# AFRL-ML-WP-TP-2006-421

# PROCESSING AND CHARACTERIZATION OF SHAPE MEMORY POLYMER NANOCOMPOSITES (PREPRINT)

**David Fortener**

## FEBRUARY 2006

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**14. ABSTRACT**
This report was developed under a SBIR contract.

Cornerstone Research Group, Inc. (CRG) has demonstrated the feasibility of adding nanoparticulates into their shape memory polymer (SMP) resin systems. Under thermal or other stimuli, SMPs can exhibit a radical change from a rigid polymer to a flexible, elastic state, and then back to a rigid state again. Nanoparticles, including carbon nanofibers and carbon nanotubes, are expected to increase material properties such as electrical conductivity, thermal conductivity, and mechanical properties. CRG has demonstrated this on a lab-scale. For end use purposes, these results must be transitioned to large-scale manufacturing. This paper will discuss the process of transitioning SMP nanocomposites from lab-scale to large-scale production. In order to verify the properties after scale-up, the scaled-up samples of various percent loading of nanoparticles will undergo thorough testing to include, but not be limited to, electrical conductivity, thermal conductivity, and dispersion study in the resin using a scanning electron microscope (SEM).

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9 February 2006

Shane Juhl
Material Research Engineer
AFRL/MLBP
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1. Draft SAMPE Proceedings
2. Draft AFRL Form 6, "Public Affairs Security and Policy Review, Request for Public Release Clearance"

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# Processing and Characterization of Shape Memory Polymer Nanocomposites

David Fortener
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## ABSTRACT

Cornerstone Research Group, Inc. (CRG) has demonstrated the feasibility of adding nanoparticulates into their shape memory polymer (SMP) resin systems. Under thermal or other stimuli, SMPs can exhibit a radical change from a rigid polymer to a flexible, elastic state, and then back to a rigid state again. Nanoparticles, including carbon nanofibers and carbon nanotubes, are expected to increase material properties such as electrical conductivity, thermal conductivity, and mechanical properties. CRG has demonstrated this on a lab-scale. For end use purposes, these results must be transitioned to large-scale manufacturing. This paper will discuss the process of transitioning SMP nanocomposites from lab-scale to large-scale production. In order to verify the properties after scale-up, the scaled-up samples of various percent loading of nanoparticles will undergo thorough testing to include, but not be limited to, electrical conductivity, thermal conductivity, and dispersion study in the resin using a scanning electron microscope (SEM).

KEYWORDS: Nanocomposites, Smart Materials, Advanced Composite Materials

## 1. INTRODUCTION

Polymer nanocomposites (PNC) are a new class of composites in which polymers have been reinforced with small quantities of nano-sized particles having high aspect ratios (L/h > 300) [1]. PNCs have been shown to offer remarkable property enhancements relative to conventionally scaled composites [2]. The addition of low-volume fractions of nanoparticles, such as carbon nanofibers or carbon nanotubes, into polymer matrix has provided unique thermal and electrical properties not normally possible with traditional fillers [3]. Thus, CRG can envision enhanced performance and new capabilities, such as unique triggering mechanisms for shape recovery beyond direct external thermal heating, by integration of these two material concepts (shape recovery polymers and nanocomposites).

Nanoparticle additives can provide benefits in comparison to both their conventional filler counterparts and base polymer. Mechanical, thermal, and electrical properties have been shown to undergo substantial improvements [1]. The addition of nanofibers to increase electrical conductivity is already being used in conductive paint applications, in the case of automotive

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side mirror housings [4]. In addition, it is important to recognize that nanoparticulate loading offers significant property improvements with very low loading levels. This, in turn, can result in significant weight reductions (of obvious importance for various military and aerospace applications) compared with conventional composite materials for similar performance [1].

At the forefront of shape memory polymer (SMP) technology, CRG has been researching SMP since 1998 under the trade name Veriflex™. SMPs are polymers whose qualities have been altered to give them dynamic shape "memory" properties. Under thermal stimuli, SMPs can exhibit a radical change from a rigid polymer to a very flexible elastic state, and then back to a rigid state again. In its elastic state, SMP will recover its "memory" shape if left unrestrained. Veriflex™ is used as the resin matrix for composites, which offers several benefits in terms of manufacturing composite parts. SMP composites are similar to other high-performance composites, with the exception that SMP resin is used as the matrix resin, which makes these composites extraordinary. SMP composites allow for easy manipulation of the composite above the activation temperature, while affording high strength and stiffness at lower temperatures. The SMP composite acquires some SMP characteristics, making it a unique material for use in dynamic structures and other applications requiring both load strength and "shape-shifting" modulus flexibility.

There are three types of shape memory polymers: partially cured resins, thermoplastics, and fully cured thermoset systems. Through years of research, CRG has found limitations and drawbacks to the first two types of SMP. Partially cured resins continue to cure during operation and change properties with every cycle. Thermoplastic SMP "creeps," which means it gradually "forgets" its memory shape over time. With this supporting research and a thorough understanding of the chemical mechanisms of SMP, CRG has developed fully cured, high-performance thermoset systems. These systems are trademarked as Veriflex™ (see Figure 1). The memorized shape is the flat, rectangular coupon. Veriflex™ can be heated, then twisted, rolled, stretched, or compressed. It can then be cooled to maintain the new shape in a rigid state. Heated above its glass transition temperature again, it will return to its memorized shape.

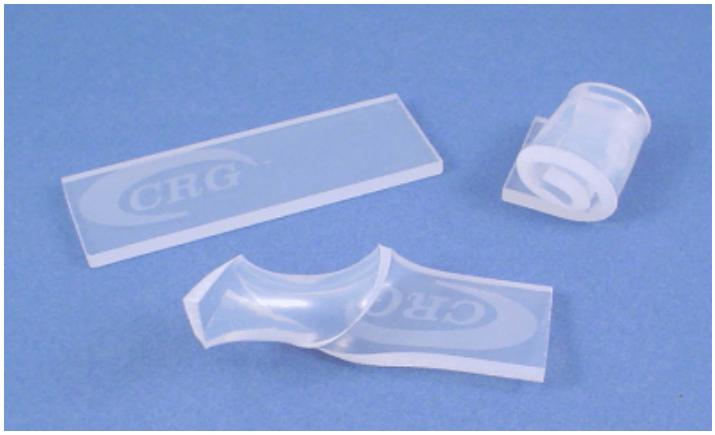
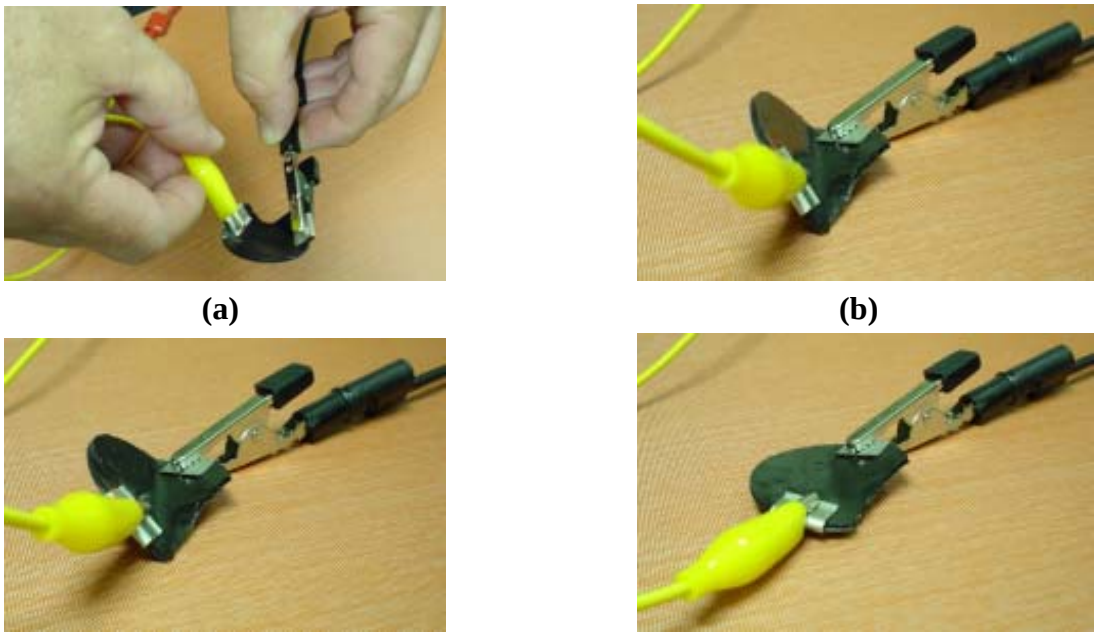



**Figure 1: Veriflex™ shape memory polymer coupons**

A polymer engineered with shape memory characteristics provides a unique set of material qualities and capabilities that enhance traits inherent in the polymer system itself. Veriflex™ can

be chemically formulated with a glass transition temperature to match any application need. It can be cast and cured into a wide variety of "memorized" shapes, including thick sheets, concave dishes, and even very tiny parts. Developing alternative activation mechanisms will improve response time, efficiency, and lessen the general drawbacks with external thermal heating. The addition of electrically and thermally conductive nanoparticles will allow for these alternative means. Figure 2 depicts the ability of SMP nanocomposites to be activated using Joule heating.



