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## **Field-Structured Composite Studies**

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## Abstract

Field-structured composites (FSCs) were produced by hosting micron-sized gold-coated nickel particles in a pre-polymer and allowing the mixture to cure in a magnetic field environment. The feasibility of controlling a composite's electrical conductivity using feedback control applied to the field coils was investigated. It was discovered that conductivity in FSCs is primarily determined by stresses in the polymer host matrix due to cure shrinkage. Thus, in cases where the structuring field was uniform and unidirectional so as to produce chainlike structures in the composite, no electrical conductivity was measured until well after the structuring field was turned off at the gel point. In situations where complex, rotating fields were used to generate complex, three-dimensional structures in a composite, very small, but measurable, conductivity was observed prior to the gel point. Responsive, sensitive prototype chemical sensors were developed based on this technology with initial tests showing very promising results.



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# Introduction

Field structured composites (FSCs) are anisotropic particle composites produced in a magnetic or electric field. Because the work described herein deals only with FSCs produced in magnetic fields, the discussion is limited to magnetically structured samples. Samples are prepared by hosting magnetic particles (e.g. Ni, Fe, or Co) in a liquid monomer and polymerizing the mixture in a magnetic field. When a sample is placed in a static, uniform field, chains form along the field lines as a result of induced dipolar forces between the magnetized metal particles. If, instead of a static field, an oscillating field is used, the same result is obtained. Inverting the field direction periodically has no net effect on the process because the force between particles is independent of the field direction's sign. If a slowly rotating, uniform magnetic field is applied to the sample, the resulting particle chains rotate in the fluid. However, the linear chains distort and then begin to break up as the frequency of rotation is increased due to hydrodynamic drag. Sheets form in the plane of the rotating field at frequencies of a few hundred Hertz and higher. Thus, uniform uniaxial or rotating biaxial fields may be used to produce FSCs exhibiting chain or sheet-like structures.

Suppose that a uniform uniaxial field is applied to a sample simultaneously with a rotating biaxial field in such a way that the uniaxial field is orthogonal to the plane of the rotating field. Assume further that the uniaxial field strength is initially zero and that it is slowly increased. Under these conditions, one correctly expects the uniaxial field to begin to interrupt the sheet structures as its strength reaches a value commensurate with that of the rotating field. At very large relative strengths, the uniaxial field completely dominates the rotating field and chains form. Interestingly, when the uniaxial field strength is exactly twice the strength of the rotating field, the two cancel and no net first order interaction between particles is expected. [1]

FSCs can exhibit highly anisotropic properties. For example, an FSC composed of linear chains in a non-conducting polymer matrix might show excellent electrical conductivity along the direction parallel to the chains but poor conductivity in directions orthogonal to this. Furthermore, because the conductivity depends on the metal particles contacting one another, it is sensitive to mechanical strain parallel to the chain axes as well as expansion of the sample through swelling or heating. [2]

As originally envisioned, the main thrust of this work was to build a triaxial field device and use feedback to control electrical conductivity in FSCs. The idea was to control about the null point where the effects of an applied uniaxial field are cancelled by a rotating biaxial field. By measuring conductivity through the sample and feeding this back to the uniaxial field amplifier, it was conjectured that the system could be biased toward chain formation by just the right amount to produce material with the reference conductivity.

Upon assembling a resonant, triaxial Helmholtz coil system and using it to produce FSCs under a variety of field conditions, we discovered that the combination of uniaxial and biaxial fields created complex particle structures under balanced field conditions

where we originally thought the interactions should cancel. These structures resulted from non-vanishing induced-dipole/induced-dipole interactions between the particles, a subject treated in a recent paper. [1] Although this direct attempt at feedback control produced unexpected effects, it resulted in a means of creating a large variety of particle agglomerates, from simple particle chains and sheets, to particle foams and honeycomb structures. Experiments on particle suspensions showed that some of these complex particle agglomerates had conductivities much larger than simple chains. Thus, by manipulating structure, conductivity of the final composite might be controlled.

Because structural control must occur during the pre-gel phase of polymerization, it is important to characterize the relationship between the pre-gel conductivity and the conductivity of the fully cured composite. Thus, a major part of the work described herein focuses on characterizing the evolution of the composite conductivity during polymerization. The results show that the final conductivity is nearly completely determined by stresses accompanying cure shrinkage, eliminating any possibility of using feedback methods to actively control final properties.

Given the overall goal of controlling properties through structure, open-loop control must be established if closed-loop feedback cannot be used. This requires manipulating the pre-gel process to consistently produce material with the target properties. In other words, mixtures of the same concentration processed under identical laboratory conditions (temperature, catalyst concentration, gas content of the sample, magnetic environment) must produce composites with identical properties. This is possible only if all the relevant process variables are accessible and can be controlled to the necessary level of accuracy. This turned out to be problematic.

A major source of variability in this system is particle-to-particle contact resistance. Early in this work it was discovered that consistently good samples were difficult to make. This was attributed to corrosion of the nickel particles by atmospheric oxygen, which served to degrade conductivity. The particles were coated with gold to passivate them toward oxygen attack, however, this proved only partially effective because of incomplete coverage. With careful pre-treatment, samples with relatively consistent properties could be produced, but these degraded within days after exposure to air. A new method of coating was sought that ensured complete coverage with non-porous gold. Two synthesis routes were tried. The first involved coating microscopic PMMA beads with low sulfur (magnetic) nickel followed by electroless gold coating. The second method involved directly coating microscopic nickel particles. The first method proved difficult because of problems with adherence between the nickel coating and the PMMA surface and relatively weak magnetism in the fully coated particles. The second method was successful, yielding uniformly coated particles with properties that remain stable indefinitely.

Using the new coating technique, stable, electrically conductive FSCs could be routinely produced in the laboratory. These materials were shown to have conductivities that were extremely sensitive to strain, temperature, and chemical and magnetic environment, showing great promise as ultra-sensitive sensors. Several prototype sensors



(chemi-resistors) were produced and tested. The results of these initial tests are very encouraging.

