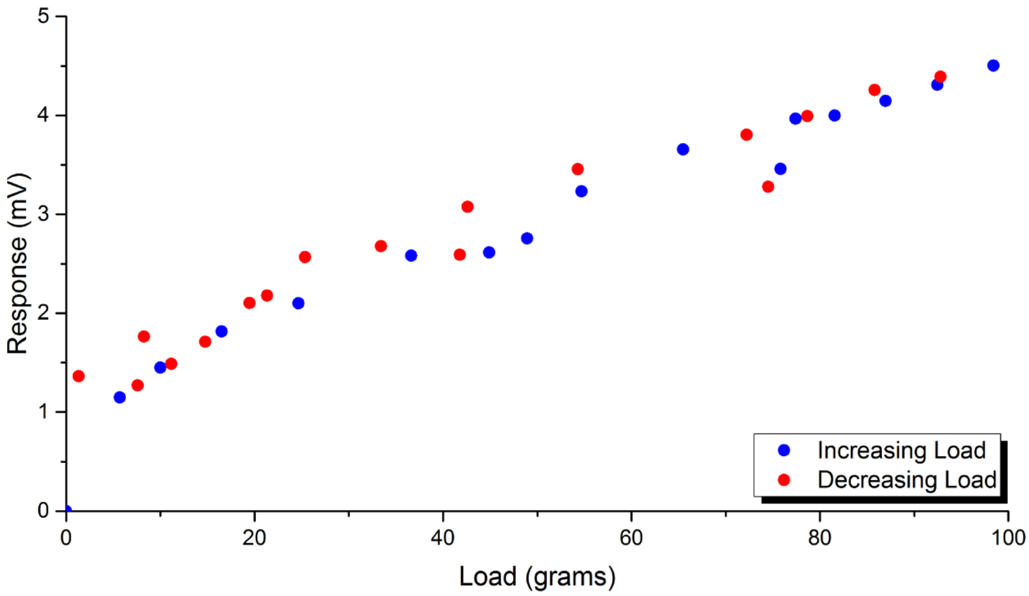

Figure 10. Experimental results showing minimal memory effect in the steady state mechanoelectrical response under increasing (*blue*) and decreasing (*red*) stepped loading.

load is held constant, the DBA molecules will observe hysteresis due to the dipolar nature and will remain somewhat polarized leaving a non-zero steady state response to the stepped input as seen in experiments. This decay is a much slower response and may explain the slow decay to steady state seen in experimental work.

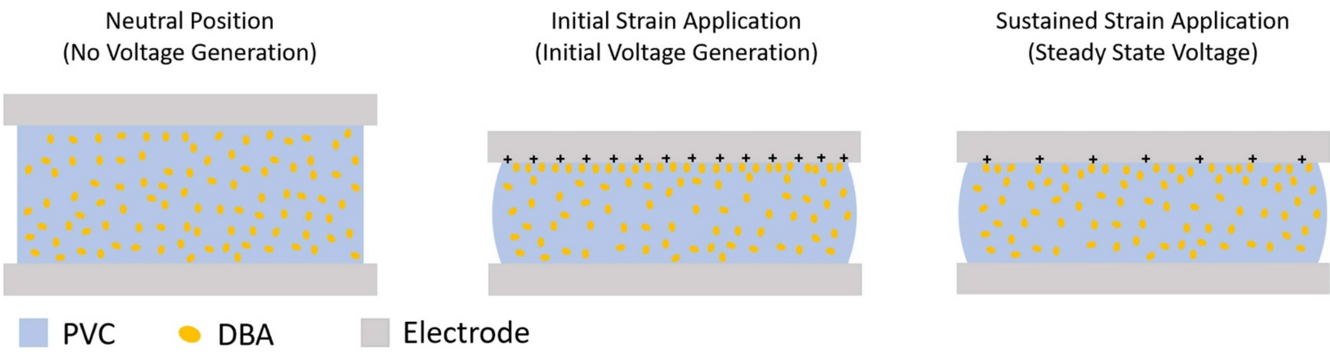

Figure 11. Illustration of proposed mechanoelectrical transduction mechanism from left to right starting in a neutral position with random DBA orientation and no voltage response (*left*), DBA polarization under applied strain with associated large initial voltage generation (*center*), and DBA dipolar relaxation under constant sustained strain application causing response decay to steady state voltage (*right*).

4. Conclusion

A fundamental study on mechanoelectrical behavior in PVC gels was conducted to display mechanoelectrical properties of PVC gels. Using a stepped loading input multiple gels were tested to determine the mechanoelectrical transduction dependencies. The steady state response of the gels was shown to be strain rate independent. A higher plasticizer content resulted in a larger mechanoelectrical response from the gels including higher sensitivity in the low force region. This is potentially useful for tuning PVC gel sensors to specific force inputs. Under lower force inputs a higher plasticizer content would be desirable due to lower material modulus and increased sensitivity. In these experiments it was found that the mechanoelectrical transduction in the gel samples was a surface dominated property. This is deduced from results of varying sample thickness and cross-section. Also, some interesting surface properties were examined showing a polarity inversion in the electrical response for tensile and compressive loading on PVC gels. Hysteresis was also briefly investigated showing minimal memory effects in steady state mechanoelectrical response to stepped loading cycles in the experimented region. This is a fundamental study aimed to broaden the applications of PVC gel sensors and future work will focus on dynamic sensing, modified materials, and electrode configurations to improve performance and applicability for these sensors.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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Author contributions

J N and T H fabricated all testing samples. J N, Z O, Z F, and K J K designed and performed experiments. J N, Z O, and K J K analyzed data. J N, Z O, and Z F prepared all figures. The manuscript was prepared by J N and K J K with help from Z O and Z F. All authors reviewed the manuscript.

Conflict of interest

The authors declare no competing interest.

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