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MECHANICAL PROPERTIES OF GRAPHENE NANOPLATELET/ EPOXY COMPOSITES

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MECHANICAL PROPERTIES OF GRAPHENE NANOPLATELET/EPOXY COMPOSITES

By

Danielle René Klimek-McDonald

A DISSERTATION

Submitted in partial fulfillment of the requirements for the degree of

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This dissertation has been approved in partial fulfillment of the requirements for the Degree of DOCTOR OF PHILOSOPHY in Chemical Engineering

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Preface

The work contained in this dissertation was conducted in the department of Chemical Engineering at Michigan Technological University from August 2011 to August 2015. This dissertation is a compilation of published journal articles, journals articles accepted for publication, and journal articles to be submitted. The layout of the journal articles have been edited to present a flow of topics throughout the dissertation.

Material contained in Sections 4.1.1, 4.2.1, 4.3.1, 4.6.1, and 5.1 have been published in the following peer reviewed article:

J. A. King, D. R. Klimek, I. Miskioglu, G. Odegard; "Mechanical Properties of Graphene Nanoplatelet/Epoxy Composites" *Journal of Applied Polymer Science*, Vol 128, No. 6, pp.4217-4223, June 2013.

Content in Sections 4.1.2, 4.2.2, 4.6.2, 5.2, and 5.3 have been published in the following peer reviewed article:

J. A. King, D. R. Klimek, I. Miskioglu, G. Odegard; "Mechanical Properties of Graphene Nanoplatelet/Epoxy Composites" *Journal of Composite Materials*, Vol 49, No. 6, pp.659-668, March 2015.

Portions of Chapter 4 (4.1.4 and 4.6.3) have been submitted to *Carbon*. Parts of Chapter 4 and 5 (4.1.3, 4.2.3, 4.6.4, and 5.4) will be submitted for publication in *Polymer Composites* in August 2015.

This dissertation was written by Danielle Klimek-McDonald. All experiments and measurements discussed within this dissertation were performed by Danielle Klimek-McDonald with the assistance of the undergraduate researchers acknowledged in the acknowledgement section of this dissertation.

Additional contributions included writing formatting, journal article submissions and technical advice from Dr. Julia King. Dr. Gregory Odegard provided project guidance and technical advice throughout this project. Technical advice was obtained from Dr. Ibrahim Miskioglu with regard to nanoindentation and tensile properties.

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This research was funded by NASA under the Subsonic Fixed Wing Program (Grant NNX11AO72A), without it this project would not have been made possible. I would like to thank XG Sciences Inc. and Asbury Carbons for donating the graphene nanoplatelets used for this work.

Thank you to all of the undergraduate student researchers that have worked with me over the last 4 years. I could not have done it without all of the help from the ~60 students that helped with the various aspects of this project.

I would like to thank my family, especially my parents, for all their love and support throughout the years. I would not be where I am today without their guidance. Despite being so far away they always found a way to give me support when I needed it.

Lastly, I would like to thank my loving husband, Jacob McDonald, for all of his help and support on this project. If I needed an extra set of hands he would always come to help.

Abstract

Due to their high specific stiffness, carbon-filled polymer composites are commonly used in the construction of structural components of subsonic fixed-wing aircrafts, such as the fuselage and control surfaces. In this work, neat epoxy (EPON 862 with EPIKURE Curing Agent W) was fabricated along with 1- 6 wt% of three types of GNP available from XG Sciences Inc. and 1-4 wt% of another type of GNP available from Asbury Carbons added to epoxy. The curing cycle for this epoxy was 121 °C for 2 hours followed by 177 °C for two hours.

GNP are short stacks of individual layers of graphite that are newly developed and available at a low cost. XG Sciences Inc. xGnP®-M-15 has a platelets diameter of 15 µm and a thickness of 7 nm. xGnP®-M-5 has a diameter of 5 µm and a thickness of 7 nm. The specific surface area for both M-grades is 130 m^2/g. xGnP®-C-300 has a diameter of 2 µm and a thickness of 2 nm with a specific surface area of 300 m^2/g. Asbury Carbon's TC307 GNP has a particle size <1 µm diameter and ~8 layers (1.1 nm) thick and a specific surface area of 350 m^2/g.

Development of good dispersion techniques was the most important contribution of this project. Proper dispersion is very important for obtaining a good composite. For each of the four types of GNP used in this study, a unique dispersion method was developed. High shear mixing was used in combination with sonication to exfoliate the GNP and disperse it into the epoxy matrix. An optical microscope was used to monitor the dispersion during mixing progression.

The composites were tested for their mechanical properties using typical macroscopic tensile testing, nanoindentation, and dynamic mechanical analysis. The addition of any of the four types of GNP resulted in an increase in the modulus (stiffness). The modulus can be predicted using the Halpin-Tsai model. The Halpin-Tsai model takes into account the mechanical properties of the polymer and the filler and also the filler geometry. The 2D randomly oriented filler Halpin-Tsai model was useful for predicting the modulus for xGnP®-M-15 in epoxy and xGnP®-M-5 in epoxy, this was confirmed visually through microscopy. The 3D randomly oriented filler Halpin-Tsai model predicted the modulus well for the xGnP®-C-300 and the TC307, microscopy visually confirmed that the filler was oriented in all three planes.

From the mechanical properties, one type of GNP was chosen for use in a unidirectional carbon fiber composite. The continuous carbon fiber composites were tested via macroscopic tensile tests. The GNP chosen did not have an effect on the composite's axial modulus, but increased the transverse modulus. The carbon fiber's mechanical properties dominate over the GNP/epoxy properties in the axial direction. As far as current literature available, continuous carbon fiber has never been used as a reinforcement for GNP/epoxy composites.

1 Introduction

1.1 Introduction and Motivation

Structural components of sub-sonic fixed wing aircrafts, such as fuselage and control surfaces, are commonly constructed using carbon-filled polymer composites. Polymer composites are used for aerospace applications because they have high specific (per unit mass) mechanical properties. There are many types of carbon available for use in carbon-filled polymer composites. Carbon nanotubes (CNT), carbon black, and graphite are a few of the common carbon materials used in polymer composites.

Carbon consists of laminar structure of carbon hexahedral lattice planes. There are two general classes of carbons, turbostratic (disordered laminar structure) and graphitic (ordered laminar structure). Graphite is a three dimensional allotrope of carbon. Graphite is the crystalline (ordered) form of elemental carbon and is derived from two sources, natural and synthetic. Natural graphite is mined from the earth. Synthetic graphite is manufactured carbonaceous material that is heat treated to temperatures above 2500 °C. There are three major types of synthetic graphite: primary graphite (such as electrodes for the electric arc furnace steel-making process), secondary graphite (typically recycled primary graphite), and carbon fibers. Two types of carbon fibers exist, petroleum pitch-based carbon fiber and polyacrylonitrile (PAN) -based carbon fiber, which differ in the feedstock used to produce them [1].

Individual layers of graphite, called graphene, have recently become commercially available. Graphite is made out of individual layers of graphene layers that are weakly bonded by van der Waals forces. Graphene nanoplatelets (GNPs) are short stacks of graphene sheets. GNP's are a low cost carbon filler ($15-$50/lb) that can be used to improve the composites tensile modulus and conductivity properties [2]. Carbon nanotubes can also be used to increase the tensile modulus, but have a much higher cost associated with them (~$50-$500/g) [1-6]. Improved composite mechanical properties are related to the fillers' aspect ratio as well as its surface-to-volume ratio [3]. GNP has a higher surface-to-volume ratio than carbon nanotubes due to lack of access to the inner surface of the nanotube [4].

The element carbon has the following orbitals: $1s^2$, $2s^2$, $2p^2$ (6 electrons total). Graphene has unique electronic orbitals: $1s^2\ 2s^1\ 2p_x^1\ 2p_y^1\ 2p_z^1$. In the graphene hexagonal lattice, each carbon atom is bonded to 3 other carbon atoms by the overlap of three sp^2 hybrid bonds. The sp^2 hybridization between the s orbital and the two p orbitals leads to a trigonal planar structure with a formation of a sigma bond between carbon atoms separated by 1.42 angstroms. These bands have a filled shell. The sp^2 orbitals arise from the hybridization of $2s^1$, $2p_x^1$ and $2p_y^1$ electrons. Thus, each carbon keeps a pure p_z orbital (perpendicular to the planar structure) that can bind covalently with neighboring carbon atoms, causing the formation of a pi bond. This hybridization results in the following electron orbitals: $1s^2\ (sp^2)^3\ 2p_z^1$. Often functional groups containing oxygen, nitrogen, etc. can be added to the edges of graphite [1, 5].

The unique electron orbitals of graphene is important for the load transfer between the filler and the matrix. Load transfer can occur when there is micromechanical interlocking of the filler and the matrix, chemical bonding between the filler and the matrix, or is there is van der Waals bonding between the filler and the matrix. Interfacial shear results in load transfer. If the load transfer is poor then the other mechanical properties will suffer. Nanostructured materials have been shown the increase the stiffness of a composite, but they also can reduce the strength and elongation of a composite.

There are two primary nanostructured carbon fillers, carbon nanotubes and graphene nanoplatelets. Nanostructured materials are characterized by having at least one constituent whose character length that is tens of nanometers or smaller. The literature contains many references concerning adding carbon nanotubes to epoxy to increase the tensile modulus [6-9]. GNP is a newly developed low cost material that is a very promising alternative to carbon nanotubes that also increases the tensile modulus [2, 10-15]. Specifically, Fukushima et al. demonstrated that the tensile modulus from ~2.75 GPa to ~3.1 GPa for 3 vol% GNP/ 97 vol% epoxy. Schadler et al. showed an increase from 3.1 GPa for neat epoxy to 3.71 GPa for 5 wt% CNT/95 wt% epoxy.

Tensile modulus models are useful predicting the behavior of a composite. The Halpin-Tsai model is a widely accepted model for predicting the tensile modulus for discontinuous carbon fibers. The model can be adapted for many different filler geometries, platelets being one of them. The Halpin-Tsai model takes into account the tensile properties of the polymer matrix and the filler as well as the filler geometry [16-19]. It is useful to have models that will predict the mechanical properties of composites so that time and money is not wasted on fabrication and testing.

In order to make aircrafts more fuel efficient, the material used for their fabrication needs to be lighter. Carbon-filled polymer composites are lighter than the metals they replace, and have the potential to be just as strong. This research was funded by NASA under the Subsonic Fixed Wing Program (Grant NNX11AO72A). The epoxy (polymer) chosen for this study was Momentive EPON^{TM} Resin 862 with EPIKURE Curing Agent W with GNP as the carbon filler and continuous carbon fiber for structural reinforcement. Very few studies have been conducted on this particular epoxy with this curing agent. The mechanical properties of GNP added to this particular epoxy composites have not been determined. As far as current literature available, continuous carbon fiber has never been used as a reinforcement for GNP/epoxy composites. It is believed that adding GNP to epoxy will increase the tensile modulus. This should also increase the overall stiffness of a continuous carbon fiber composite.

1.2 Objectives

There is one primary goal for this project, to characterize GNP/epoxy and continuous CF/GNP/epoxy composites in terms of their tensile properties (stress, strain, and modulus). The specific objectives of this project are listed below.

- Objective 1: Graphene nanoplatelet (GNP)/epoxy composites were fabricated and tested for mechanical properties (measured by ASTM D638 and nanoindentation).
- Objective 2: Continuous carbon fiber/GNP/epoxy composites were fabricated and tested for mechanical properties (measured by ASTM D3039).
- Objective 3: Based on the data collected in Objectives 1 and 2, Halpin-Tsai models will be were applied to predict the tensile modulus.