**(a)** **(b)**

**(c)** **(d)**

**Figure 2: Deformation and deployment of CNF-SMP nanocomposite (7 wt % CNF) by Joule heating. (a) Deformation after the nanocomposite was heated up past the $T_g$ by passing an electric current through the sample; (b, c, and d) different stages of deployment from the rigid state to the original shape as the nanocomposite was heated by Joule heating**

CRG has demonstrated the addition of nanofibers to SMP on a lab-scale. For end-use purposes, these results must be transitioned to large-scale manufacturing. This paper will discuss the process of taking SMP nanocomposites from lab-scale to large-scale production. In order to verify the properties after scale-up, the scaled-up samples of various percent loading of nanoparticles will undergo thorough testing to include, but not be limited to, electrical conductivity, thermal conductivity, and dispersion study in the resin using a scanning electron microscope (SEM).

## 2. MATERIALS

The resin used in this study is a styrene-based shape memory polymer (SMP) designed and developed by Cornerstone Research Group. The resin is styrene-based SMP, VF-62 (CRG Industries, Dayton, OH). This resin is a thermoset resin that must be cured in a closed mold to inhibit the styrene from boiling off. From previous research, it was found that with the addition of carbon nanofibers, the resin must cure at 85 °C for twenty-four hours. The carbon nanofibers used in this study are Pyrograf® PR-24-XT-LHT-LD. These fibers were obtained from Applied

Sciences, Inc. (Cedarville, OH). Pyrograf® fibers are produced through vapor growth, which is a catalytic process of hydrocarbons in the vapor state [5]. Their configuration is what gives the fibers their physical properties, including electrical and thermal conductivity [6]. The nanofibers themselves tend to clump together due to Van der Walls forces [7]. This can be seen with a SEM image of the fibers received as is in Figure 3. The use of a continuous mixer will aid in the breaking up and dispersion of these nanofibers.

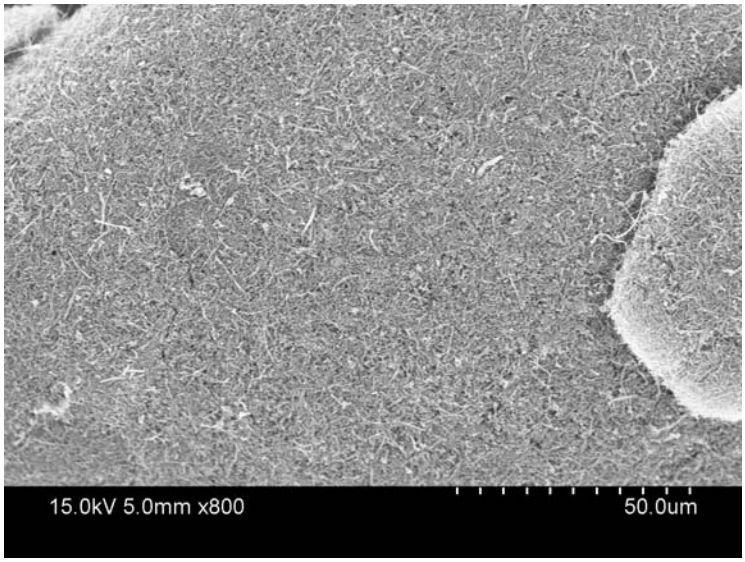



**Figure 3: SEM of Pyrograf® PR-24-XT-LHT-LD carbon nanofibers as received**

## 3. EXPERIMENTAL

**3.1 Large-Scale Production** Creating a homogenous mixture of the SMP and a nanoparticulate on a large scale is a difficult task. A process must be implemented that will result in repetitive successful homogenous mixtures of the resin and nanoparticulate. Handmixing the resin and the nanoparticulate will be one approach to scale up from lab-scale to manufacturing-scale process. A more economical and efficient mixing method is the use of a continuous mixer. CRG is a beta-site for a Readco Continuous Mixer which will be used for the scale-up process for the SMP nanocomposites. Figure 4 shows the machine to be used for the mixing of the SMP and the nanoparticles.

**Figure 4: Readco 2-inch Continuous Processor**

The paddle configuration inside the Readco continuous mixer can be changed in order to optimize the mixing process. The mixing can vary from very low shear to very high shear mixing. Figure 5 shows a sample of the paddle configuration inside the barrel of Readco continuous processor.

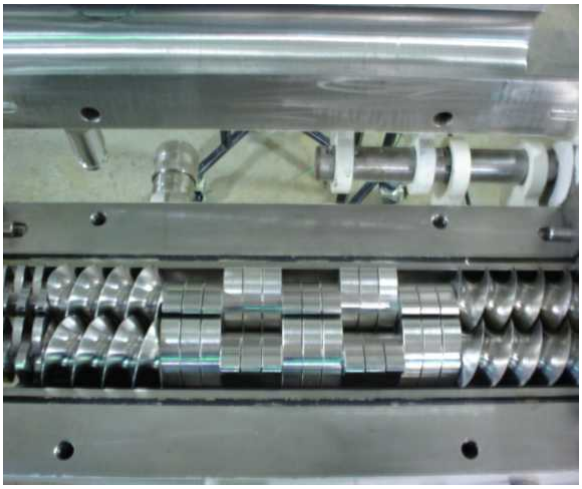


**Figure 5: Paddle Configuration of the Readco Continuous Mixer**

**3.2 Dispersion Verification** Verifying that the carbon nanofibers are dispersed into the SMP matrix is a critical step. The improvement of both electrical and thermal conductivity relies on the nanofibers creating a continuous network throughout the polymer. An uneven dispersion would cause the nanofibers to act as filler, rather an improvement. In order to verify that the resulting nanocomposites contain evenly dispersed nanofibers, samples of different weight percentage will be examined using a scanning electron microscope (SEM). This high powered microscope will allow CRG to determine how well the nanofibers are being mixed into the SMP after scale up. Figure 6 shows dispersion of carbon nanofibers and SMP mixed by a small-scale automated mixer.

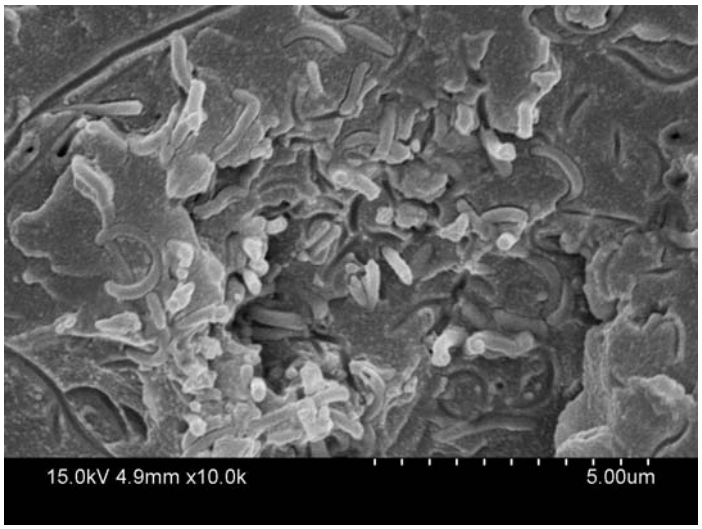



**Figure 6: SEM of SMP nanocomposite showing good dispersion of nanofibers in polymeric matrix**

**3.3 Characterization** Using in-house equipment, CRG has tested the nanocomposites to verify the improved electrical and thermal properties of the product. Using an automated 4-point probe and computer software, the electrical conductivity of the nanocomposite will be determined. Carbon nanofibers are a highly conductive material and the addition of these fibers into an inherently non-conductive polymer will result in an electrically conductive SMP. The resulting SMP will be able to be activated using electricity, instead of and external heating element. The new, intrinsically conductive SMP can then be implemented into any number of systems needing quick shape shifting mechanisms without needing an external heating mechanism. The ability of an SMP to be heated quickly and evenly is extremely important. The addition of carbon nanofibers, which are good thermal conductors, are expected to increase the thermal conductivity. The resulting nanocomposite can then be heated quickly and be employed in operation needing quick activation to accommodate shape change.