The next section of this report describes the experimental techniques and procedures used to produce the FSC materials. This includes descriptions of the triaxial field apparatus, conductivity test circuit, and electroless gold coating procedure. Results from the conductivity and sensor tests are then presented and then discussed. The final section presents conclusions.

## Experimental Procedures

### Resonant, triaxial Helmholtz coil apparatus

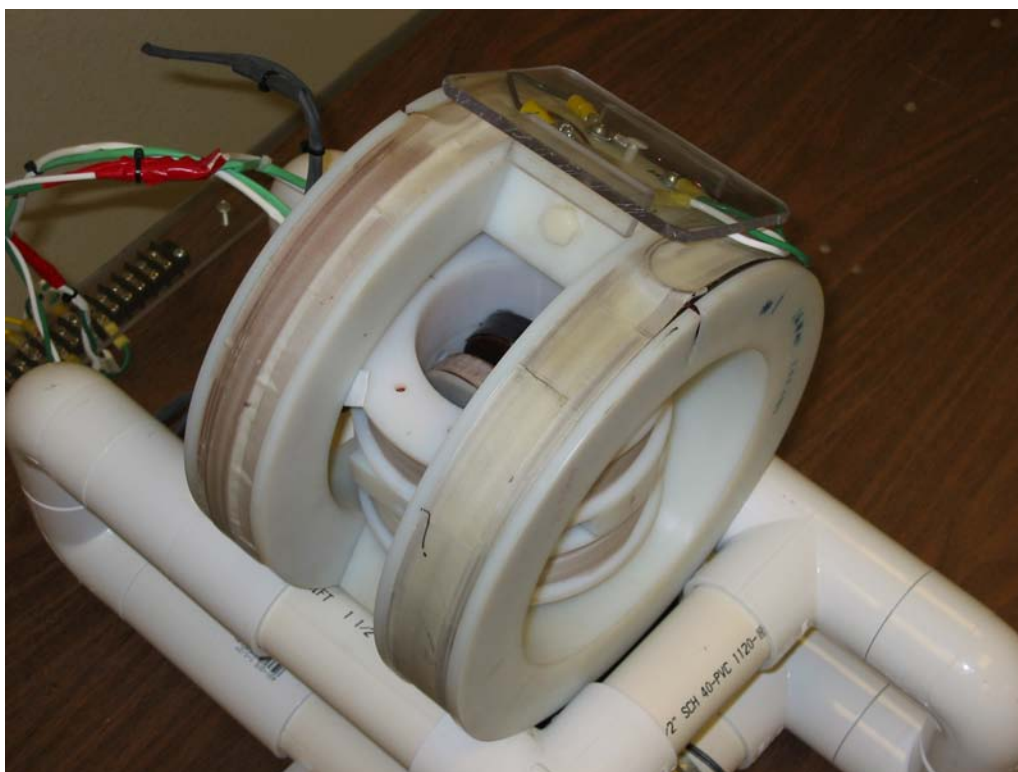
To create a uniform, triaxial magnetic field we constructed three nested, orthogonal Helmholtz coil pairs (Figure 1). The coil properties are listed in Table 1. Although all three fields are equivalent, it is useful to think of one component as the uniaxial field and the other two as forming a biaxial field. The uniaxial field is produced either by applying dc current to one set of coils or applying ac current with a fixed capacitor in series with the large coil pair to create a series resonance at 203.7 Hz with an impedance of ~2.6 ohm.

**Table 1. Triaxial coil properties.**

	Inner Coil Pair	Middle Coil Pair	Outer Coil Pair
Turns/coil	252	299	300
Series Resistance	2.44 ohms	2.24 ohms	3.00 ohms
Inductance@100Hz	8.5 mH	25.0 mH	26.4 mH
dc Response	87.7 G/A	44.6 G/A	23 G/A

The ac biaxial field components are produced by connecting two coil sets to tunable, computer-controlled, series-parallel capacitor banks to create series LRC circuits with high quality factors and a resonant impedance of ~2.2 ohm. [3] The capacitor banks use high quality Silicon-Controlled Rectifier capacitors from General Electric that have high current and voltage specifications, and low stray inductance. The banks use 12 capacitors each, covering a range in capacitance of 1–100  $\mu$ F, and in combination can produce ~354,000 capacitance values spanning three decades of capacitance, enough to create series resonance at selected coil frequencies from ~125-1500 Hz. Potter and Brumfield power relays with a 4 kV standoff are used to switch the capacitors and the 24 V switching voltage is supplied through a control board based on 4 kV standoff optical isolation relays. The relays are driven by logic pulses from a 96-channel I/O board (National Instruments) installed in a Power Macintosh computer. Software was

developed using the LabVIEW™ programming language (National Instruments). Extremely accurate calibration of the capacitors is essential, and was accomplished with an Agilent Mo. 4284A LCR bridge. Driving these circuits with an ATI model 1504 audio power amplifier, it is possible to create magnetic induction fields as large as 500 G at frequencies up to ~1500 Hz in the smallest coil. Amplifier input signals are provided by phase-locked multifunction synthesizers (Agilent, Mo. 8904A).



**Figure 1. Photograph of the triaxial field device mounted in its cradle.**

### **Procedure of coating microscopic Ni particles with gold**

Nickel particles (Goodfellow Cambridge Ltd.) in the size range of 4-7 microns were coated with gold to passivate them against oxidation and ensure stable conduction. Particles were coated in 1.0 g batches using chemicals supplied by Enthone Inc. (West Haven, CT). 100 mL of coating solution was made by dissolving 0.59 g Lectroless Prep Unit A ( $\text{KAu}(\text{CN})_2$ ) and 5.3 g Lectroless Prep Unit B (EDTA, citric acid, and proprietary ammonium compounds) in deionized water, and adjusting the pH to 4.0 by adding 20 wt.% KOH. The solution was then heated to 85 °C and 1.0 g Ni powder (untreated) added with constant stirring. Note that special attention must be given to setting up the stirring so that particles do not collect in eddy currents at the bottom of the reaction vessel and “clump” together. The self-limiting reaction requires approximately 12 minutes to reach

completion at 85 °C. Small amounts of heated deionized water were used to periodically replenish evaporation losses. After coating, the particles are filtered and washed. Microscopic inspection of the resulting particles revealed uniformly coated, bright yellow particles that tended to exist in small clumps consisting of several particles. The clumps do not form during the coating procedure but are present in the nickel powder as received from Goodfellow. The coating is soft and can be deformed by crushing the particles with a lab spoon.