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2 Materials

2.1 Materials

For this project there were four different graphene nanoplatelets, one continuous carbon fiber, and one epoxy matrix studied. The epoxy matrix analyzed was Momentive EPON™ Resin 862 with EPIKURE Curing Agent W. The carbon fillers studied were: XG Sciences xGnP®-M-15, xGnP®-M-5, xGnP®-C-300 graphene nanoplatelets, Asbury Carbons ThermoCarb TC307 graphene nanoplatelets, and Hexcel HexTow® AS4-GP-3K Continuous Carbon Fiber. The following sections will cover each material in more detail.

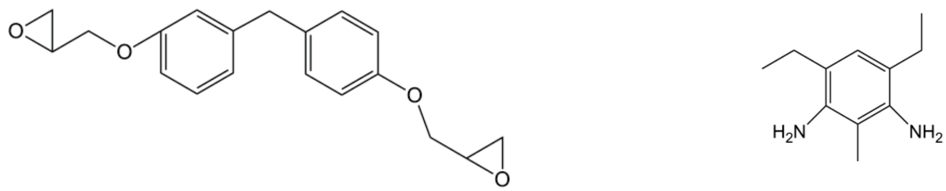
2.2 Matrix Material

The epoxy matrix used in this study was Momentive EPON™ Resin 862 (diglycidyl ether of bisphenol F, DGEBF) with EPIKURE Curing Agent W (diethyltoluenediamine, DETDA) (EPON 862/EPIKURE W). The chemical structures of EPON™ Resin 862 and EPIKURE Curing Agent W are shown in Figure 2-1. EPON™ Resin 862 is a low viscosity, liquid epoxy resin manufactured from epichlorohydrin and bisphenol F [1]. The formation of EPON™ Resin 862 from Bisphenol F is shown in Figure 2-2. The amine groups on the EPIKURE Curing Agent W react with the epoxide groups on the EPON™ Resin 862 as the reaction continues the system becomes a branched crosslinked structure as shown in Figure 2-3. EPON 862/EPIKURE W is a thermoset epoxy system with a curing cycle of 121 °C for 2 hours followed by 177 °C for

2 hours. Table 2-1 contains physical properties of the EPON^{TM} Resin 862 and EPIKURE Curing Agent W.

Table 2-1: Properties of Momentive's EPON^{TM} Resin 862 and EPIKURE Curing Agent W [1]

Tensile Strength	78.6 MPa
Tensile Modulus	2.72 GPa
Tensile Strain	7.1%
Density	1.20 g/cm^3



EPON^{TM} Resin 862 EPIKURE Curing Agent W

Figure 2-1: EPON^{TM} Resin 862 and EPIKRUE Curing Agent W Structures -drawn from chemical name given in vendor literature [1]

Epoxide Bisphenol F EPON^{TM} Resin 862

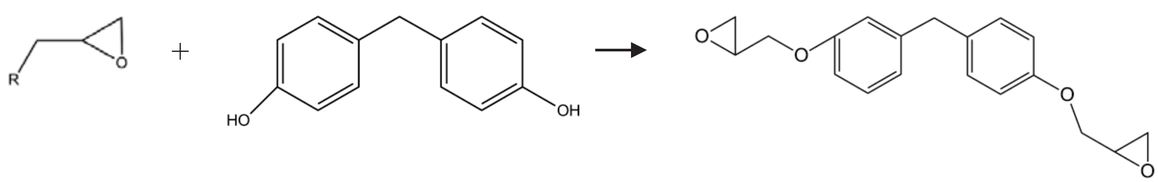


Figure 2-2: EPON^{TM} Resin 862 formation from Bisphenol F

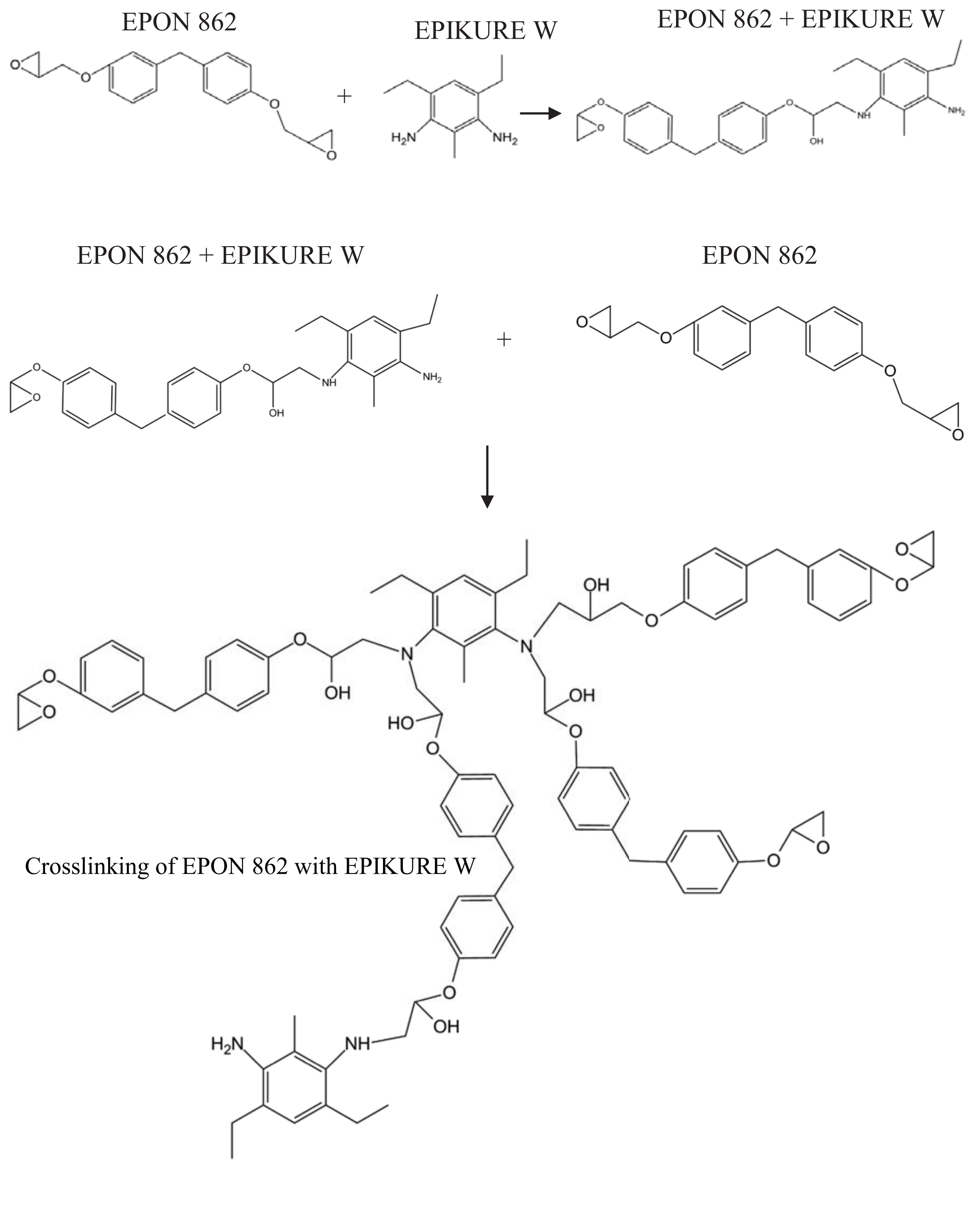


Figure 2-3: Crosslinking of EPON™ Resin 862 with EPIKURE Curing Agent W

2.3 Fillers

2.3.1 Graphene Nanoplatelets (GNP)

Graphene nanoplatelets (GNP) are short stacks of graphene sheets. A graphene sheet is defined as a single layer of graphite. GNPs are available in many different particle diameters and thicknesses, they can have various surface areas and surface modifications. The structure of graphene can be seen in Figure 2-4.

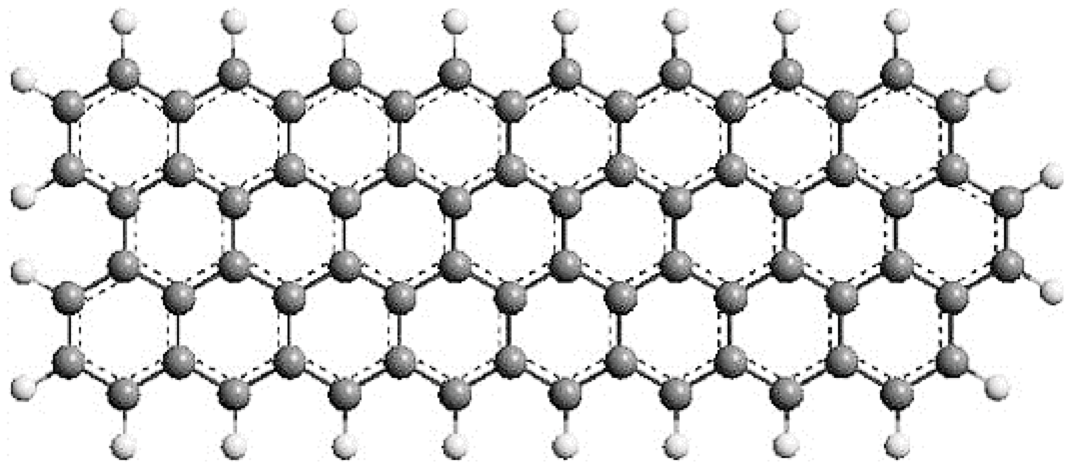
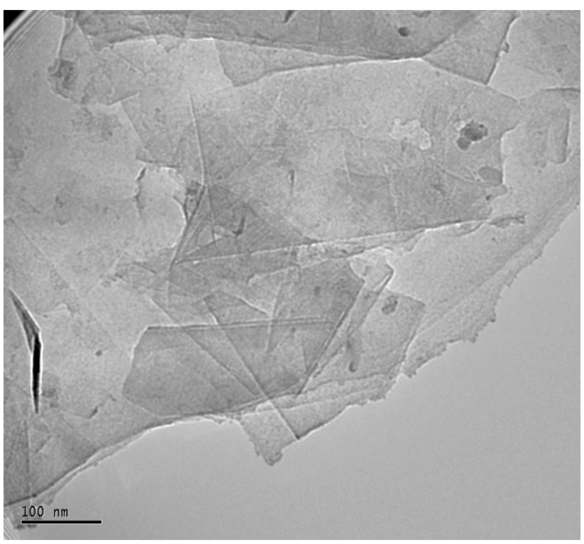


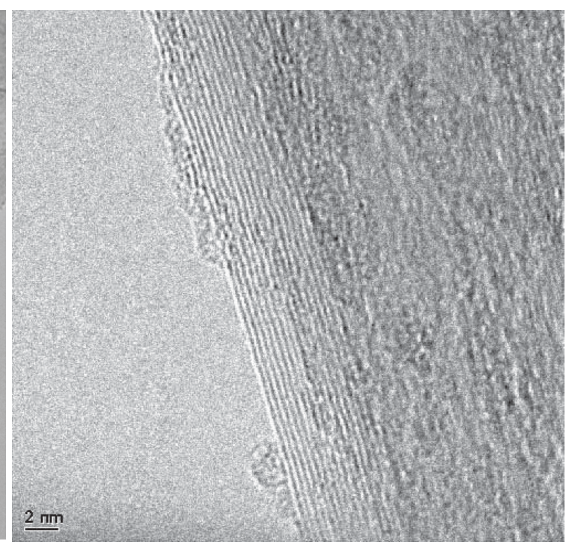
Figure 2-4: Graphene Structure

Three of the four GNPs are from XG Sciences. XG Sciences has two different grades of GNP. The first type is M grade, which are manufactured by heating Graphite Intercalated Compounds (GIC). First, the GICs were fabricated by intercalating mixtures of acids into natural graphite flakes. Then the GIC samples were produced thermally. After the treatment, these graphite flakes showed significant expansions due to the vaporization of intercalated acids in between the graphite layers. The expanded graphite flakes were then pulverized to a specific size. The average thickness and size of the xGnP® can be controlled by changing the process conditions. Two types of M Grade xGnP® were used. The first type is xGnP®-M-15, which has an average particle diameter of 15 microns and a thickness of 7 nm. The second type is xGnP®-M-5, which has an average diameter of 5 microns, and a thickness of 7 nm. The specific surface area of both M grade materials is 120-130 m^2/g.