## 4. RESULTS AND DISCUSSION

**4.1 Large-Scale Production** Initially, CRG used a handmixing method to incorporate the nanofibers into the SMP resin matrix to fabricate larger parts. This resulted in unevenly dispersed fibers in the matrix, and it is not conducive to large-scale production of either resin or cured sheets of material. Handmixing on a small scale has been replaced with a small-scale automated mixer. This new process is getting good dispersion as seen from Figure 6 as well as through electrical conductivity measurements. An issue with curing was also a problem with handmixing the nanofibers into the resin. “Hot spots” appeared in sample made with hand mixing, as seen in Figure 7.

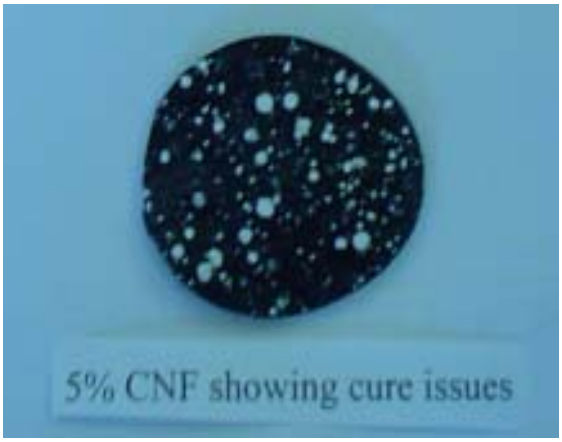



**Figure 7: Nanocomposite showing "hot spots" from cure issues incurred during the hand mixing of nanofibers into SMP**

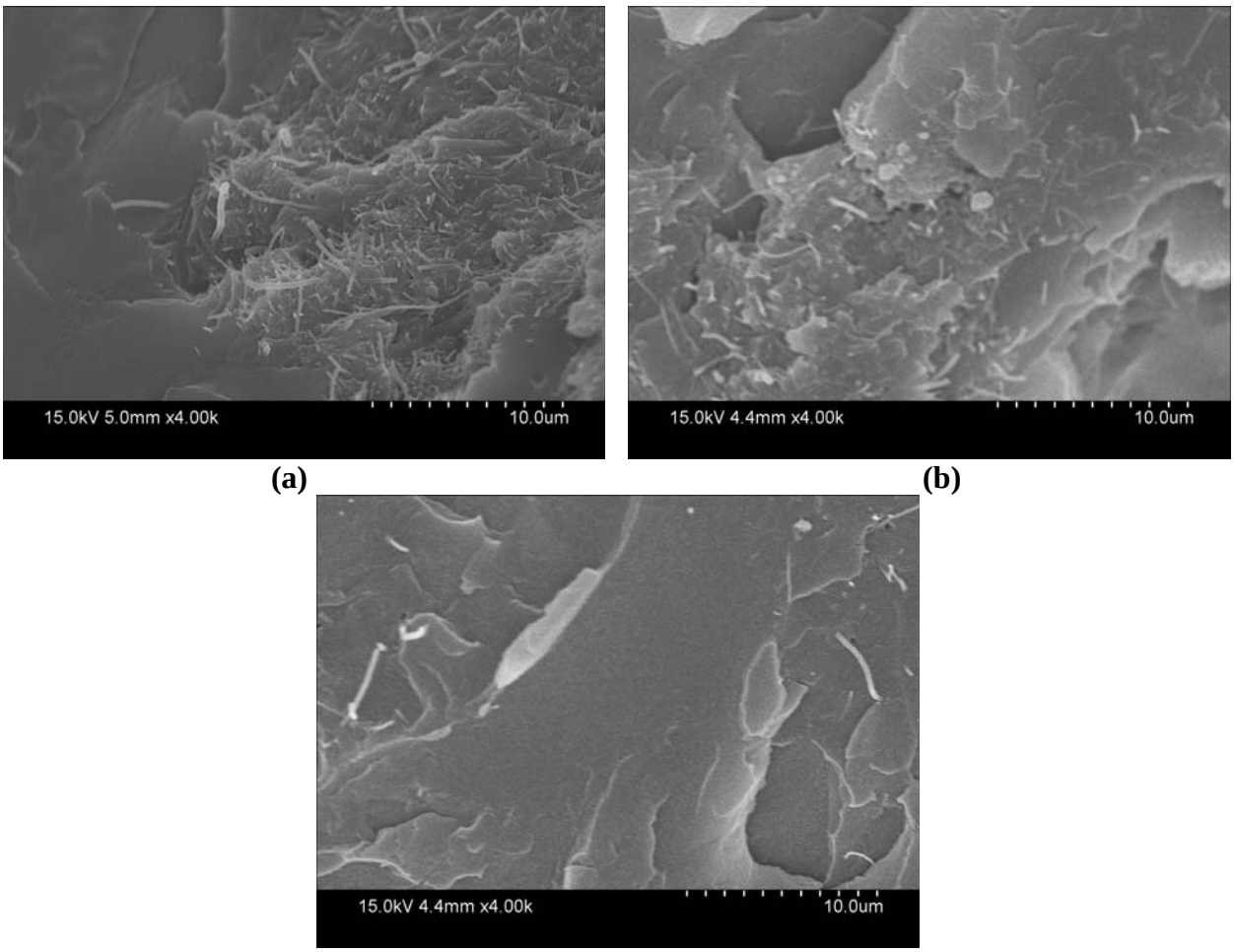
The next trial for scale up was to incorporate the Readco Continuous Mixer into the mixing process. This process was less time consuming and more efficient in mixing in the nanofibers. Figure 8 shows two sheets of cured material of different weight percent loadings, with no sign of cure issues or of "hot spots."

**(a)** **(b)**

**Figure 8: Cured nanocomposite of varying weight percentage. Item (a) had a loading of 2% by weight while item (b) has a 7% by weight loading.**

The continuous mixing process reduced the time and man hours needed to mix the nanofibers into the SMP resin. The continuous mixer also allows for much higher weight % loadings than can be attempted by hand. The mechanical energy produced by the machine far exceeds that of human-powered mixing.

**4.2 Dispersion Verification** In order to confirm that the mixing process in the Readco was producing well dispersed nanocomposite, the samples needed to be examined at under high magnification, as discussed in Section 2.2. Figure 9 shows SEMs of 2% by weight nanocomposite and a 7% by weight nanocomposite.




**(a)** **(b)**

**(c)**

**Figure 9: (a) SEM at 4000x of a 2% by weight loading nanocomposite showing an uneven dispersion of the nanofibers in the SMP matrix. SEM at 4000x of a 7% by weight loading nanocomposite showing both fiber rich (b) and resin rich areas (c).**

The first iteration of using the Readco to incorporate the carbon nanofibers into the SMP was successful in that no cure issues were encountered as well as demonstrating a more efficient process for integrating the nanofibers into the SMP. However, the dispersion of the CNF in these nanocomposites were not ideal, as shown in Figure 9, further iterations with more aggressive mixing paddle configurations inside the Readco mixing chamber should result in a more evenly dispersed resin.

**4.3 Characterization** Two of the most important properties that the addition of carbon nanofibers will improve are thermal and electrical conductivity. SMP is not an electrically conductive polymer. Previous testing of the nanofibers for electrical conductivity showed that loadings under 2% did not demonstrate any detectable change in the properties of the material. Once the fiber loading was over 2%, noticeable changes ensued. The following table shows the results of electrical conductivity of a nanocomposite with 5% nanofibers hand mixed on a small scale.

| Position in X Direction mm | Position in Y Direction mm | Sheet Resistance (ohm) | Resistivity o-cm | Thickness microns |
|---|---|---|---|---|
| 0 | 0 | 939.3 | 126.8 | 1350 |
| 90 | 0 | 1290.1 | 174.1 | 1350 |
| 0 | 90 | 329.7 | 44.5 | 1350 |
| -90 | 0 | 673.6 | 90.9 | 1350 |
| 0 | -90 | 1556.8 | 210.1 | 1350 |

**Table 1: Electrical conductivity data for a 5% by weight loading of carbon nanofibers into SMP**

Resistivity is the inherent property of the material which gives it electrical resistance. It is sometimes called specific resistance. Sheet resistance is the resistance of a thin sheet of material which when multiplied by the thickness (in cm) gives the value of resistivity. As can be seen from the data, the 5% loading of carbon nanofibers handmixed into the SMP results in a conductive polymer. When the nanocomposites mixed using the continuous mixer were tested, the inadequate mixing of the fibers into the polymeric matrix was apparent. The material behaved as if there were no fibers in the composite and did not register any conductivity. However, once the mixing of nanofibers into SMP using the Readco is optimized, results comparable to these can be expected.