## Procedure for conductivity tests

Current flow through the sample was measured during polymerization of the composite using a picoammeter (Keithley Mo. 485, Keithley Instruments Inc., Cleveland, OH). Coated nickel particles were hosted in either a polyester/styrene blend to form glassy composites or silicone polymer to form rubbery composites. Pre-polymer/particle mixtures were made at a concentration of 6.8 vol. percent. Each suspension was degassed prior to being loaded into a sample cell of 1 cm square cross section. Two opposing sides of the cell were lined with copper tape to serve as electrodes. The cell was then placed in the triaxial field device with the electrodes oriented so that particle chains/structures would bridge the electrodes when formed. Wires attached to the electrodes were twisted and oriented so as to minimize field-induced currents. All connections between the sample and circuit were soldered to minimize contact resistances.

A simple voltage divider circuit was designed to provide ~1 mV across the sample electrodes. The sample was placed in parallel with a 0.1 ohm resistor, this parallel circuit element being in series with a 146 ohm resistor and a ~1.5 V battery. The picoammeter was placed in the measurement branch of the circuit in series with the sample. The measurement circuit is shown in Figure 2. Analysis of this circuit yielded the following expression for the sample resistance,  $R_s$ , in terms of the measured current,  $I_s$ :

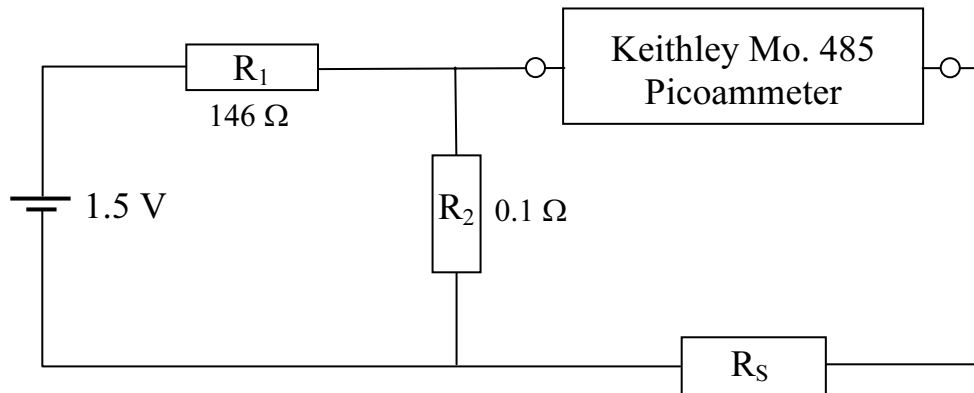
$$R_s = \frac{(V_{bat} - I_s R_1) R_2}{I_s (R_1 + R_2)} \quad (1)$$

where  $V_{bat}$  is the battery voltage,  $R_1$  is 146 ohm, and  $R_2$  is 0.1 ohm. Note that this leads to  $I_s \approx 10^{-3}/R_s$  when  $R_s$  is very large, and that  $I_s$  saturates at  $\sim 10^{-2}$  A in the limit  $R_s \ll R_2$ .

After a sample was placed in the field apparatus and the leads soldered to the measurement circuit, the structuring magnetic field was applied to the sample. In the case of the silicone polymer, 5 amperes of dc current were applied to a single Helmholtz coil pair to produce a 325 gauss uniaxial, dc field in the region of the sample across the electrodes. The coil was housed in an oven and the temperature held at 55 °C to reduce the cure time of this system to ~2 hours (room temperature requires ~24 hours). The polyester/styrene-based composites were cured at room temperature either in an

oscillating uniaxial field (150 gauss rms field strength) or in a heterodyned triaxial field. An inductor was used to filter unwanted induction currents from the ac field components.

Sample current,  $I_S$ , was measured as soon as the structuring field was turned on. Data were collected over an IEEE 488 interface using a PC equipped with a National Instruments PCI-GPIB card (National Instruments). After the data were collected, the current data were converted to resistance using Eq. (1) and the conductivity obtained from the sample dimensions.