The second grade that XG Sciences manufactures is, C grade xGnP®, which is made by a different proprietary process. The type used for this study was xGnP®-C-300, which has an average particle diameter of 2 microns and ~2 nm thickness, the specific surface area is ~300 m^2/g. C grade materials are smaller and thinner, because of this they have more edge groups present so there are more functional groups [2]. TEM images of xGnP® can be seen in Figure 2-5.



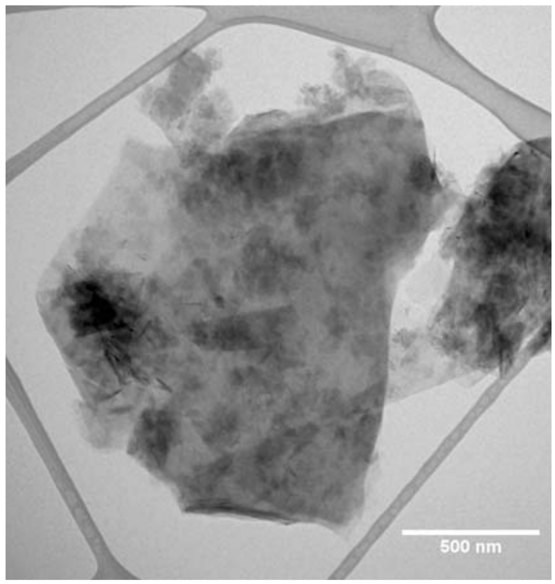
A: Scale bar = 100 nm



B: Scale bar = 2nm

Figure 2-5: TEM images of xGnP® [2]

The last GNP used is available from Asbury. Asbury TC307 GNP are manufactured using a proprietary process. The parent carbon used in this particular GNP material is a high quality calcined needle coke, which is heat treated to temperatures above 2500 °C to effect graphitization. A transmission electron micrograph of TC307 can be seen in Figure 2-6. TC307 has an average primary particle diameter of below 1 µm, a density of ~2 g/mL, and a nominal specific surface area of 350 m^2/g. TC307 has a nominal primary lamella thickness of ~8 (~1.1 nm) graphene layers [3].



Scale bar = 500 nm

Figure 2-6: Transmission Electron Micrograph of Asbury TC307 [4]

2.3.2 Hexcel HexTow® AS4-GP-3K Continuous Carbon Fiber

The continuous carbon fiber used in this analysis is fabricated by Hexcel. Hexcel's HexTow® AS4-GP-3K is a PAN (polyacrylonitrile) based carbon fiber, containing 3,000 fibers per bundle (3K tow) [5]. There is 1 wt% sizing consisting of a proprietary, uncured epoxy resin. The properties are shown in

Table ***2-2***.

Table 2-2: Properties of Hexcel HexTow® AS4-GP-3K continuous carbon fiber [5]

Tensile Strength	4,620 MPa
Tensile Modulus	231 GPa
Density	1.76-1.82 g/cm^3
Fiber Diameter	7.3 microns

2.4 Formulation Naming Convention

To make sample identification simpler, a naming convention has been created. Most samples look very similar to one another so it is important to label every sample with a unique name. Labels were applied after fabrication. The naming system is as follows:

A862-AS4– b – w – date – z - #

Where:
A= project description (NASA project)
AS4= HexTow® AS4-GP-3K, if not used in the composite then do not use in naming
b= filler type (M15= xGnP®-M-15, M5= xGnP®-M-5, C300= xGnP®-C-300, TC307= Asbury TC307), if none are contained in the composites then do not use in naming
w= weight percent of xGnP® graphene nanoplatelets.
date= date of specimen fabrication
z= specimen type (T= tensile bar, D= electrical resistivity disks)
#= specimen number

An example of the naming system would be A862-AS4-C300-2-11-8-13-T-8. This would represent the 8^{th} tensile bar containing HexTow® AS4-GP-3K continuous carbon fiber and 2 wt % xGnP®-C300 fabricated on November 8, 2013.

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3 Fabrication and Experimental Methods

3.1 Fabrication Methods

For each type of GNP an optimum mixing procedure had to be developed. In the earlier stages this was done through trial and error. The mixing speed and time were varied and samples were tested to see if the optimum properties were obtained. This is how the mixing procedures for the xGnP®-M-15 and xGnP®-M-5 were found. The xGnP®-C-300 proved to be a challenge for mixing. The xGnP®-C-300 size properties were drastically different from the two M-grades that the trial and error method would not work.

For the xGnP®-C-300 the mixing rate was set as high as it would go before the mixture began to splatter out of the beaker (3500 rpm). To find the optimal mixing time for the xGnP®-C-300 an optical microscope was used. Samples were taken from the batch as it was mixing at 10 min time intervals. A pea sized sample was flattened between two microscope slides and the xGnP®-C-300 was viewed under the microscope at 500x magnification. Once the GNP was no longer getting visibly smaller, the mixing was complete. It was also found that sonication helped to exfoliate the GNP further. Sonication was tried on the M-grade GNPs, but it did not make a difference. The same procedure used for the xGnP®-C-300 was then applied to disperse the TC307 GNP in epoxy. The finalized mixing procedures are described in the following subsections.

3.1.1 Neat epoxy test specimen fabrication

To fabricate the neat epoxy, 100 g of EPON 862 was added to 26.4 g of EPIKURE Curing Agent W at 23°C and mixed by hand for 3 minutes. The mixture was degassed inside an oven at 90°C and 29 inches Hg vacuum for 30 min and then poured into rectangular molds. The molds were heated in an oven to 121°C over 30 min, held at 121°C for 2 h, heated to 177°C over 30 min, held for another 2 h at 177°C, and finally cooled to ambient temperature [1-3].

3.1.2 xGnP®-M-15/epoxy test specimen fabrication

To produce the xGnP®-M-15/epoxy composites, the appropriate amount of xGnP®-M-15was added to EPON 862 and mixed using a 2 in diameter disperser blade in a Ross high shear mixer HSM-100 LSK-I (see Figure 3-1) at 2500 rpm for 40 minutes. The corresponding amount of EPIKURE Curing Agent W (100 g EPON 862 added to 26.4 g of EPIKURE Curing Agent W) was added to the xGnP®-M-15/EPON mixture and stirred with the Ross mixer at 1000 rpm for 3 minutes at 23°C. The mixture was degassed inside an oven at 90°C and 29 inches Hg vacuum for 30 min and then poured into rectangular- and disc-shaped molds. The same curing cycle was used as described for the neat epoxy. For the neat epoxy and the xGnP®-M-15/epoxy systems, the fabricated samples were rectangular bars (165 mm long by 19 mm wide by 3.3 mm thick) [4].

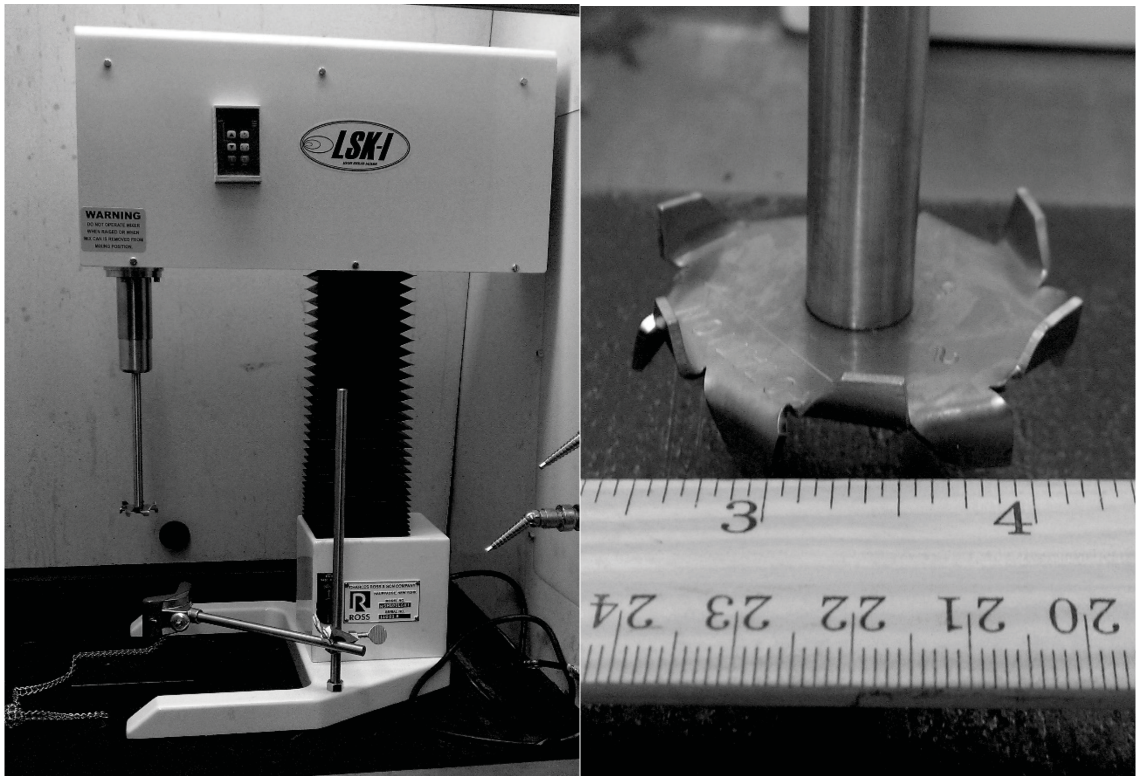

Figure 3-1: (left) Ross high shear mixer HSM-100 LSK-I with (right) 2” dispersion mixing blade

3.1.3 xGnP®-M-5/epoxy test specimen fabrication

To produce the xGnP®-M-5/epoxy composites, the appropriate amount of xGnP®-M-5was added to EPON 862 and mixed using a 2 in diameter disperser blade in a Ross high shear mixer HSM-100 LSK-I at 2500 rpm for 20 minutes. The corresponding amount of EPIKURE Curing Agent W (100 g EPON 862 added to 26.4 g of EPIKURE Curing Agent W) was added to the xGnP®-M-5/EPON mixture and stirred with the Ross mixer at 1000 rpm for 3 minutes at 23°C. The mixture was degassed inside an oven at 90°C and 29 inches Hg vacuum for 30 min and then poured into rectangular- and disc-shaped molds. The same curing cycle was used as described for the neat epoxy. For the xGnP®-M-5/epoxy system the fabricated samples were rectangular bars (165 mm long by 19 mm wide by 3.3 mm thick) [5].

3.1.4 xGnP®-C-300/epoxy test specimen fabrication

To produce the xGnP®-C-300/epoxy composites, the appropriate amount of xGnP®-C-300 was added to EPIKURE Curing Agent W (100 g EPON 862 added to 26.4 g of EPIKURE Curing Agent W) and mixed using a 2 in diameter disperser blade in a Ross high shear mixer HSM-100 LSK-I at 3500 rpm for 150 minutes. The mixture was then placed in a Branson Bath Sonicator CPX2800H (see Figure 3-2) operating at 40 kHz for 60 minutes at 23°C. The corresponding amount of EPON 862 was added to the xGnP®-C-300/Curing Agent W mixture and stirred with the Ross mixer at 1000 rpm for 3 minutes at 23°C. The mixture was degassed inside an oven at 90°C and 29 inches Hg vacuum for 30 min and then poured into rectangular- and disc-shaped molds. The same curing cycle was used as described for the neat epoxy. For the xGnP®-C-300 /epoxy system the fabricated samples were rectangular bars (165 mm long by 19 mm wide by 3.3 mm thick) [5].