The thermal conductivity unit measures thermo-physical properties according to ASTM D5930 [8]. Values for thermal conductivity are express in units W/mK, are determined within the range of -15 °C and 50 °C. Thermal conductivity data of the first iteration of mixing was determined using this apparatus. Table 2 shows the results of the thermal conductivity testing.

| Weight % of carbon nanofibers | Thermal Conductivity (W/mk) |
|---|---|
| 0 | 0.17 |
| 2 | 0.177 |
| 7 | 0.164 |

**Table 2: Thermal conductivity data carbon nanofibers-SMP nanocomposite**

The thermal conductivity data demonstrates that the non-uniform dispersion of the nanofibers in the SMP results in an inconsistent improvement of the thermal conductivity. A slight increase was seen in the 2% loading while a slight decrease was seen in the 7% loading. Theoretically, the thermal conductivity should not decrease at higher loading. This characterization method requires a material sample large enough to acquire a bulk property representation. The thickness of the samples tested was not thick enough to accommodate this requirement and had to be stacked on top of each other to achieve an adequate thickness. In stacking the samples, the interface between each sheet introduces an additional variable that may have skewed the results. Thicker samples will be fabricated and analyzed to confirm this theory.

## 5. SUMMARY AND CONCLUSIONS

CRG has demonstrated the ability to incorporate carbon nanofibers and improve the properties of the material on a lab-scale. The ability to transition this success to a larger scale is a difficult, long process. The first few steps toward this process have been accomplished. CRG has demonstrated the ability to mix nanofibers into SMP on a large-scale and have not encountered any cure issues. By changing the paddle configuration on the Readco continuous mixer, the mixer can vary the shear level from high shear to little or no shear mixing. This capability should enable even dispersion of the nanofibers. Future work will optimize the paddle configuration for the Readco to enable even dispersion for the improvement of electrical and thermal conductivity.

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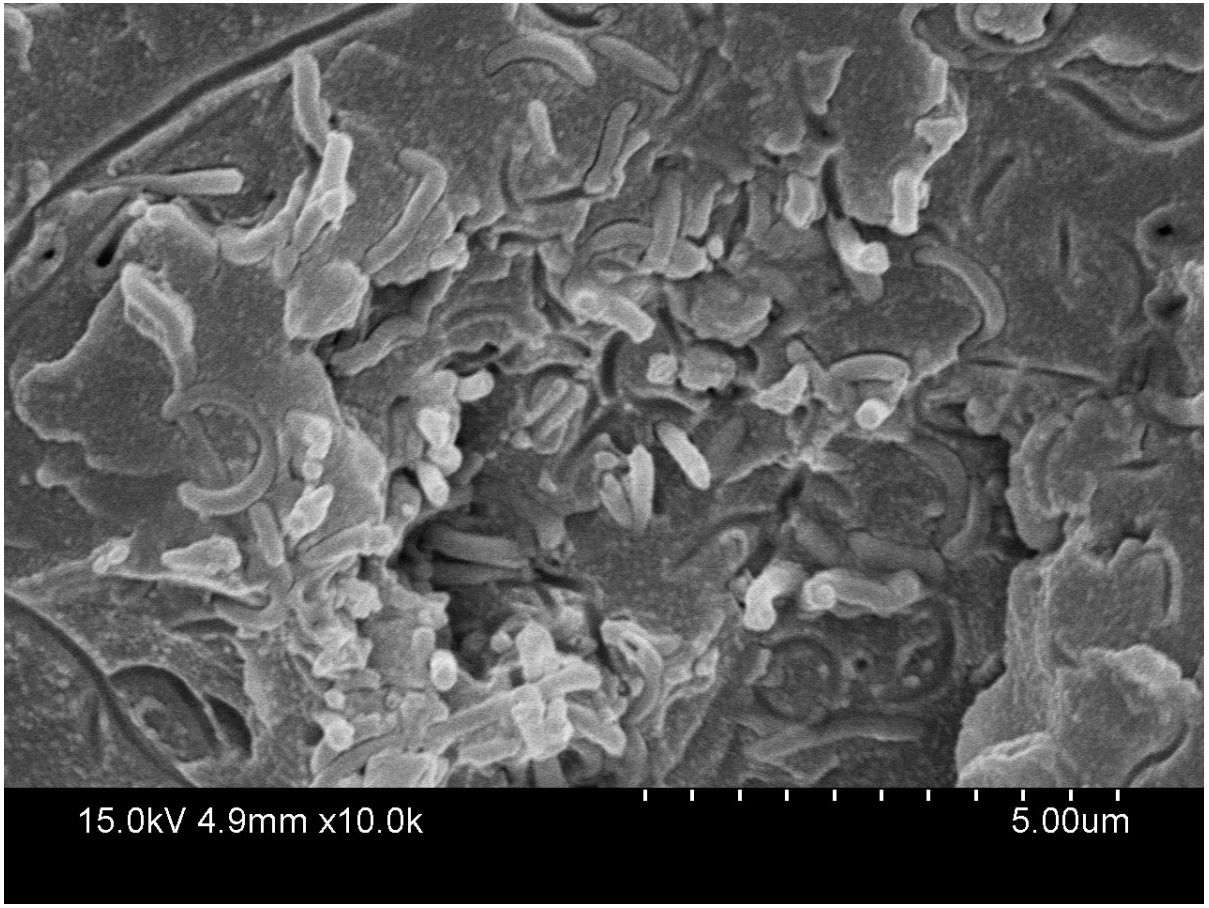
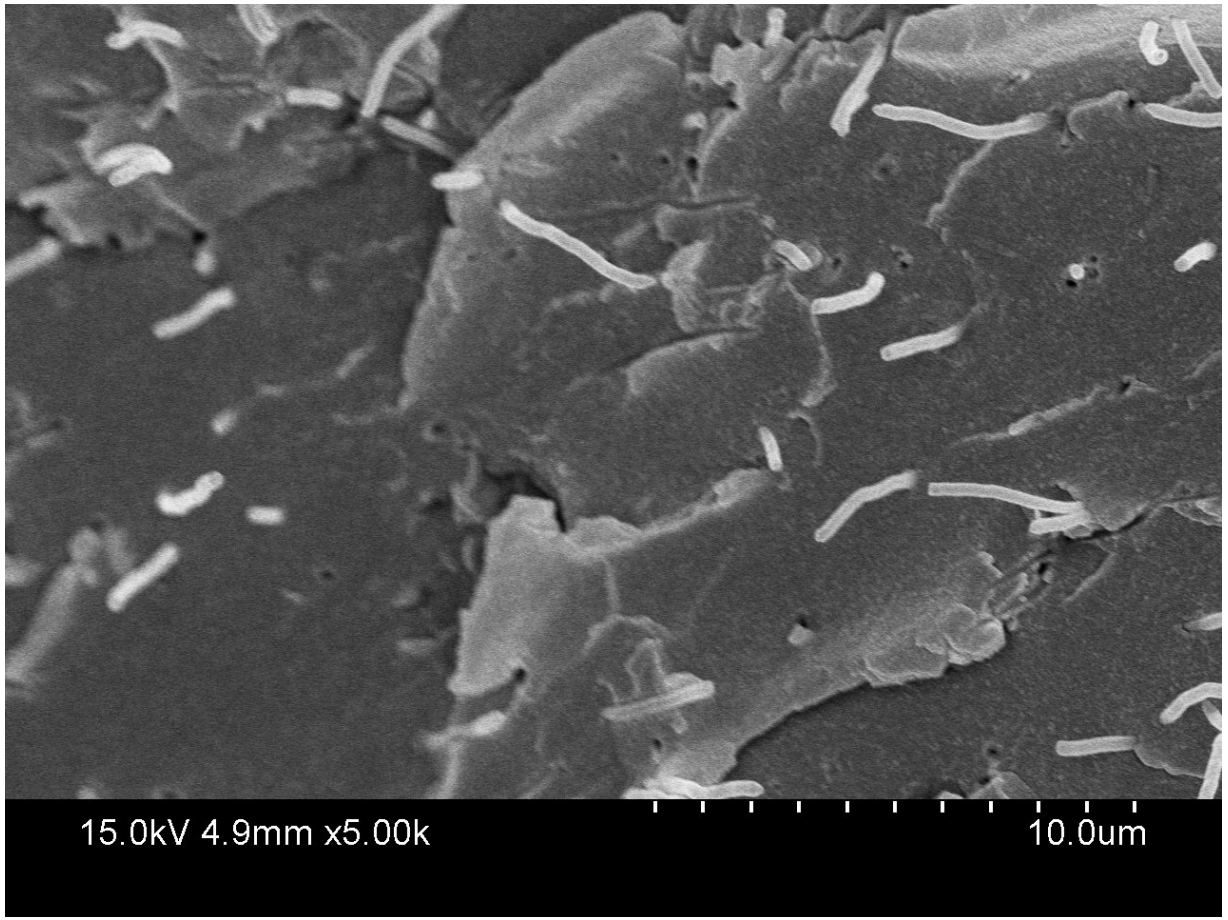
# SMP Nanocomposite