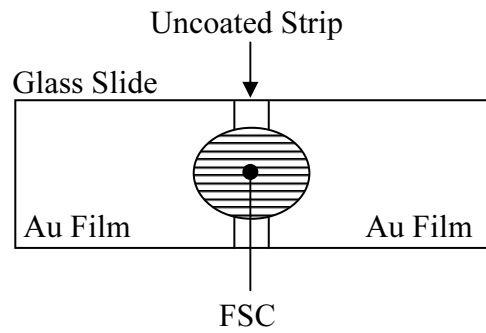


**Figure 2. Test circuit for measuring the current through a FSC sample.**

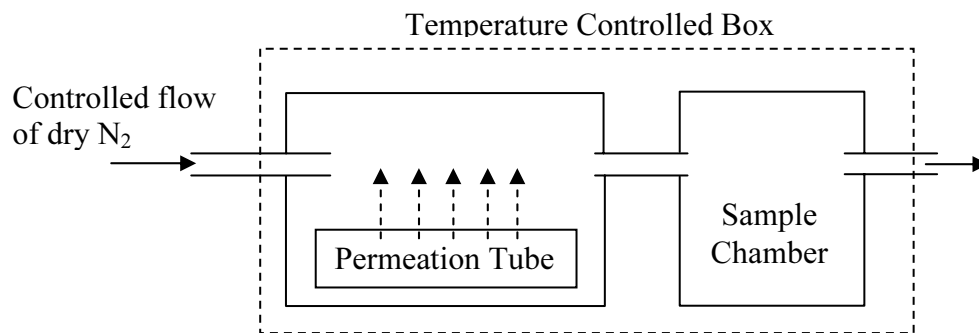
## Prototype sensor tests

Prototype sensors were prepared by placing a drop of pre-polymer/particle mixture (6.8 vol. percent) on a substrate consisting of a glass slide onto which two gold strips had been sputtered (Figure 3). The drop is placed so that it spans the non-conductive space between the gold strips. The sample was then placed on top of two spaced, planar permanent magnets and allowed to cure in the edge field. This configuration was chosen so that the field lines would “arch” through the sample leading to arched particle chains connecting the sensor electrodes. Qualitative tests were performed by connecting leads to the sensor electrodes and measuring the resistance in a saturated vapor of pentane or toluene at room temperature. These simple tests were repeated over an extended period of time to confirm that sensors were stable and recovered after use.

Measurements were also made by placing a sensor in a flow chamber and exposing it to known partial pressures of pentane while measuring the current flow using the test circuit described above. A schematic depiction of the flow chamber is shown in Figure 4.



**Figure 3. A schematic depiction of a prototype sensor.**

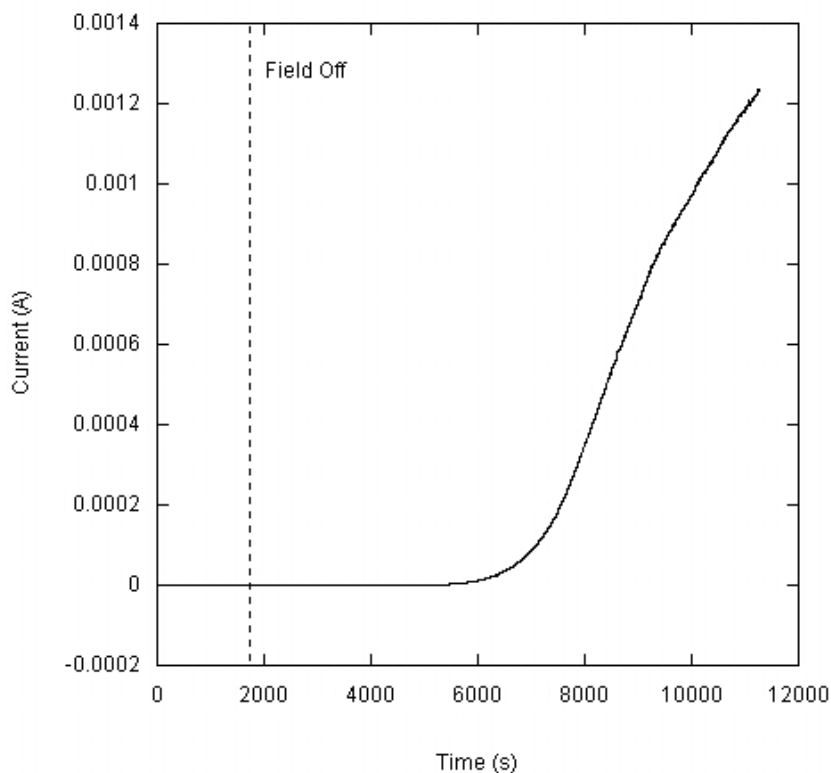


**Figure 4. Schematic depiction of the flow chamber apparatus used for sensor tests.**

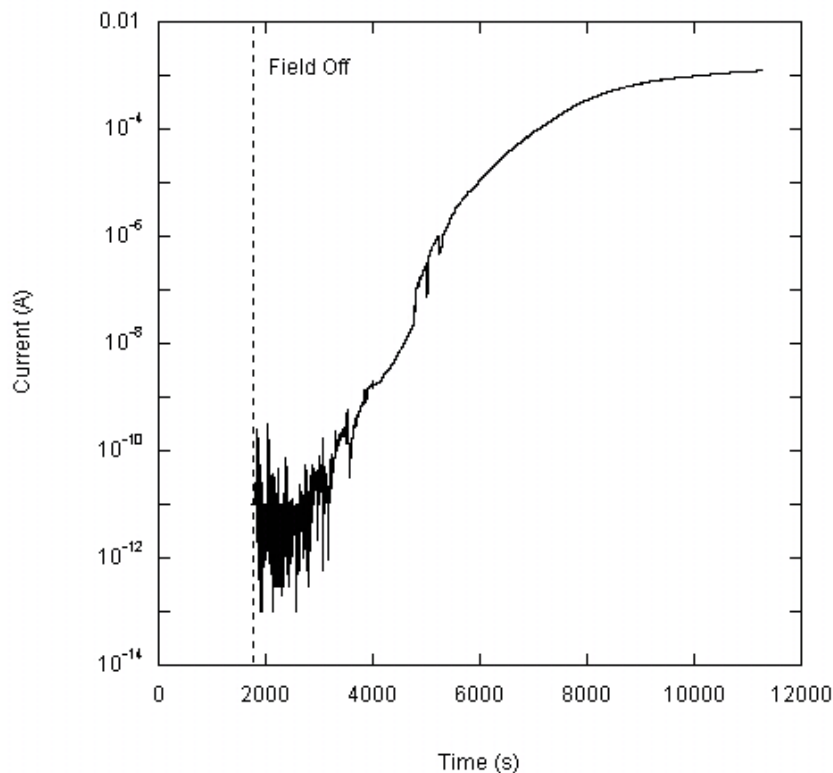
# Results

## Conductivity test results

The evolution of the conductivity of 6.8 vol.% particles in a dc uniaxial field of 150 G is shown in Figure 5 for the polyester/styrene polymer cured at 23 °C. At  $t=1740$  s the polymer reached the gel point and the magnetic field was turned off, the chains now trapped by the gel. At this time, the measured current was beneath the resolution of the picoammeter, so turning off the magnetic field had no measurable effect. In fact, there is no measurable conductivity until well beyond the gel point. At  $\sim 6000$  s, a smooth increase in the conductivity can be observed on a linear scale. A logarithmic plot of the conductivity (Figure 6) shows that after  $\sim 3000$  s the conductivity becomes measurable with the picoammeter, increasing about eight decades at the final fully cured state. Experiments showed that final conductivity is unaffected by turning off the field at or after the gel point.