Figure 3-2: Branson Bath Sonicator CPX2800H

3.1.5 Asbury TC307/epoxy test specimen fabrication

To produce the Asbury TC307/epoxy composites, the appropriate amount of TC307 was added to EPON 862 and mixed using a 2 in diameter disperser blade in a Ross high shear mixer HSM-100 LSK-I at 3000 rpm for 60 minutes. The mixture was then placed in a Branson Bath Sonicator CPX2800H operating at 40 kHz for 60 minutes at 23°C. The corresponding amount of EPIKURE Curing Agent W (100 g EPON 862 added to 26.4 g of EPIKURE Curing Agent W) was added to the TC307/Curing Agent W mixture and stirred with the Ross mixer at 1200 rpm for 10 minutes at 23°C. The mixture was degassed inside an oven at 90°C and 29 inches Hg vacuum for 30 min and then poured into rectangular- and disc-shaped molds. The same curing cycle was used as described for the neat epoxy. For the TC307 /epoxy system the fabricated samples were rectangular bars (165 mm long by 19 mm wide by 3.3 mm thick).

3.1.6 AS4 carbon fiber/epoxy test specimen fabrication

To fabricate the continuous unidirectional carbon fiber/epoxy composites, 100 g of EPON 862 was added to 26.4 g of EPIKURE Curing Agent W at 23 °C and mixed by hand for 3 minutes. The corresponding amount of epoxy (100 g EPON 862 added to 26.4 g of EPIKURE Curing Agent W) and carbon fiber were mixed to produce a unidirectional composite containing 67 wt% carbon fiber and 33 wt% epoxy. The uncured epoxy/carbon fiber was cut into sheets (248 mm by 248 mm) and placed in a picture frame mold. To fabricate the unidirectional composite plate, five plies were placed with the carbon fiber in the 0° direction. A Wabash Compression Molding Machine Vantage Series Model V75H-18-CLX (see Figure 3-3) was used. Initially, the composite plate was heated to 121 °C and held at a constant pressure of 30 psi for 2 hours. The press was then ramped up to 177 °C and held at a constant pressure of 1000 psi for 2 hours. Cooling water was used to cool the press until the platen temperature was 30°C, then the composite plate (1.7 mm thick) was removed [6].

Figure 3-3: Wabash Compression Molding Machine Vantage Series Model V75H-18-CLX

3.1.7 AS4 carbon fiber/ xGnP®-C-300/epoxy test specimen fabrication

To fabricate the xGnP®-C-300/carbon fiber/epoxy hybrid composites, the appropriate amount of xGnP®-C-300 was added to 26.4 g EPIKURE Curing Agent W and mixed using a 2 in diameter disperser blade in a Ross high shear mixer HSM-100 LSK-I at 3500 rpm for 150 minutes. Next the mixture was placed in a Branson Sonicator CPX2800H operating at 40 kHz for 60 minutes at 23°C. The corresponding amount of epoxy (100 g EPON 862 added to 26.4 g of EPIKURE Curing Agent W) was added to the xGnP®-C-300/ Curing Agent W mixture and stirred with the Ross mixer at 1000 rpm for 3 minutes at 23°C. The carbon fiber was strung through a resin bath containing xGnP®-C-300/

epoxy to produce a unidirectional continuous carbon fiber composite containing the following compositions:

- 1 wt% xGnP®-C-300/67 wt% carbon fiber/32 wt% epoxy
- 2 wt% xGnP®-C-300/67 wt% carbon fiber/31 wt% epoxy
- 3 wt% xGnP®-C-300/67 wt% carbon fiber/30 wt% epoxy

The uncured xGnP®-C-300/carbon fiber/epoxy composite was cut into sheets and cured as described for the neat epoxy [6].

3.2 Experimental Test Methods

3.2.1 Neat Epoxy and GNP/Epoxy Mechanical Tensile Property Test Method

The neat epoxy and GNP/epoxy composites were tested for tensile properties at ambient conditions according to ASTM D638 at a crosshead rate of 1 mm/min. The test specimens are ASTM Type I sample geometry, 165 mm length with 3.3 mm thickness. An InstruMet Sintech screw-driven mechanical testing machine was used, shown in Figure 3-4. Stress results are recorded by the testing machine and an extensometer was used to collect the strain values.

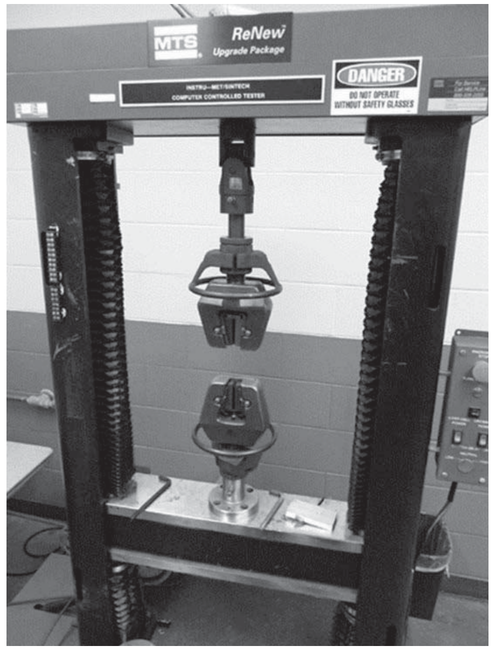

Figure 3-4: Instru-Met Sintech mechanical testing machine with tensile apparatus installed

ASTM D638 was used to determine the tensile strength, strain, and modulus [7]. Modulus is determined by the initial slope of the stress-strain curve. At least 5 samples of each composite were tested. The samples were conditioned at 23 °C and 50% relative humidity for 2 days prior to testing.

3.2.2 Continuous Carbon Fiber Mechanical Tensile Property Test Method

The continuous carbon fiber composites were tested for tensile properties at ambient conditions according to ASTM D3039 at a crosshead rate of 2 mm/min [8]. The samples were cut to 165 mm length, 12.5 mm width, and 3.3 mm thickness. An InstruMet Sintech screw-driven mechanical testing machine was used. Stress results are recorded by the testing machine and an extensometer was used to collect the strain values.

ASTM D3039 was used to determine the tensile strength, strain, and modulus. Modulus is determined by the initial slope of the stress-strain curve. At least 6 samples of each composite were tested. The samples were conditioned at 23 °C and 50% relative humidity for 2 days prior to testing.

3.2.3 Nanoindentation Test Method

Nanoindentation tests were performed on samples cut from untested tensile specimens for all composite formulations, 10mm x 10 mm x 3.3 mm. The samples were mounted in an epoxy puck and tested with an Agilent Nano Indenter XP, shown in Figure 3-5. The typical test was run to a depth of 1500 nm and data was recorded at a rate of 5 Hz.

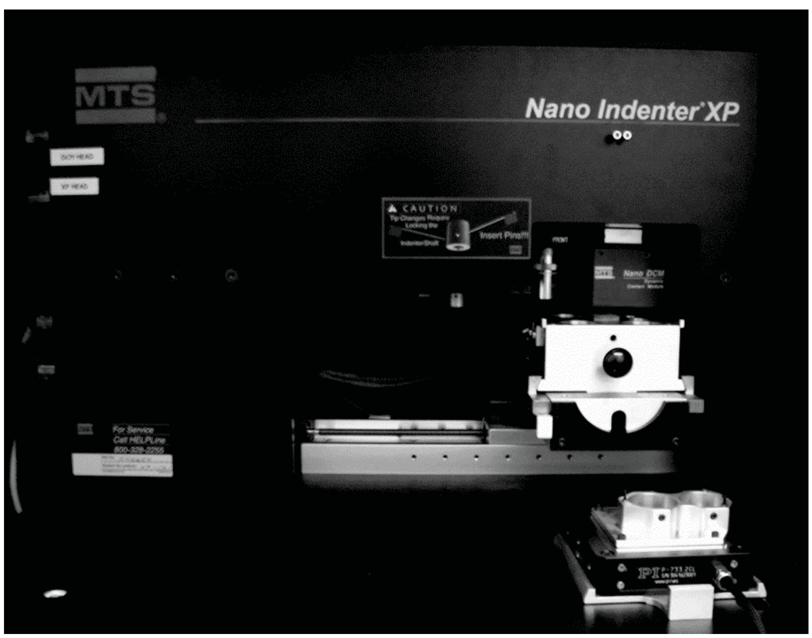

Figure 3-5 Agilent Nano Indenter XP

For each sample, 36 indents were made in a 6 x 6 pattern with 50 µm spacing in both directions. A Berkovich indenter was used for the tests. Data collected included load on the sample, penetration of the indenter, hardness of the sample, and modulus. The modulus (E) and hardness (H) of the sample was calculated using the contact stiffness per Oliver-Pharr method [9]. In general, the modulus was obtained from the slope of the load-displacement curve during unloading. The approach results in the calculation of E and H at the maximum indentation depth. Data collected was also accomplished by the continuous stiffness method (CSM), in which a small oscillation was superimposed on the primary loading. This method allows determination of E and H as a continuous function of the indenter penetration. The frequency of the oscillations was set at 45 Hz for the CSM method.

Creep tests were also conducted on all composite formulations. The creep loads were set at 2, 5, 10, 15, 25, 35, and 45 mN. The load was increased to the creep load at a rate of 1 mN/s and held at the creep load for 150 s. The creep data was analyzed following the method proposed by Tehrani et al. [10]. The relation between strain and the creep load is described in Equation 3-1.

$$\varepsilon(t) = \sigma_0 J(t) \tag{3-1}$$

where J(t) is the creep compliance and is defined in Equation 3-1.

$$J(t) = \frac{A(t)}{(1-\upsilon)P_0 \tan(\theta)} \tag{3-2}$$

In Equation 3-2, A(t) is the contact area, υ is Poisson's ratio =0.35 for epoxy [9-10], P_0 is the constant applied load (2, 5, 10, 15, 25, 35, and 45 mN), and θ is the effective cone angle (70.3° for Berkovich indenter). The approach takes into account how the contact area under the Berkovich indenter changes as displacement into the surface changes.

3.2.4 Mounting Samples in Epoxy Puck

A sample must be cut to the size desired to be mounted in epoxy. To prepare the mold cups, mold cups are sprayed with Mann Ease Release 300 (mold release) then aluminum foil is pressed smoothly into the bottom of the cup. Carefully remove the bottom of the cup with the aluminum foil taking care not to add wrinkles to the aluminum foil. A small piece of carpet (double stick) tape is placed onto the aluminum foil. A Buehler® Sampl-Klip®I is wrapped around the cut sample and the face of interest is placed face down on the tape. Carefully replace the top part of the mold cup.

To mix up the epoxy 12 g of Aka-Cure is added to 100 g of Aka-Resin. These are weighed into a paper cup and mixed by hand until no streaks are visible. Approximate 15 g of epoxy is poured into each mold cup and are allowed to cure at room temperature overnight. To remove the epoxy mounts, open the bottom and peel the aluminum foil and tape off. Place the mold cups upside down and use the handle of a screwdriver and press firmly until the epoxy mount releases.

3.2.5 Grinding

Using a 320 grit SiC paper on a Diamond Pacific rotating lap with a water drip (See Figure 3-6) grind the edges of the epoxy mount to a smooth finish so that there are no sharp edges. If a meniscus was formed then grind the sample flat. Using the same grit, grind the epoxy mounts down until the sample surface is exposed. Once all the samples surfaces are exposed, place all the epoxy mounts into a ten sample holder shown in Figure 3-7. If there are not ten samples to fill the holder use 'blank' samples to fill the sample holder.

Figure 3-6: Diamond Pacific rotating lap with water drip apparatus

Figure 3-7: 10–Sample Holder

Once the samples are secured in the holder grind them until all the surfaces are at the same level. Place sample holder in a bath sonicator for about 5 min. Meanwhile, remove the 320 grit paper and replace with a 600 grit SiC paper. Grind samples with a water drip on the 600 grit paper until no more visible scratches from the 320 grit paper. Place the sample holder in the bath sonicator again for about 5 min. Remove the 600 grit paper and replace with a 1200 grit SiC paper. Grind samples once more with the water drip until no more visible scratches from the 600 grit paper. Put the sample holder back in the sonicator and run for about 10-15 min.