**Shape memory polymer composite with 3% wt. CNF using Veriflex™ resin at 10.0k magnification**

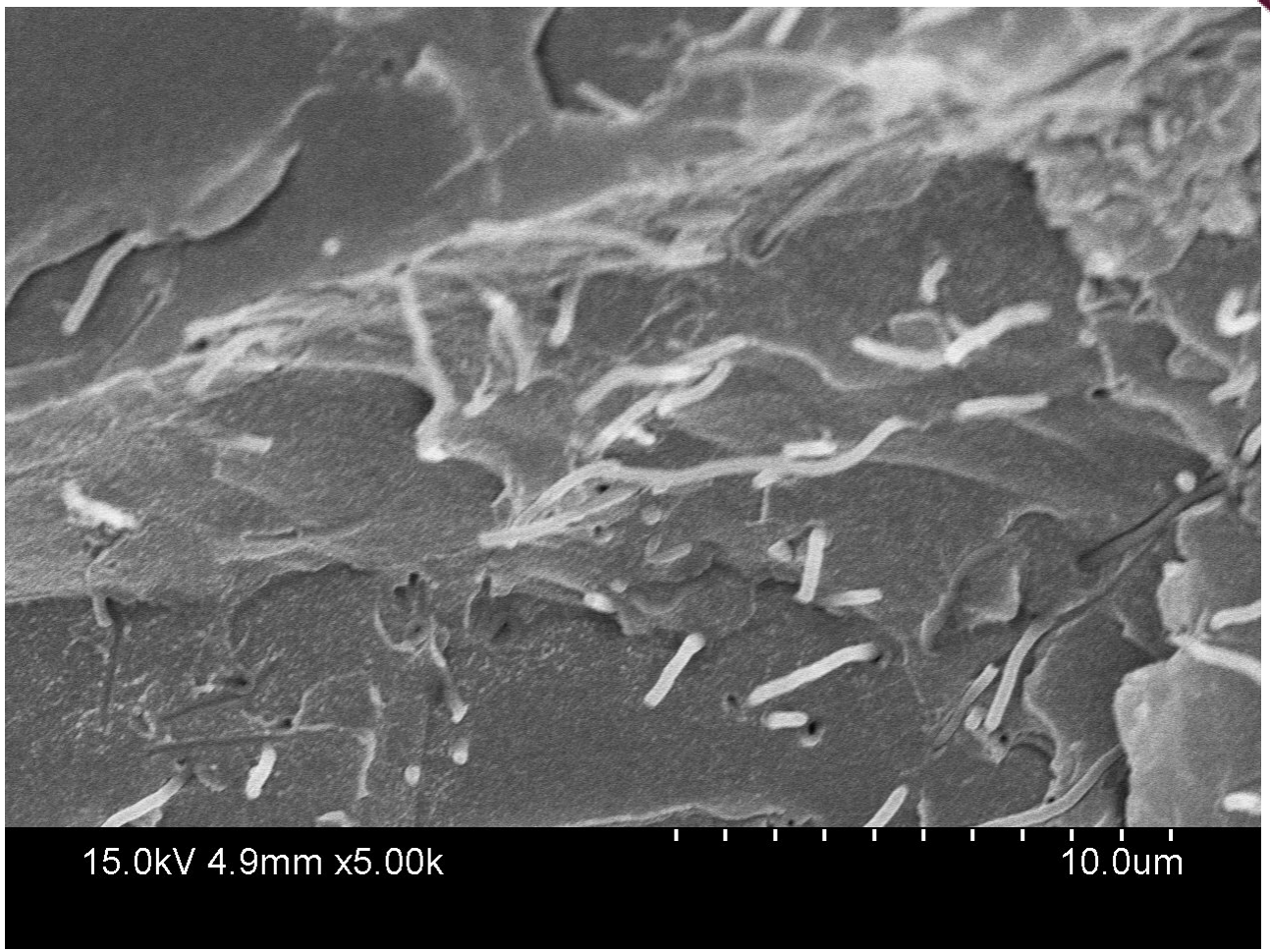
# SMP Nanocomposite




**Shape memory polymer composite with 3% wt. CNF using Veriflex™ resin at 5.00k magnification**

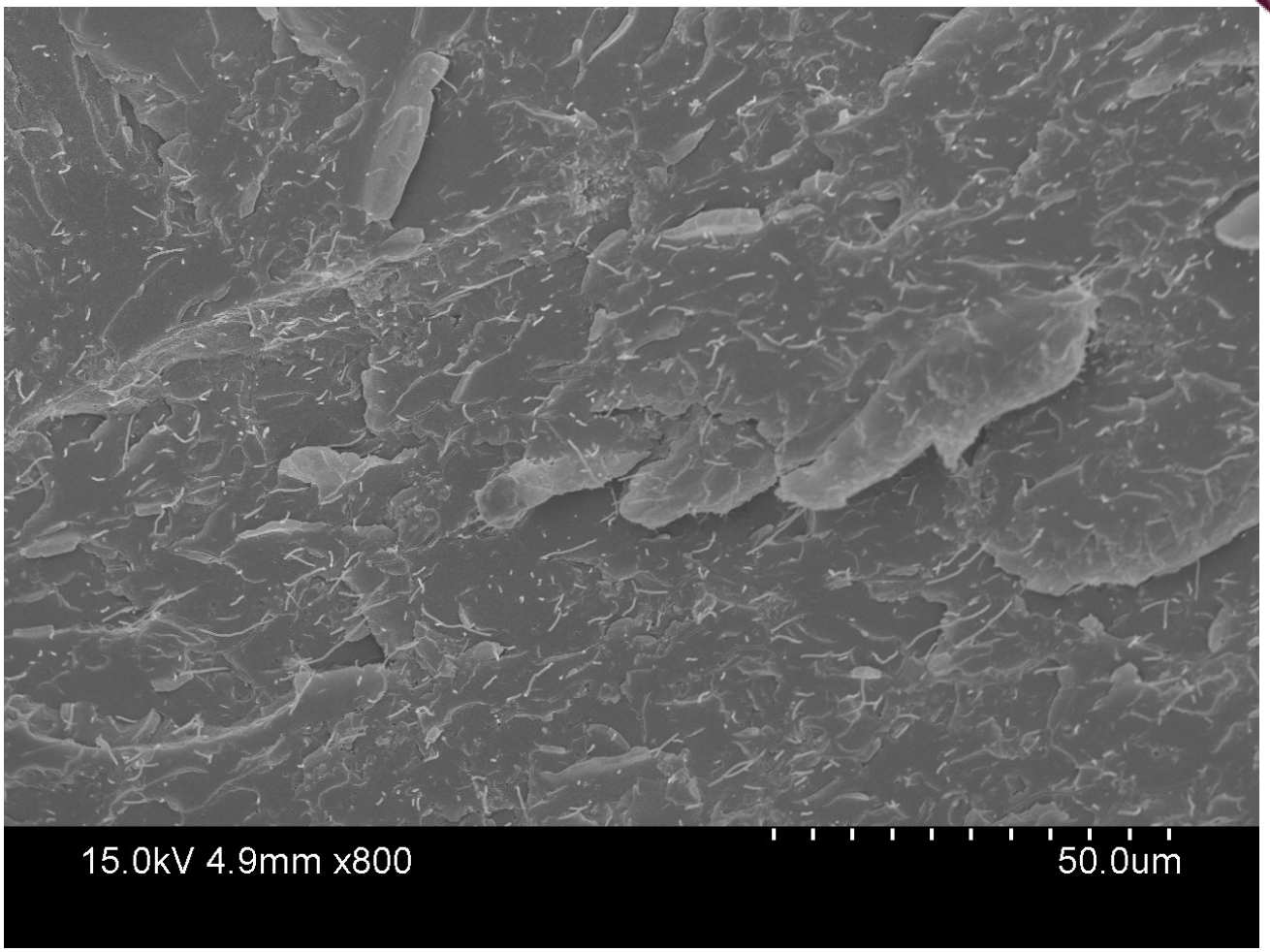
Cornerstone Research Group, Inc.