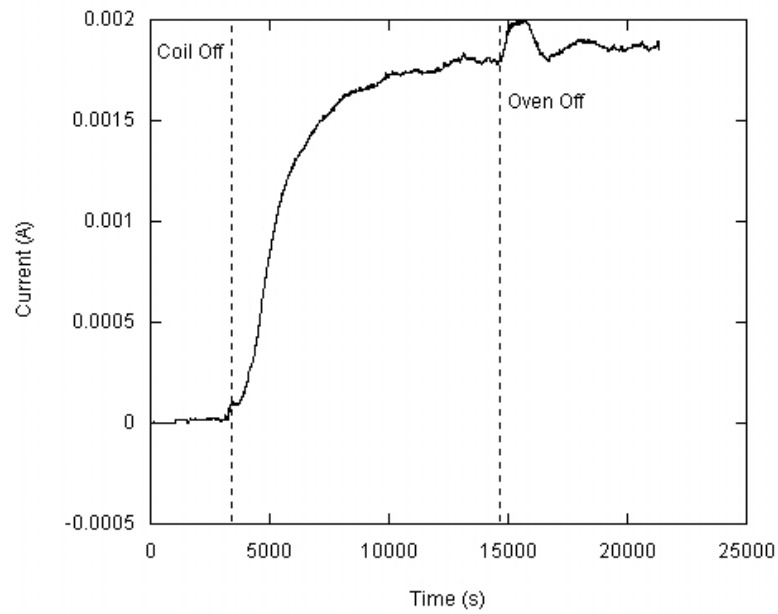


**Figure 5. A plot showing the evolution of the conductivity of 6.8 vol.% Ni particles hosted in polyester/styrene during the curing process.**

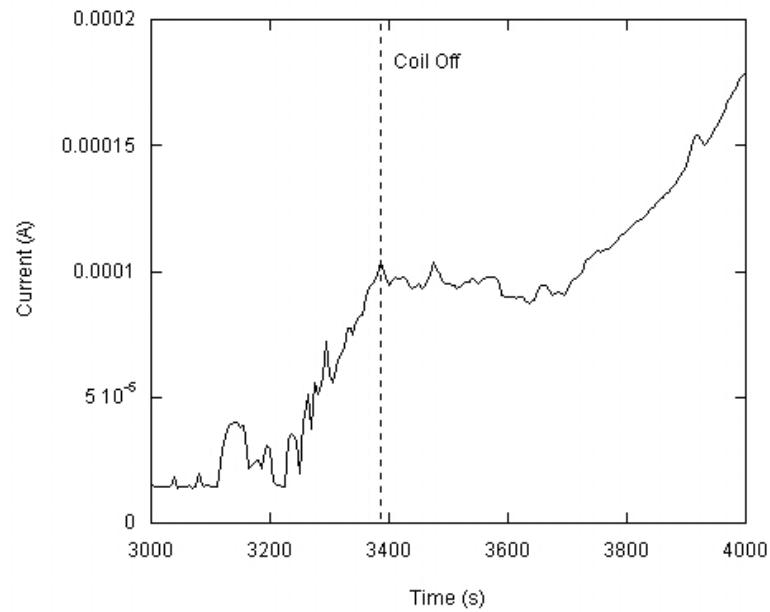


**Figure 6. A semi-logarithmic plot of the same data shown in Figure 5.**

Similar experiments to those just described were conducted using a soft silicone polymer, with the results shown in Figure 7. In this case, appreciable conduction was observed at the gel point, and a slight decrease occurred when the field was turned off (Figure 8). The final conductivity is quite similar to that of the polyester/styrene composite, supporting the conclusion that cure-induced stresses are the mechanism by which conduction evolves.



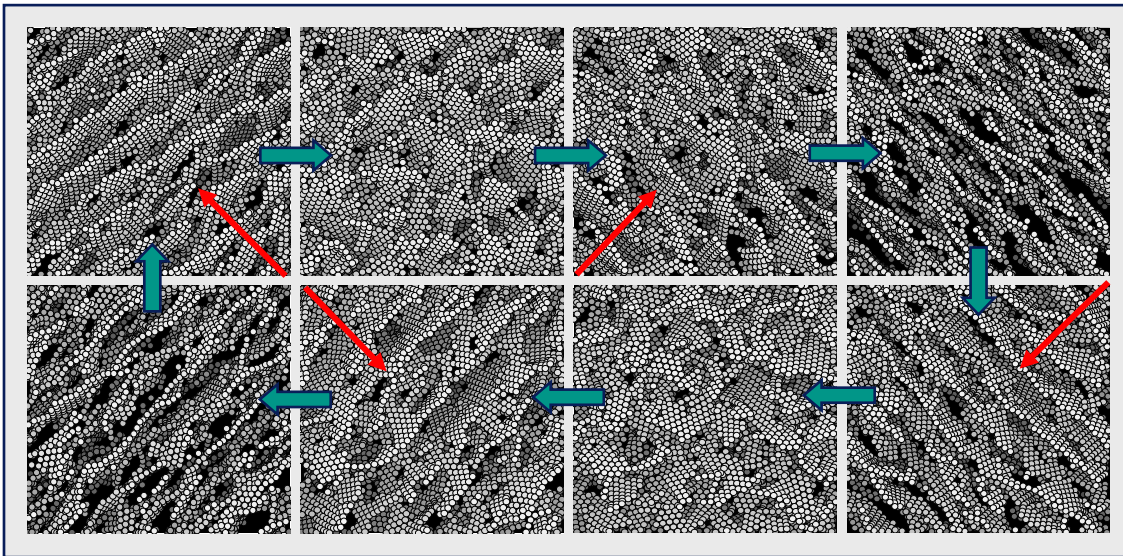
**Figure 7. Current conducted through a silicone polymer based FSC.**