3.2.6 Polishing

After sonication rinse the samples with distilled water. Place the 9 µm (red) platen on the Buhler Ecomet 4 variable speed grinder-polisher (Figure 3-8), shake the 9 µm (red) diamond suspension until no more diamonds are visible on the bottom of the spray bottle. Spray on platen and then insert the sample holder into the head. Set the rotation speed to 150 rpm, set the force to 6 lb and use the counter clockwise rotation for the head. Run the 9 micron polish until no more visible scratches from the 1200 grit paper.

Figure 3-8: Buhler Ecomet 4 variable speed grinder-polisher

Rinse the sample holder with distilled water and place into bath sonicator (be sure to replace water and clean the grit out before this step). Remove the 9 μm (red) platen and replace with the 3 μm (green) platen. Shake the 3 μm (green) diamond suspension until there are no more visible diamonds on the bottom of the spray bottle. Spray on platen and then insert the sample holder into the head. Set the rotation speed to 120 rpm, set the force to 5 lb and use the counter clockwise rotation for the head. Run the 3 micron polish until no more visible scratches from the 9 micron polish.

Rinse the sample holder with distilled water and place into bath sonicator. Remove the 3 μm (green) platen and replace with the 1 μm (white) platen. Shake the 1 μm (white) alumina suspension until well mixed. Squirt on platen and then insert the sample holder into the head. Set the rotation speed to 120 rpm, set the force to 5 lb and use the counter clockwise rotation for the head. Run the 1 micron polish until no more visible scratches from the 3 micron polish.

Remove the samples from the sample holder and rinse with distilled water and place into bath sonicator taking care to place the wrong side of the samples face to face and the polished sides face to face in a repeating pattern. Remove the 1 μm (white) platen. Shake the 0.05 μm (white) alumina suspension until well mixed. Place a MasterTex cloth on a Buehler Vibromet 1 vibratory polisher. (Note: May have to clean the Kerosene from the Vibromet first). Pour 0.05 micron slurry onto the cloth, place the samples into the Vibromet single sample holders (can only run 4 at a time). Put the samples face down into the slurry and put the cover over the Vibromet. Turn up the intensity of the table slowly with the knob until about 7-8 on the dial. Let the samples 'dance' around the edge for about 2 hours.

Remove the samples from the sample holders and rinse with distilled water. Clean the bath sonicator once more and replace with fresh water. Put the samples in the sonicator as mentioned before (face to face and end to end) sonicate for 15 min. Remove the samples and rinse with distilled water, then rinse well with ethanol, and allow to dry. Place a red cap on top so that the surface will not get scratched in transport.

Table 3-1: Grinding and Polishing Steps

Lubricant	Abrasive Type/Size	Paper/Cloth	Machine	Time	Increments	Force per Specimen (lb)	Speed (RPM)	Relative Rotation
Water	SiC – 320 grit	320 grit SiC	Grind Wheel	30 min	10 min			
Water	SiC – 600 grit	600 grit SiC	Grind Wheel	30 min	10 min			
Water	SiC – 1200 grit	1200 grit SiC	Grind Wheel	45 min	15 min			
Red Soln.	METADI Supreme – 9 µm	ULTRAPOL	Ecomet	30 min	5 min	6	150	Contra
Green Soln.	METADI Supreme – 3 µm	Texmet 1000	Ecomet	30 min	5 min	5	120	Contra
White Soln.	Alumina Suspension - 1 µm	Microcloth	Ecomet	30 min	5 min	5	120	Contra
White Soln.	Master Prep – 0.05 µm	MASTERTEX	Vibromet	2 hr	30 min			

3.2.7 Through-Plane Electrical Resistivity Test Method

For samples with an electrical resistivity > 10^{10} Ω-cm, ASTM D257 was used to measure the volumetric electrical resistivity at 23 °C [12]. In this method a constant voltage, typically 100 V, was applied to the test specimen. Resistivity was measured using a Keithley 6517A Electrometer/High Resistance Meter with an 8009 Resistivity Test Fixture (see Figure 3-9). The test specimen tested were disks that were cut to 6.4 cm in diameter and 3 mm thick. At least 4 samples were tested for each formulation. Two days prior to testing, the samples were conditioned at 23 °C and 50% relative humidity.

Figure 3-9: (left) Keithley 6517A Electrometer/High Resistance Meter, (right) Keithley 8009 Resistivity Test Fixture

3.2.8 In-Plane Electrical Resistivity Test Method

For samples with an electrical resistivity < 10^{10} Ω-cm, ASTM D4496 was used to measure the in-plane volumetric electrical resistivity of a rectangular bar at 23 °C [13]. Two probes were used to conduct the tests. The test specimen tested were bars (12.5 mm wide and 3 mm thick) that were cryogenically broken to 60 mm long, this creates a fracture surface at both ends. The fracture surfaces were painted with silver paint. At least 4 samples were tester for each formulation. Two days prior to testing, the samples were conditioned at 23 °C and 50% relative humidity. One probe was placed on each silver-painted fracture surface, and a constant voltage was applied. The voltage source was a Keithley 2400 Source Meter. The resulting current was measured and the volume electrical resistivity was calculated using Equation 3-3.

$$\mathrm{ER} = \frac{(\Delta \mathrm{V})(\mathrm{w})(\mathrm{t})}{(\mathrm{i})(\mathrm{L})} \qquad (3\text{-}3)$$

Where ER is the volume electrical resistivity (Ω-cm), ΔV is the voltage (volts), w is the sample width (cm), t is the sample thickness (cm), i is the current (amps), and L is the length which ΔV is measured across (cm).

3.2.9 Differential Scanning Calorimeter (DSC) Test Method

A Metter Toledo 823E differential scanning calorimeter (see Figure 3-10) was used to measure the glass transition temperature (Tg) of every composite. Approximately 10 mg of 'crushed' epoxy sample was placed in a sealed aluminum pan with a 'vent hole' in the lid. Nitrogen purge gas was used. The following heating cycle was used and Tg was determined on the second heating cycle. Tg was determined by a change in heat flow (i.e. heat capacity).

The following heating method was used:

1) Heat 40°C to 180°C at 10°C/min

2) Cool 180°C to 40°C at 10°C/min

3) Hold 40°C and hold for 5 min

4) Heat 40°C to 180°C at 10°C/min

Figure 3-10: Metter Toledo 823E Differential Scanning Calorimeter

3.2.10 Dynamic Mechanical Analyzer (DMA) Test Method

The DMA applies a sinusoidal deformation to a sample of known geometry and measures the stiffness and dampening which it reports as modulus and tan delta. The storage modulus is a representation of the stored energy and shows the elastic portion of the epoxy resin. The loss modulus measures the energy dissipated as heat which represents the viscous portion of the epoxy resin. The ratio comparison of the two moduli is known as the tan delta, which can be used to determine the glass transition temperature. The glass transition temperature is the transition of a polymer from the "glassy" state to the "rubbery" state. This is represented as a range of temperatures determined from the storage modulus onset, peak of the loss modulus curve, and the peak of the tan delta curve. The following equations (Equations 3-4, 3-5, and 3-6) are used to calculate the storage modulus, loss modulus, and tan delta for the epoxy resin.

Storage Modulus: $$E' = \frac{\sigma_o}{\varepsilon_o}\cos(\delta) \tag{3-4}$$

Loss Modulus: $$E'' = \frac{\sigma_o}{\varepsilon_o}\sin(\delta) \tag{3-5}$$

Tan Delta: $$\tan(\delta) = \frac{E''}{E'} \tag{3-6}$$

$\sigma = \text{stress}$ $\varepsilon = \text{strain}$ $\delta = \text{phase lag between stress and strain}$

The sample tests were completed on the TA Instruments Q800 DMA (see Figure 3-11 left) using the dual/single cantilever clamp (Figure 3-11 right), for testing the single cantilever was used. The sample thickness and width was measured with calipers and entered into the program along with a set length of 17.5 mm (distance between clamps). The actual sample dimensions were 3 mm thick, 12 mm wide, and 35 mm long, cut from bars with 3 mm thickness, 25 mm width, and 150 mm long. Using tweezers so as not to touch the sample, it was carefully loaded into the clamps and tightened down using 9 in-lbs of torque. The tweezers were used to prevent oil from getting on the sample due to contact with hands and fingers.

Figure 3-11: left) TA Instruments Q800 DMA right) Dual/single cantilever clamp

Once the sample was in place in the clamps, an amplitude test was performed to determine if the machine and program were operating correctly. The test mode was chosen to be “DMA Multi Strain” with the test set to “Strain Sweep”. The frequency was kept at 1 Hz with the isothermal temperature at 35^{o}C and a soak time of 5 minutes, the amplitude was swept from 5 μm to 50 μm. Following the test, the graph of amplitude versus loss modulus was analyzed to determine the amplitude needed for the temperature sweep test. The amplitude that is chosen should be in the linear portion of the amplitude loss modulus plot. This linearity is shown in Figure 3-12, along with the 30 μm amplitude chosen, this amplitude was used for all formulations.

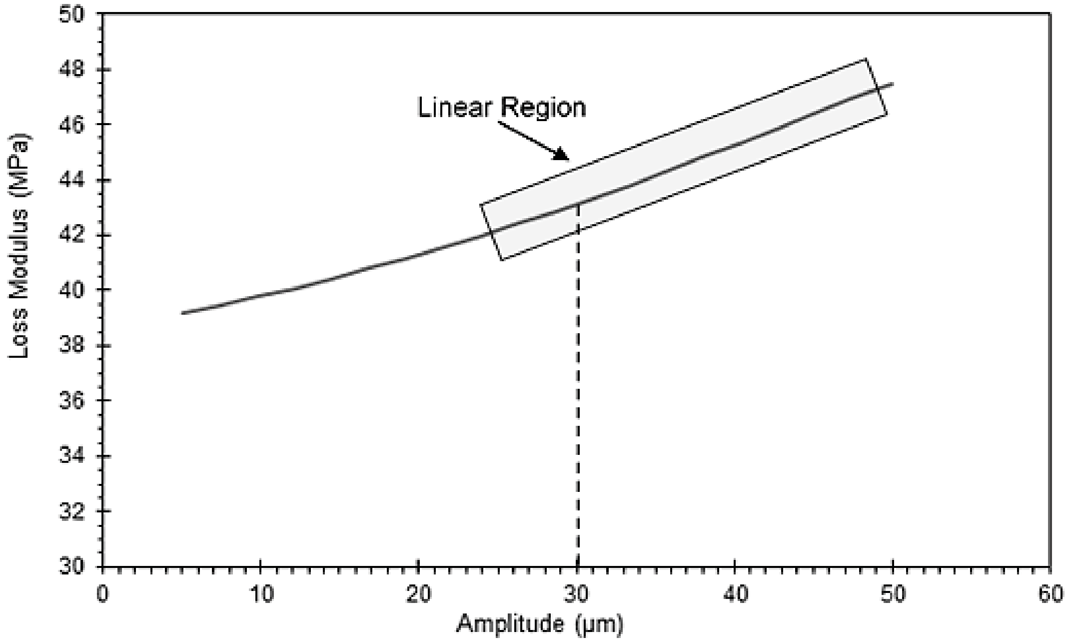

Figure 3-12: Amplitude test for neat CTC Resin 310-008/Hardener 320-009, showing that the plot is linear for an amplitude of 30 µm

The sample parameters entered into the software for the test to determine the modulus and tan delta were similar to that of the amplitude test. The thickness and width were entered along with a fixed length of 17.5 mm. The program mode was set to "DMA multi frequency strain" and the test was "temp ramp/frequency sweep". The procedure parameters were an amplitude of 30 µm, an initial temperature of 50 ^{o}C with a soak time of 5 minutes, a temperature ramp rate of 3 ^{o}C/min, and a final temperature of 290 ^{o}C with a hold time of 5 minutes. The frequency was held at 1 Hz for the entire duration of the test. Once the test was completed, the data was analyzed to determine the storage modulus, loss modulus, and tan delta graphs, which were then used to find the glass transition temperature (T_g).