# SMP Nanocomposite



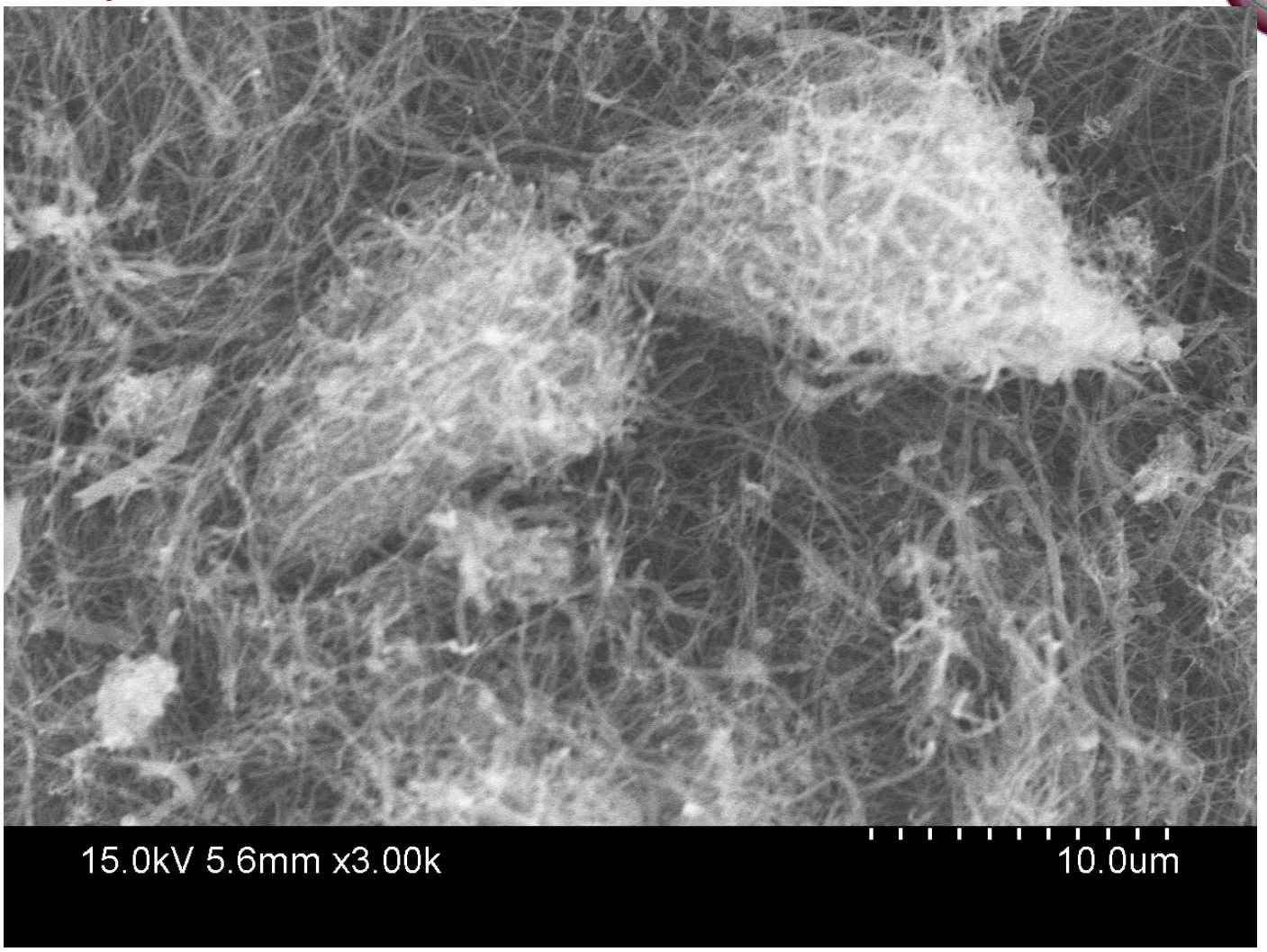

**Shape memory polymer composite with 3% wt. CNF using Veriflex™ resin at 5.00k magnification**

# SMP Nanocomposite



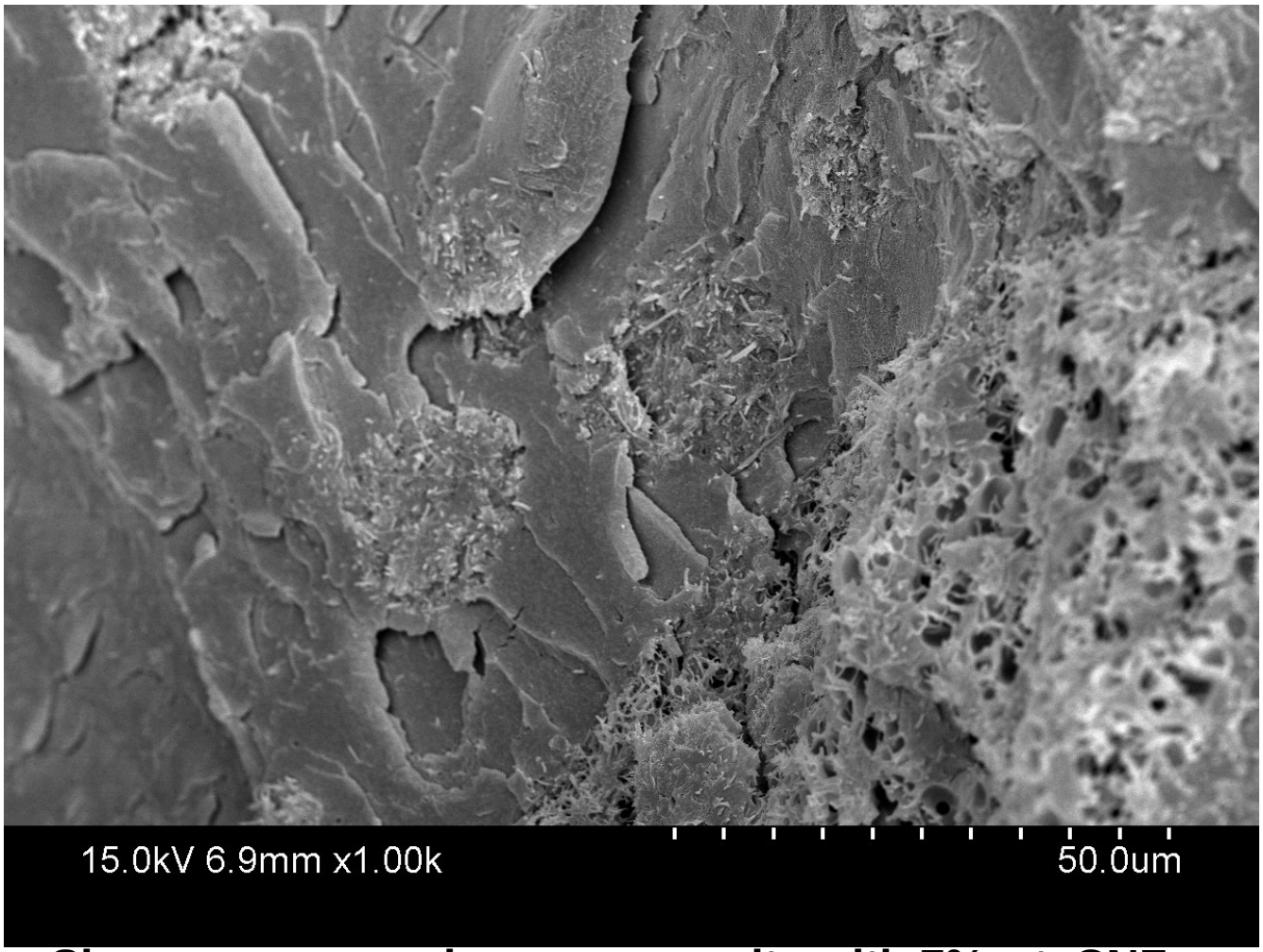

**Shape memory polymer composite with 3% wt. CNF using Veriflex™ resin at 800 magnification**

# SMP Nanocomposite



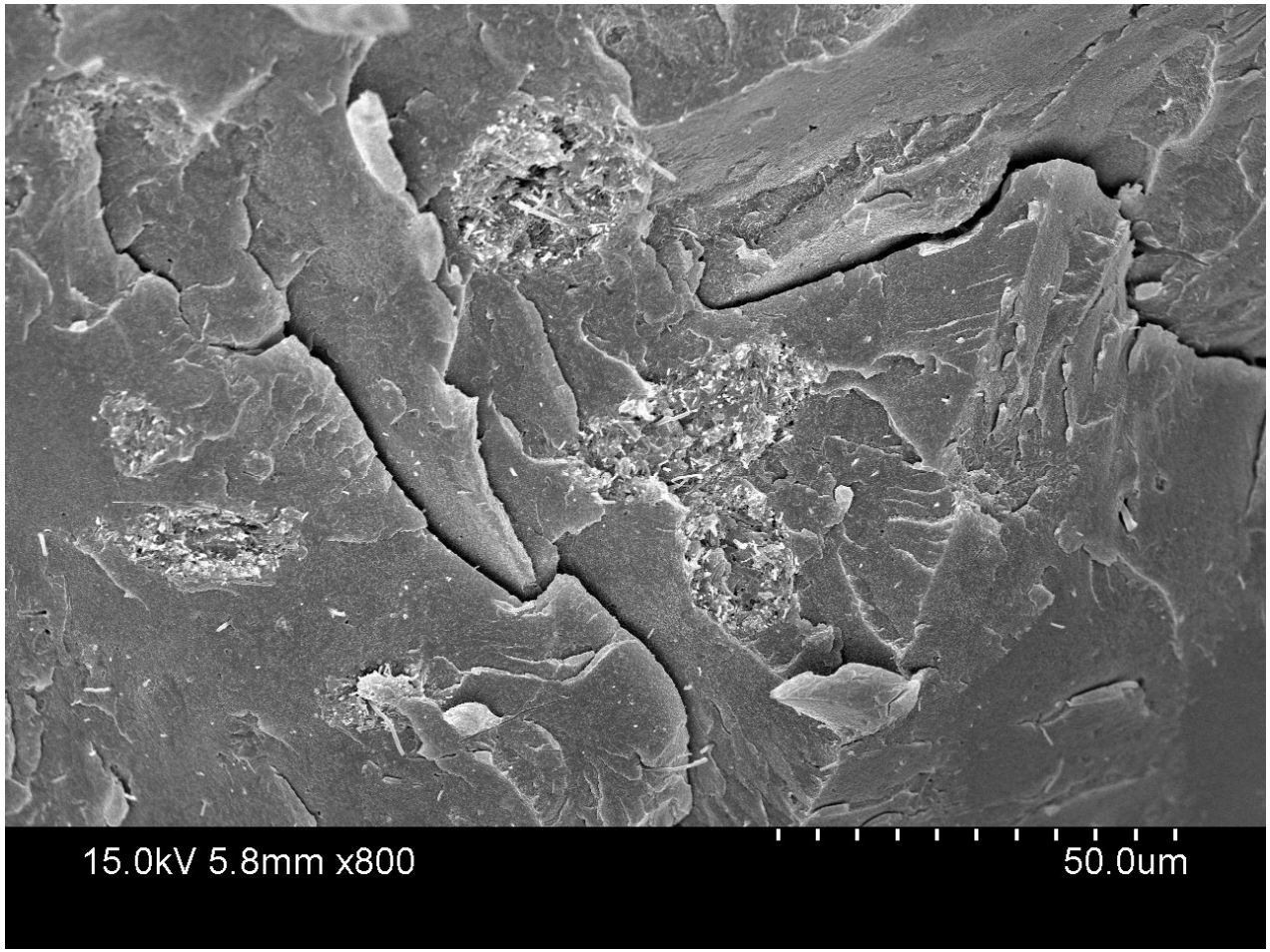