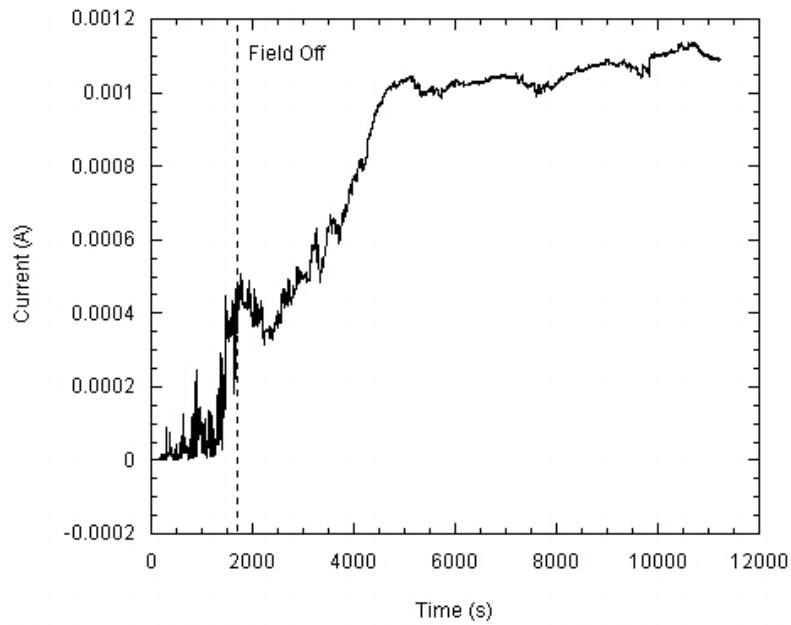
**Figure 8. Expanded view of the data plotted in Figure 7 around the region where the field was turned off.**



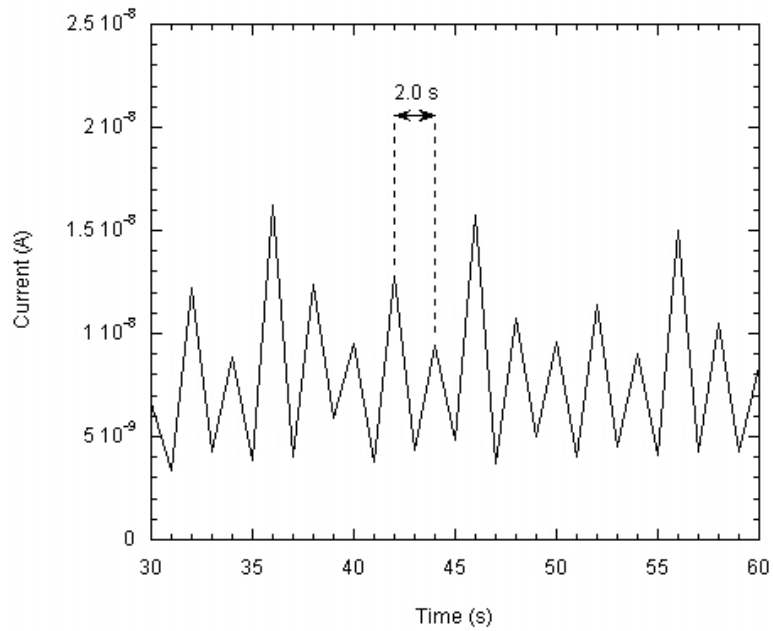
Finally, we subjected a 6.8 vol.% polyester/styrene suspension to a three-dimensional heterodyned magnetic field, in this case by setting the coil frequencies to 203.3, 203.7, and 204.2 Hz. This gives heterodyne beat frequencies of 0.4 and 0.5 Hz, for a field cycle of 10 s. The dynamics of the particle suspension are quite complex: sheets form in the suspension and cycle through a variety of orientations, eventually visiting all of the body normals to a cube whose faces are normal to each of the field components, as the simulations in Figure 9 indicate. These structures have appreciable conductivity before the gel point, as the data in Figure 10 show, probably because of the high level of connectivity in the sheet-like structures within the composite. Note from the early time data shown in Figure 11 that the baseline conductivity oscillates with a period equal to that of the heterodyned field. At the gel point, the conductivity is essentially 8 decades greater than that of the composite subjected to a uniaxial field, yet the final conductivity grows only to a value comparable to that of the uniaxial field sample.



**Figure 9. Simulation results showing the complex nature of FSCs produced in a heterodyned triaxial magnetic field environment.**



**Figure 10. Current history during sample curing for an FSC produced in a heterodyned triaxial field.**



**Figure 11. Early time baseline data from Figure 10 showing 2.0 s current oscillations.**

## Prototype sensor test results

Data from the sensor test are shown in Figures 12-14. It is clear from the data that baseline current through the prototype sensor drifted from  $\sim 26.5$  to  $\sim 29.3$   $\mu\text{A}$ . Temperature in the test chamber was held at 298 K throughout the test, so the baseline drift is not temperature induced. However, it was observed that the baseline began to drift only after the sensor was first exposed to the chemical flow. The origin of this positive drift is unknown. One would naturally assume that chemical contamination of the sensor should produce a negative drift due to swelling of the polymer, but this was not observed.