The T_g found using the storage modulus is the onset temperature. This is found from the intersection of the line tangent to the temperatures below the glass transition and the tangent of the inflection point of the storage modulus. The T_g for loss modulus and tan delta is the temperature where the maximum of the loss modulus and the tan delta occurs.

3.2.11 Field Emission Scanning Electron Microscopy (FE-SEM) Test Method

A Hitachi S-4700 Filed Emission Scanning Electron Microscope (FE-SEM), shown in Figure 3-13, was used to image the composites. Imaging was done at 2 kV accelerating voltage with a working distance of 1.9 mm. The upper secondary electron detector was used to collect the images. FE-SEM is useful for high resolution imaging. The higher resolution allows the sample surface topography to be imaged.

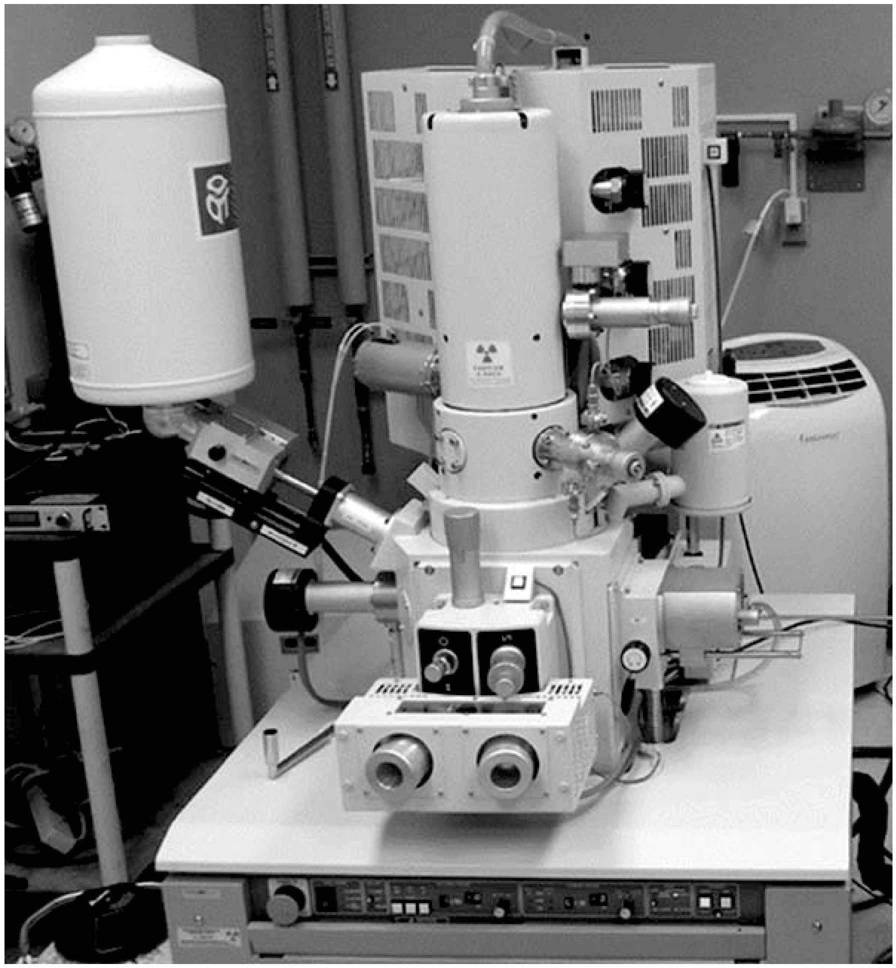

Figure 3-13: Hitachi S-4700 Field Emission Scanning Electron Microscope (FE-SEM)

Samples were cut from tested tensile specimen. The samples were cut to 2 mm x 2 mm x 10 mm using a scroll saw. The fracture surfaces were sputtered with platinum using an Anatech Hummer 6.2 Sputtering System, the platinum thickness was 2 nm to 5 nm depending on the GNP size. The samples were placed fracture surface up in a sample holder, as shown in Figure 3-14. Carbon tape was used to ground the samples with the sample holder.

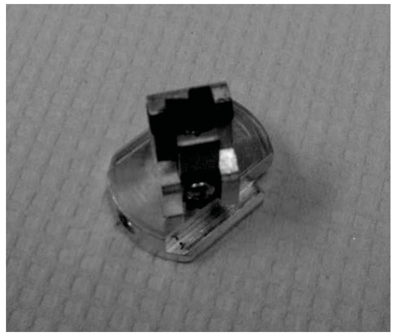

Figure 3-14: Sample mount for FESEM

3.2.12 Environmental Scanning Electron Microscopy (ESEM) Test Method

A FEI/Phillips XL40 Environmental Scanning Electron Microscope (see Figure 3-15) was used to view the surface of the GNP/epoxy composite. A 10 mm x 3mm x 10 mm sample was cut from a molded rectangular bar. The sample was prepared for observation by mounting the composite in a cast epoxy puck and then was polished. The sample was sputtered with platinum using an Anatech Hummer 6.2 Sputtering System. ESEM is useful for imaging under a low vacuum environment (close to atmospheric). The image obtained using ESEM is similar to an optical microscope, but at a higher magnification than optical imaging.

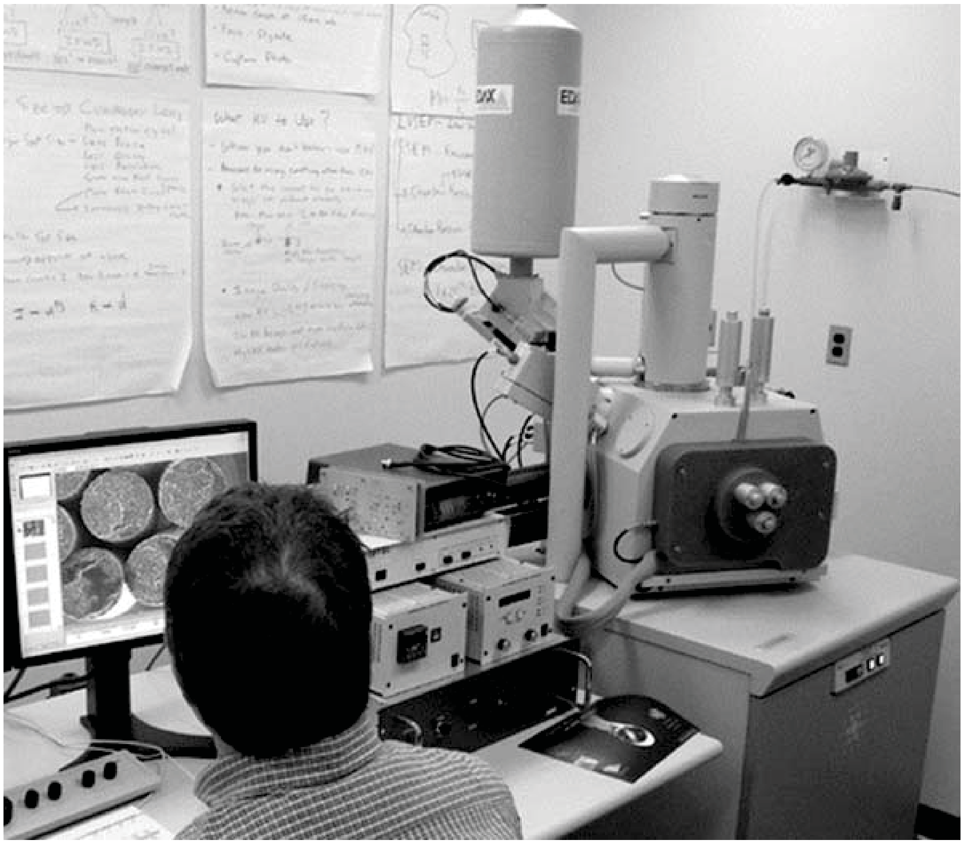

Figure 3-15: FEI/Phillips XL40 Environmental Scanning Electron Microscope

3.2.13 Optical Microscope Test Method

An Olympus PMG3 Metallograh optical microscope equipped with a Leica EC3 digital camera and Leica Application Suite EZ image capture software was used to view the surface of the GNP/epoxy composite. A 10 mm x 3mm x 10 mm sample was cut from a molded rectangular bar. The sample was prepared for observation by mounting the composite in a cast epoxy puck and then was polished.

3.3 References

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4 Results

4.1 Tensile Results

The tensile modulus measured for the neat epoxy was 2.72 GPa. The ultimate tensile stress was measured to be 77.6 MPa, and the strain at ultimate tensile stress was measured to be 8.0%. These values are shown in Table 4-1.

4.1.1 xGnP®-M-15 in Epoxy Tensile Results[1]

Figure 4-1 and Figure 4-2 show the mean [along with error bars ± 1 SD (standard deviation)] tensile modulus, ultimate tensile strength, and strain at ultimate tensile strength for the xGnP®-M-15/epoxy composites measured according to ASTM D638. Error bars are not shown for formulations where one standard deviation is less than the marker size. Table 4-1 also shows these results (mean, standard deviation, and number of samples tested). Adding GNP caused the tensile modulus to increase as well as the tensile strength and strain to decrease. The modulus increases from 2.72 GPa for neat epoxy to 3.36 GPa for the sample containing 6 wt % (3.7 vol %) GNP in epoxy. The ultimate tensile strength decreases from 77.6 (neat epoxy) to 35.5 MPa for the formulation containing 6 wt % (3.7 vol %) GNP in epoxy. The strain at ultimate tensile strength decreases from 8.0 (neat epoxy) to 1.5% for the formulation containing 6 wt % (3.7 vol

[1] The material contained within this section has been published in the journal *"Journal of Applied Polymer Science."*
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%) GNP in epoxy. These results compare well with those of Fukushima for graphene (15 μm average particle diameter) in Shell EPON 828 with curing agent Jeffamine T403 from Hunstman Petrochemical [1]. In Fukushima's work, he reported an increase in tensile modulus from ~2.75 (neat epoxy) to ~3.1 GPa for 3 vol % GNP/97 vol % epoxy [compared to our result of 3.3 GPa for 5 wt % GNP (~3 vol % GNP)] [1]. For tensile strength, Fukushima reported ~35 MPa for 3 vol % NP/97 vol % epoxy [compared to our result of 37 MPa for 5 wt % GNP (~3 vol % GNP)] [1]. This suggests that the load transfer between the GNP and the epoxy is satisfactory. The stiffness increases, but the strength and elongation decreases.