**Carbon nanofibers at 3.00k magnification**

# SMP Nanocomposite




**Shape memory polymer composite with 7% wt. CNF using Veriflex™ resin at 1.00k magnification. Shows agglomerations of CNF and poor dispersion.**

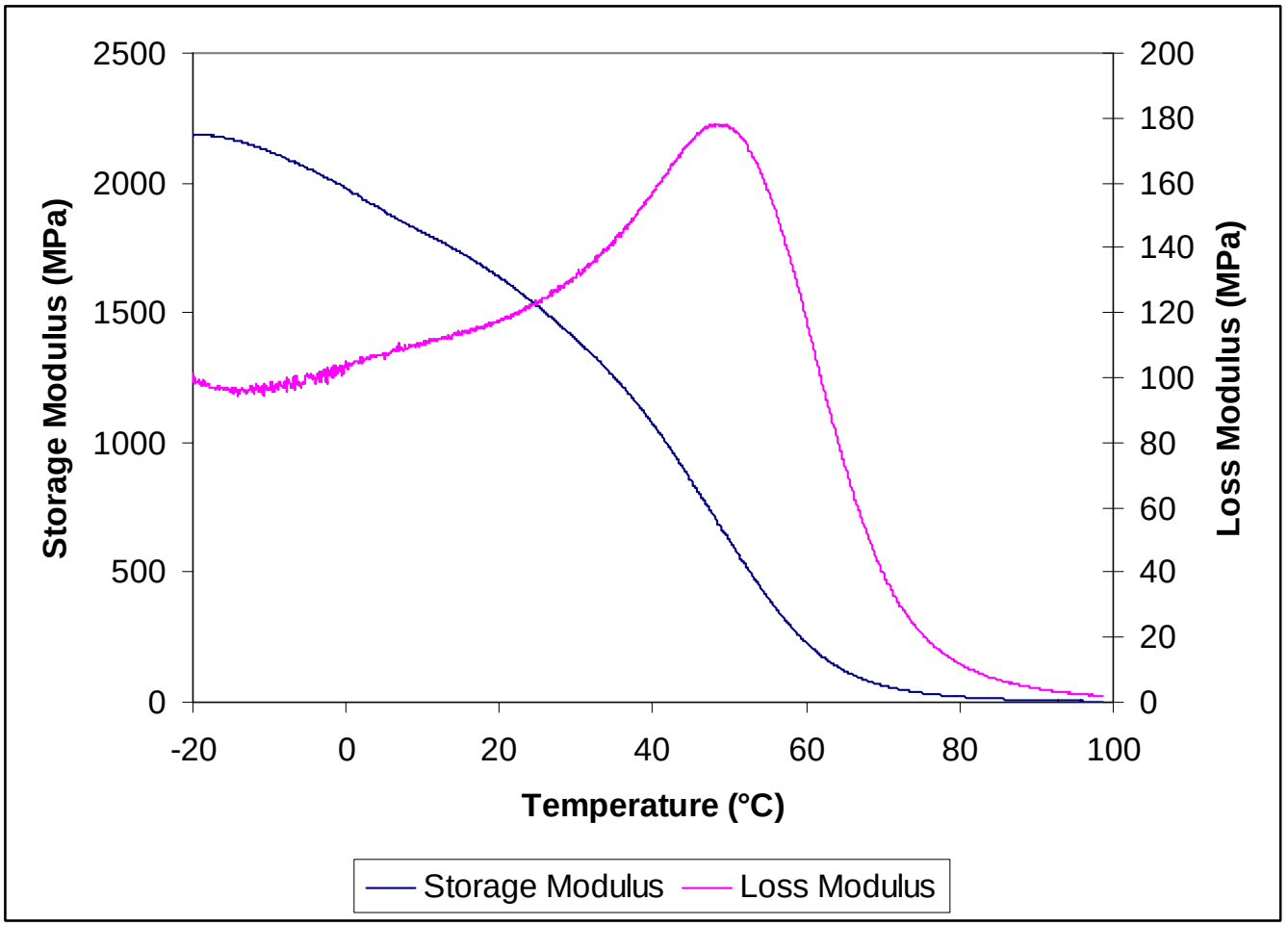
# SMP Nanocomposite




**Shape memory polymer composite with 7% wt. CNF using Veriflex™ resin at 800 magnification. Shows agglomerations of CNF and poor dispersion.**