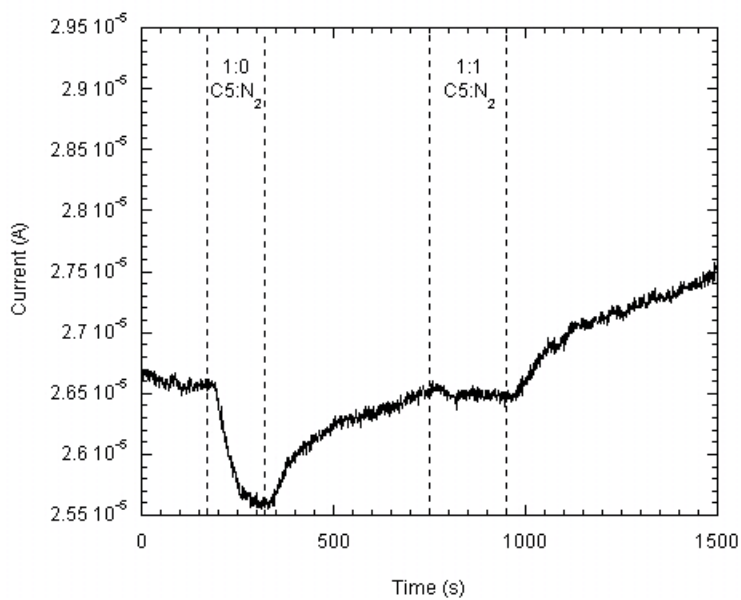
An objective of these tests was to determine approximately how much time was required for the sensor to respond to, and recover from, exposure to pentane flows at concentrations of  $6.1$   $\mu\text{g/mL}$  (no  $\text{N}_2$  dilution stream) and  $3.05$   $\mu\text{g/mL}$  (1-to-1 dilution with dry  $\text{N}_2$ ). The concentrated pentane flow was first introduced to the sensor at around  $t=171$  s, and the response in the current was observed  $\sim 20$  s later (Figure 12). The flow was shut off at  $\sim 321$  s and, again, the current response lagged by  $\sim 20$  s. Similar behavior was observed in response to the dilute flow initiated at  $t=750$  s.

Tests performed by simply placing a prototype sensor in a saturated atmosphere of toluene or pentane caused the sensor current to change by many orders of magnitude, as shown in Figure 15. This switch-like behavior is undesirable. Eventually, one would like to have the capability of determining the concentration of the chemical flow from the magnitude of the response. However, the data in Figures 12-14 clearly show that the response to the dilute steam is not as large as the response to the concentrated stream in all three tests. This is an encouraging result, indicating that it might be possible to construct a sensor capable of yielding quantitative information. On the other hand, it is also apparent from the data that the response to the concentrated stream falls off with time (note that the plots in Figures 12-14 are all plotted with the same vertical scale).

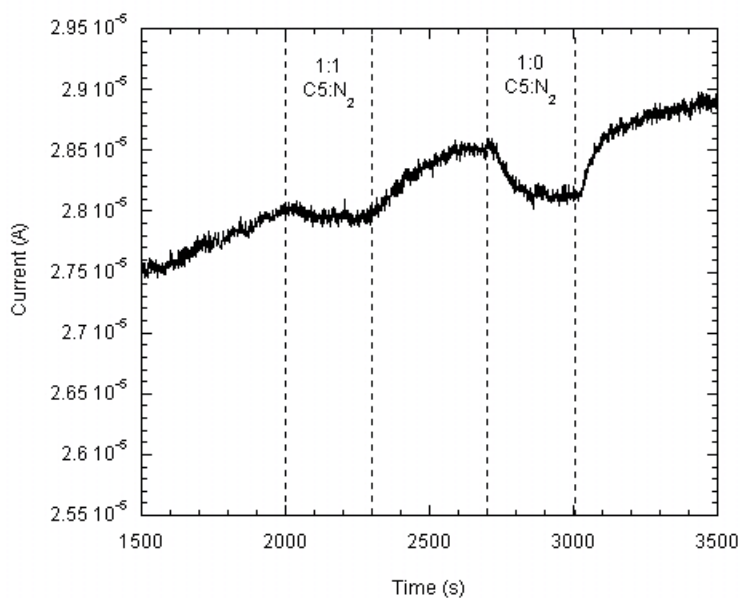
This was a preliminary test to demonstrate a concept. Further testing to investigate sensor recovery dynamics were not performed.

## Discussion

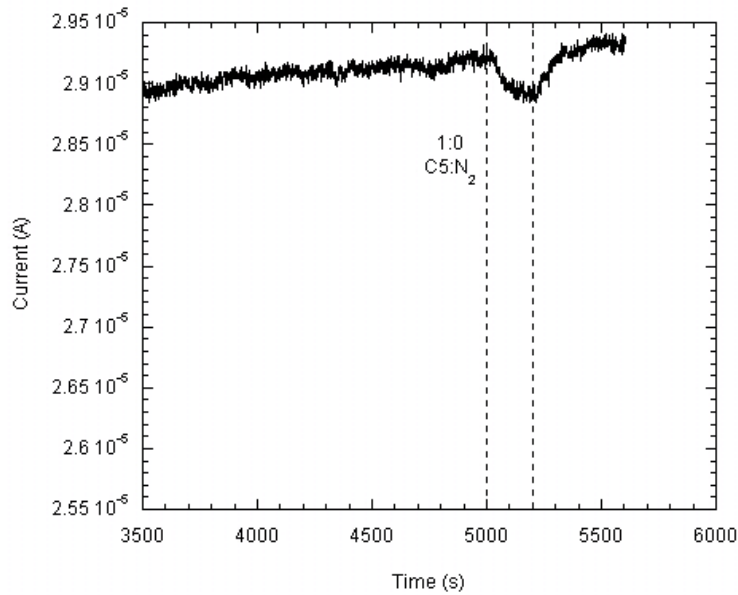
Two points are worth mentioning with respect to the conductivity tests. First, the formation of chains in the polymer before the gel point does not lead to a state with measurable conductivity, probably because the dipolar interactions between particles are too small to generate sufficient contact pressure to create appreciable current conduction capability. Second, experiments show that turning off the field at or beyond the gel point does not affect the ultimate conductivity. The evolution of cure stresses in polymeric resins, due to the cure shrinkage that accompanies essentially all polymerization reactions, is apparently a critical mechanism for conduction in this composite.