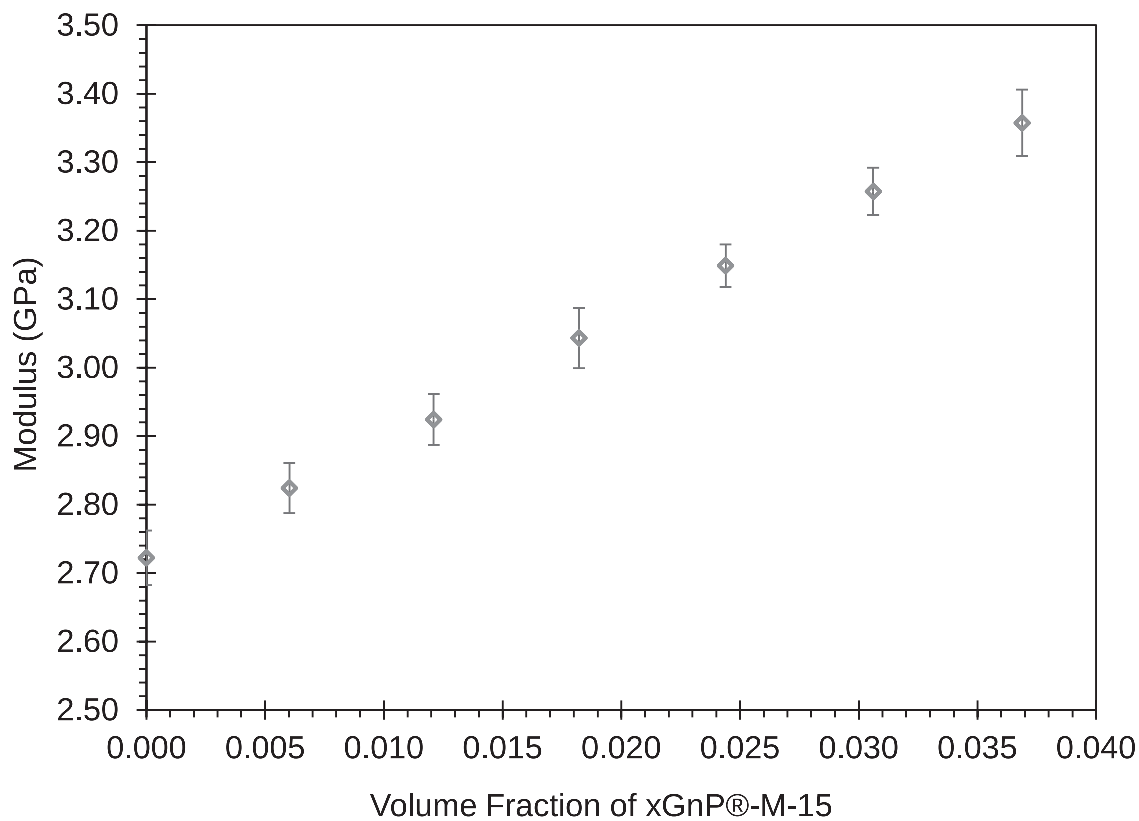

Figure 4-1: Modulus for xGnP®-M-15/epoxy composites

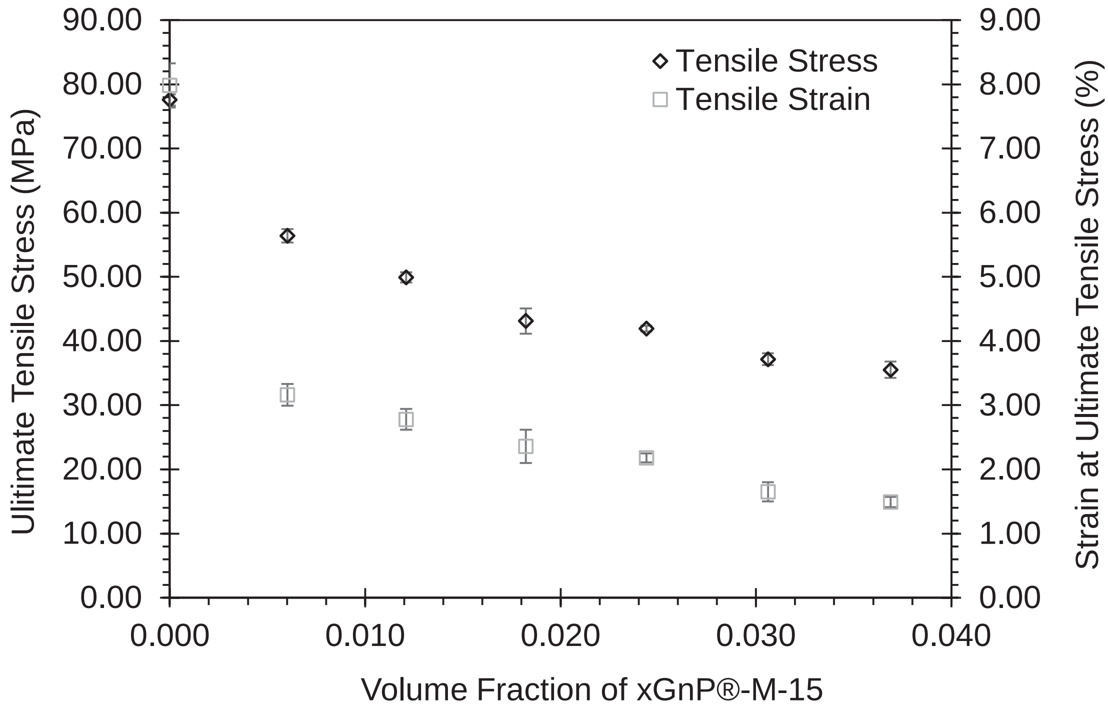

Figure 4-2: Ultimate tensile strength and strain at ultimate tensile strength for xGnP®-M-15/epoxy composites

Table 4-1: xGnP®-M-15 loading levels in epoxy and tensile results obtained from ASTM D638 test method

Formulation	GNP wt%	GNP vol %	Tensile Modulus (GPa)	Ultimate tensile strength (MPa)	Strain at ultimate tensile strength (%)
Epoxy	0	0.00	2.72 ± 0.04 n=6	77.6 ± 0.9 n=6	7.89 ± 0.35 n=6
1M15	1	0.60	2.82 ± 0.04 n=7	56.4 ± 1.0 n=7	3.16 ± 2.78 n=7
2M15	2	1.21	2.92 ± 0.04 n=7	49.9 ± 0.8 n=7	2.78 ± 0.16 n=7
3M15	3	1.82	3.04 ± 0.03 n=6	43.1 ± 2.0 n=6	2.36 ± 0.26 n=6
4M15	4	2.44	3.15 ± 0.04 n=6	41.9 ± 0.5 n=6	2.18 ± 0.07 n=6
5M15	5	3.06	3.26 ± 0.03 n=6	37.2 ± 0.9 n=6	1.65 ± 0.15 n=6
6M15	6	3.69	3.36 ± 0.05 n=6	35.5 ± 1.3 n=6	1.49 ± 0.08 n=6

4.1.2 xGnP®-M-5 in Epoxy and xGnP®-C-300 in Epoxy Tensile Results[2]

Figure 4-3, Figure 4-4, and Figure 4-5 show the mean (along with error bars = ± one standard deviation) tensile modulus, ultimate tensile strength, and strain at ultimate tensile strength for the xGnP®-M-5/epoxy and xGnP®-C-300/epoxy composites measured according to ASTM D638. Error bars are not shown for formulations where one standard deviation is less than the marker size. Table 4-2 also shows these results (mean, standard deviation, and number of samples tested). First, the xGnP®-M-5/epoxy composites will be discussed. As expected adding xGnP®-M-5 causes the tensile modulus to increase, as well as the tensile strength and ductility to decrease. The modulus increases from 2.72 GPa for neat epoxy to 3.35 GPa for the sample containing 6wt% (3.7 vol%) xGnP®-M-5 in epoxy. The ultimate tensile strength decreases from 77.6MPa (neat epoxy) to 36.4MPa for the formulation containing 6wt% (3.7 vol%) xGnP®-M-5 in epoxy. The strain at ultimate tensile strength decreases from 8.0% (neat epoxy) to 1.4% for the formulation containing 6wt% (3.7 vol%) GNP in epoxy. These results are almost identical to those obtained previously by our research group for xGnP®-M-15/epoxy composites [2]. xGnP®- M-15 is also produced by XG Sciences and has a 15 mm average particle diameter, a thickness of 7 nm, a surface area of ~130m^2/g, and a density of ~2.0 g/mL [3].

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[2] The material contained within this section has been published in the journal *"Journal of Composite Materials."*
Citation:
J. A. King, D. R. Klimek, I. Miskioglu, G. Odegard; "Mechanical Properties of Graphene Nanoplatelet/Epoxy Composites" *Journal of Composite Materials*, Vol 49, No. 6, pp.659-668, March 2015.

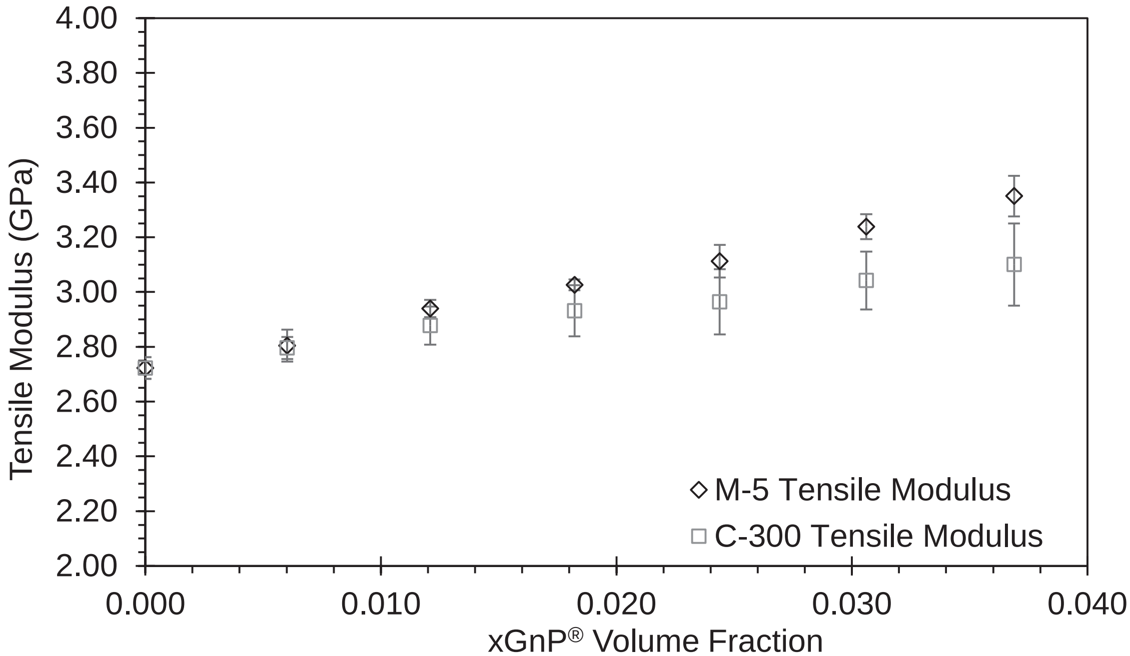

Figure 4-3: Tensile modulus for xGnP®-M-5/epoxy and xGnP®-C-300/epoxy composites

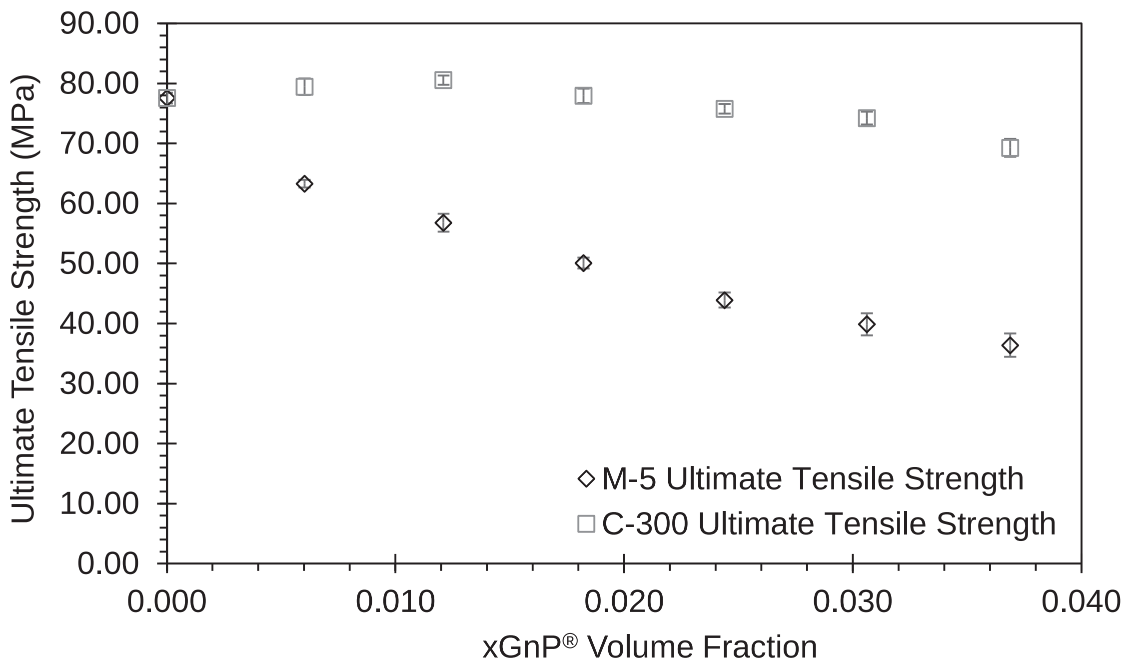

Figure 4-4: Ultimate tensile strength for xGnP®-M-5/epoxy and xGnP®-C-300 /epoxy composites