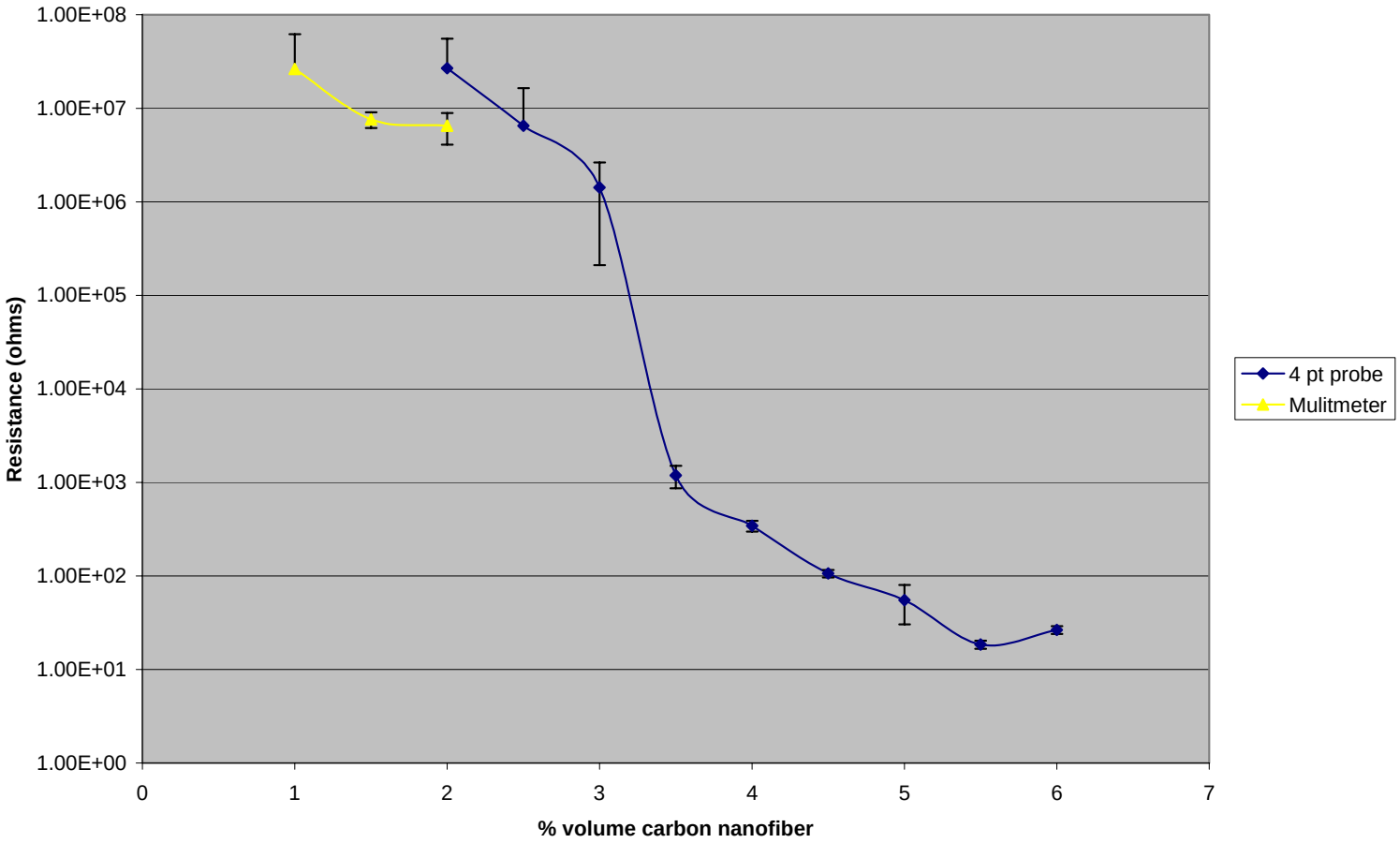
Cornerstone Research Group, Inc.

# SMP Nanocomposite



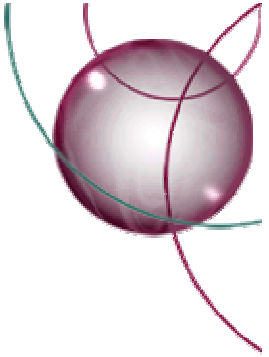

**Temperature versus storage modulus of shape memory polymer composite of Veriflex™ with 7% wt CNF**

# SMP Nanocomposite




**Percolation curve for shape memory polymer nanocomposite.**

# SMP Nanocomposite



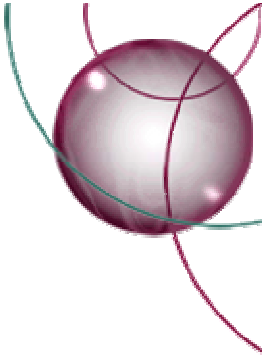
## Rheology

- Properties examined by rheology include deformation and flow.
- Viscosity can be explored as a function of shear stress applied on fluids.

## Rheological Properties of Carbon Nanofiber Resin

The level of nanofiber dispersion can affect the characteristics and conductivity of the host polymer drastically. Rheological properties were examined at various weight percent loadings under controlled testing procedures.

# SMP Nanocomposite

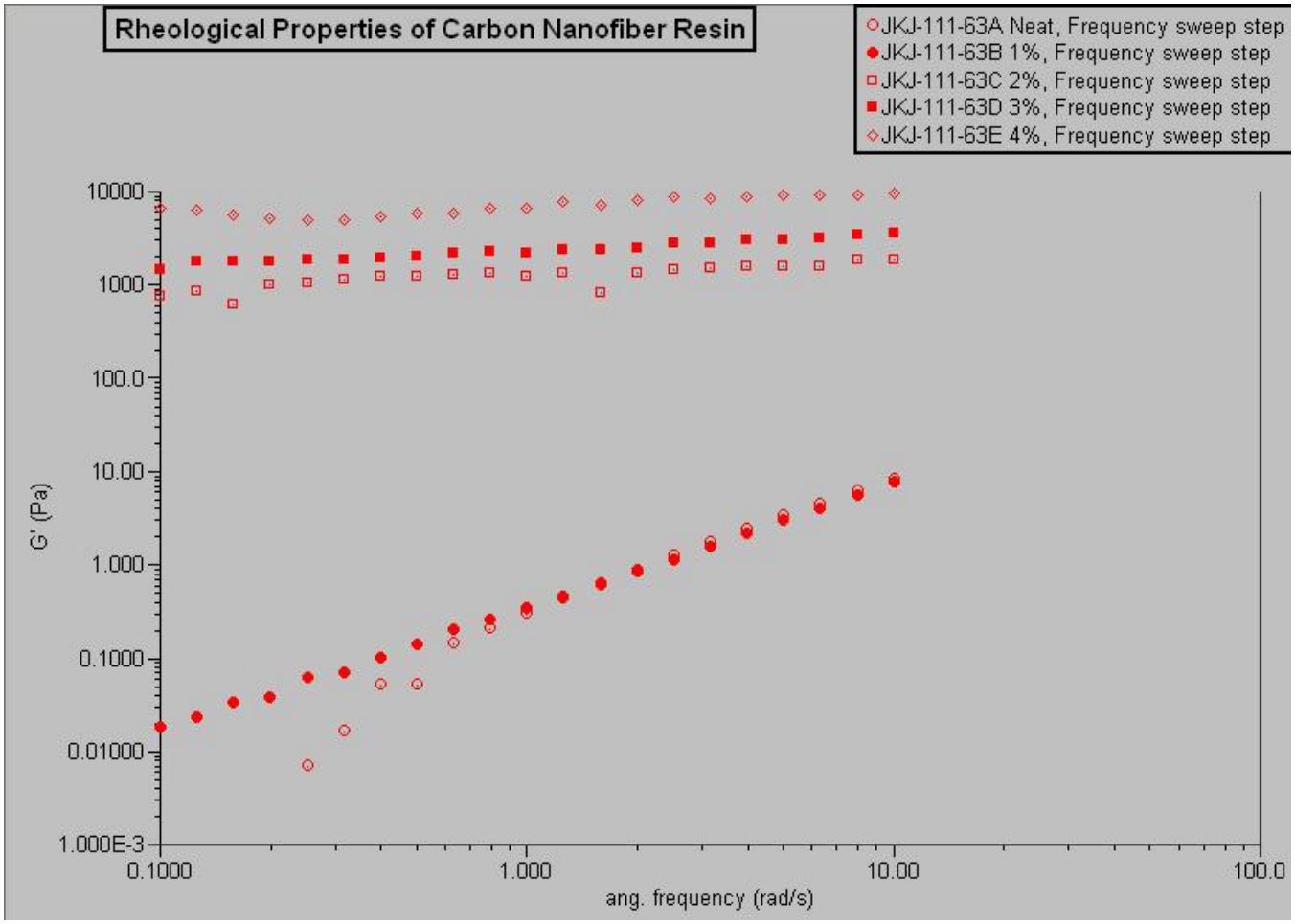


## Storage Modulus

The storage modulus (G') increases with respect to the percent loading of carbon nanofibers due to the increase in particle-particle interactions. These increased interactions can be related to the increase in conductivity of the host polymer.

The following slide exhibits the rheological properties of carbon nanofiber resin at increasing loadings from zero to four precent by weight. A dramatic increase in storage modulus is noted between one and two percent loadings with similar increase in modulus up to four percent loading.

# SMP Nanocomposite




**Rheological properties of Veriflex™ nanocomposite resin defining the quality of dispersion of the resin system.**