**Figure 12. Sensor test results (0-1500 s) showing sensor current when exposed to flows containing pentane vapor (dashed lines).**



**Figure 13. Sensor test results (1500-3500 s) showing sensor current when exposed to flows containing pentane vapor (dashed lines).**



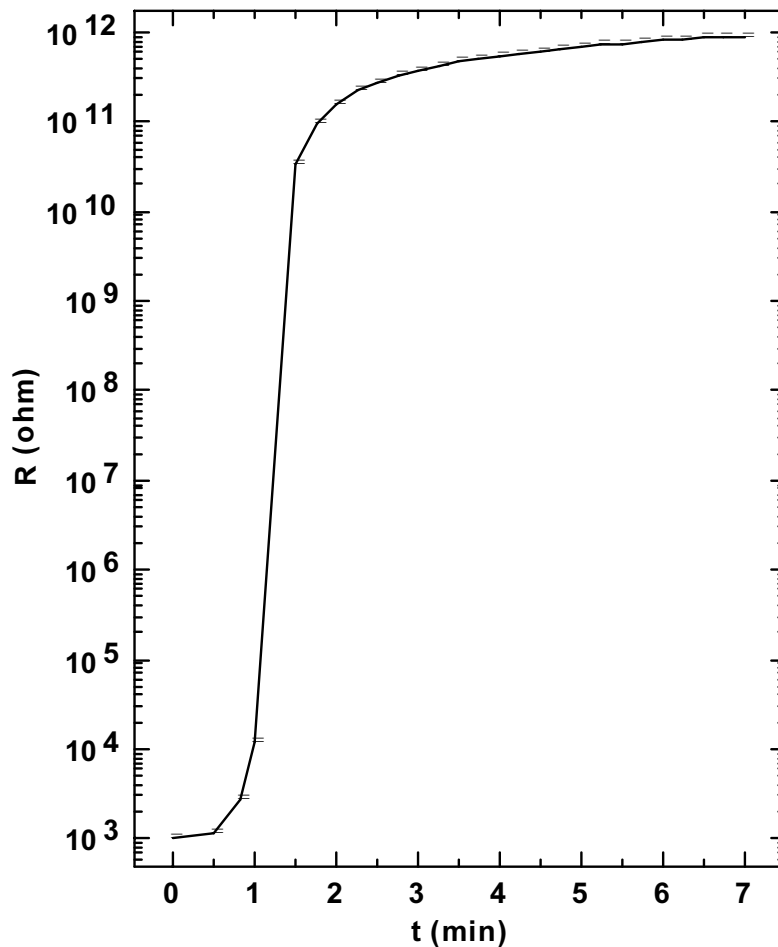
**Figure 14. Sensor test results (3500-6000 s) showing sensor current when exposed to flows containing pentane vapor (dashed lines).**

This indicates that there may be little benefit to using complex field processing techniques to attempt to enhance the conductivity of these composite materials. Instead, it might be more useful to focus on controlling stress development in the encapsulant through the selection of resins with appropriate cure shrinkages.

The importance of stress in current conduction has been reported earlier in piezoresistance measurements made on particle composites in an epoxy resin. [2] In these measurements a compressive stress was applied to a fully cured composite containing field-structured Ni particles. Measurements made both above and below the glass transition showed an 11-decade reduction in the resistance over a recoverable 6% strain.

There is some discussion in the literature on the dependence of the conductivity of a conducting particle composite on the volume fraction of particles, and whether this conforms to the expectations of percolation theory. [4-7] Typically, a critical volume fraction  $\phi_c$  of particles is found, above which the composite conducts appreciably. Above the percolation threshold the conductivity data are fit to critical form, such as expected for a random resistor-insulator network,  $C \propto |\phi - \phi_c|^t$ . Simple percolation theory predicts that the exponent  $t \approx 2$  in three dimensions, but larger exponents are often found. These larger exponents are often attributed to particle agglomeration, and this must occur since the observed percolation threshold is much lower than the random close pack particle density. However, an underlying assumption is that the particle contact resistance is

independent of particle concentration. In a technical sense, this is a good assumption, because it is doubtful that a singularity in the contact resistance occurs at the percolation threshold. However, real experimental data are taken relatively far from the percolation threshold, and so the possibility exists that the contact resistance is nonuniform over this region, thus altering the apparent exponent  $t$ . This is a complex problem because it points to the need to understand the development of local stresses in filled composites during cure. It is not at all clear that the contact pressure between particles would be independent of the volume fraction of particles, which is the necessary condition for simple percolation to apply to these systems. Our measurements, indicating 7-8 orders of magnitude change in conduction after the resin gel point, show that the dependence of the contact resistance on stress is significant. Indeed, it appears that it is the dominant mechanism in establishing particle-particle contacts capable of carrying electrical current.



**Figure 15. The prototype sensor response when exposed to a saturated vapor of toluene.**

In addition to demonstrating the significance of cure stresses in establishing current conduction in FSCs, this work has also demonstrated the feasibility of producing a practical chemical sensor based on FSC technology. Given the preliminary nature of the results shown here, it is obvious that a great deal of work remains to be done before a practical sensor is actually produced. The issues of baseline current stability and response recovery have yet to be studied. Furthermore, concentration studies and chemical specificity issues must be investigated.

## Conclusions

The following conclusions can be drawn from this work:

1. For the FSC systems studied, the inter-particle attractive forces induced by the structuring field are not sufficient to generate sufficient contact pressure to support current conduction. This is not to say that such is the case at much higher volume fractions than those used for these studies, or under bi- or tri- axial field conditions where structures of higher (fractal) dimension are produced.
2. Polymer cure shrinkage is the dominant mechanism in producing inter-particle contacts sufficient to support current conduction for FSC systems produced under the conditions of these studies.
3. Responsive, very sensitive proto-type sensors can be produced based on FSC technology, with very long shelf life. Preliminary tests show that practical sensors can be produced once issues of response dynamics and chemical specificity have been addressed.

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Distribution

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