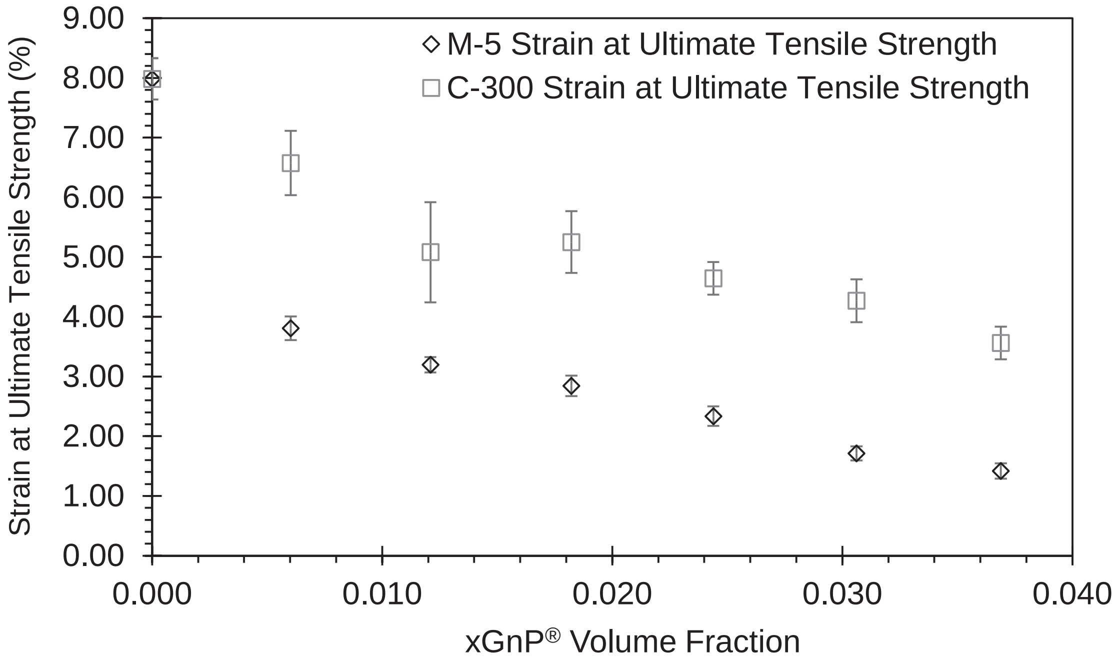

Figure 4-5: Strain at ultimate tensile strength for xGnP®-M-5/epoxy and xGnP®-C-300/epoxy composites

At this point, it is important to address the observed trend of decreasing tensile strength with increasing levels of xGnP®-M-5. Of course, it is desired to design composite materials where the strength increases with increasing filler content. However, the trends described above suggest that the GNP reinforcement serves to decrease the overall strength of the composite relative to the neat resin. This observation can be physically explained using the results from Molecular Dynamics simulation conducted by Hadden et al of this same material system (graphene- reinforced EPON 862 with Curing Agent W) [4]. Hadden et al demonstrated that the molecular structure of the epoxy monomer and hardener molecules at the interface is distinctly different than that in the bulk resin in this particular material system [4]. In fact, this interfacial zone is about 1 nm wide and has a density that varies continuously with respect to the bulk density. As a result, it is anticipated that this structural disturbance may adversely affect the strength properties of the composite system, thus potentially explaining the strength observations described herein. The results suggest that the load transfer between the M-grade GNPs and the epoxy is satisfactory. If the load transfer is poor then the GNP acts like a void and weakens the composite.

Table 4-2: xGnP®-M-5 and xGnP®-C-300 loading levels in epoxy, tensile results obtained from ASTM D638 test method

Formulation	GNP Wt %	GNP Vol %	Tensile Modulus (GPa)	Ultimate Tensile Strength (MPa)	Strain at Ultimate Tensile Strength (%)
Epoxy	0	0.0	2.72 ± 0.04 n = 6	77.6 ± 0.9 n = 6	7.98 ± 0.35 n = 6
xGnP®-M-5					
1M5	1	0.60	2.80 ± 0.06 n = 6	63.3 ± 1.4 n =6	3.81 ± 0.20 n = 6
2M5	2	1.21	2.94 ± 0.03 n = 9	56.8 ± 0.8 n = 9	3.20 ± 0.13 n = 9
3M5	3	1.82	3.03 ± 0.02 n = 7	50.1 ± 1.2 n = 7	2.84 ± 0.17 n = 7
4M5	4	2.44	3.11 ± 0.06 n = 6	43.9 ± 0.8 n = 6	2.34 ± 0.16 n = 6
5M5	5	3.06	3.24 ± 0.05 n = 7	39.9 ± 1.1 n = 7	1.71 ± 0.12 n = 7
6M5	6	3.69	3.35 ± 0.07 n = 8	36.4 ± 1.4 n = 8	1.42 ± 0.13 n = 8
xGnP®-C-300					
1C300	1	0.60	2.80 ± 0.04 n = 7	79.5 ± 0.7 n =7	6.57 ± 0.54 n = 7
2C300	2	1.21	2.88 ± 0.07 n = 8	80.6 ± 1.5 n = 8	5.08 ± 0.84 n = 8
3C300	3	1.82	2.93 ± 0.09 n = 8	77.9 ± 0.9 n = 8	5.25 ± 0.52 n = 8
4C300	4	2.44	2.96 ± 0.12 n = 7	75.8 ± 1.3 n = 7	4.64 ± 0.27 n = 7
5C300	5	3.06	3.04 ± 0.11 n = 8	74.2 ± 1.8 n = 8	4.27 ± 0.36 n = 8
6C300	6	3.69	3.10 ± 0.15 n = 8	69.3 ± 2.0 n = 8	3.56 ± 0.27 n = 8

For the xGnP®-C-300/epoxy composites, the tensile modulus is slightly lower than the xGnP®-M-5/epoxy and xGnP®-M-15/epoxy composites. The ultimate tensile strength (UTS) for the xGnP®-C-300/epoxy composites stays fairly constant between 74 and 81 MPa for those containing $\leq$ 5wt% (3.1 vol%) xGnP®-C-300. This observation is in contrast to the decreasing ultimate tensile strength as xGnP®-M-5 and xGnP®-M-15 is added to epoxy. The strain at UTS also follows this same trend. Apparently, the smaller size of the xGnP®-C-300 in epoxy produces a composite with higher strength and ductility compared to the xGnP®-M-5/epoxy composites. Since the xGnP®-C-300 maintains its strength and strain, this suggests that the GNP and epoxy has a better load transfer than the M-grade GNPs.

4.1.3 Asbury Carbon's TC307 in Epoxy Tensile Results

Figure 4-6 and Figure 4-7 show the average tensile modulus, ultimate tensile strength, and strain at ultimate tensile strength for the TC307/epoxy composites measured according to ASTM D638. Error bars shown are ± 1 standard deviation (SD). The average tensile modulus, ultimate tensile strength, and stain at ultimate tensile strength with standard deviation and number of samples tested for each is summarized in Table 4-3. Adding the TC307 to the epoxy increases the tensile modulus and decreases the tensile strength and strain. The neat epoxy has a tensile modulus of 2.72 GPa with a strength of 77.6 MPa and a strain of 7.98%. The 4 wt% (2.44 vol%) TC307 in epoxy has a tensile modulus of 2.92 GPa with a strength of 75.8 MPa and a strain of 5.25%. It has been observed that GNP increases tensile modulus, Chatterjee et al. reported an increase from ~2.65 GPa for neat epoxy to ~3.08 GPa for 2 wt% GNP in epoxy [5].

Table 4-3: Asbury TC307 loading levels in epoxy, tensile results obtained from ASTM D638 test method

Formulation	GNP wt%	GNP vol %	Tensile Modulus (GPa)	Ultimate tensile strength (MPa)	Strain at ultimate tensile strength (%)
Epoxy	0	0.00	2.72 ± 0.04 n = 6	77.6 ± 0.9 n = 6	7.98 ± 0.35 n = 6
1TC307	1	0.60	2.84 ± 0.08 n = 6	81.2 ± 1.6 n =6	5.94 ± 0.82 n = 6
2TC307	2	1.21	2.88 ± 0.07 n = 7	80.9 ± 1.9 n = 7	5.91 ± 0.51n = 7
3TC307	3	1.82	2.91 ± 0.05 n = 8	78.1 ± 0.8 n = 8	4.74 ± 0.24 n = 8
4TC307	4	2.44	2.93 ± 0.09 n = 7	75.8 ± 1.8 n = 7	4.55 ± 0.31 n = 7

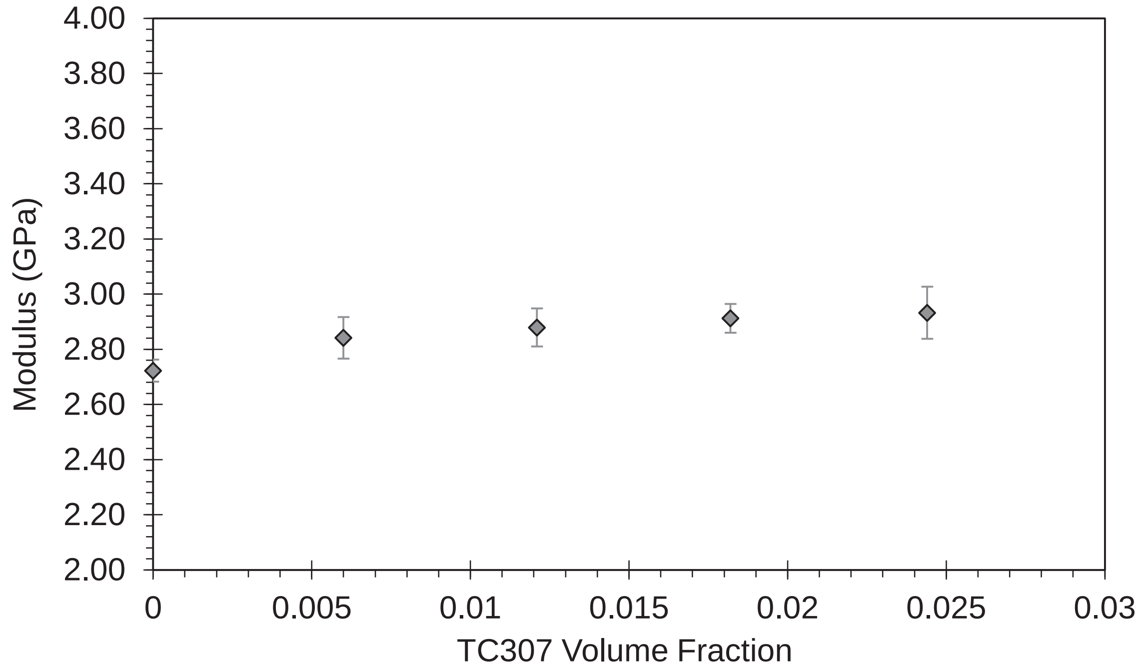

Figure 4-6: Modulus for TC307/epoxy composites

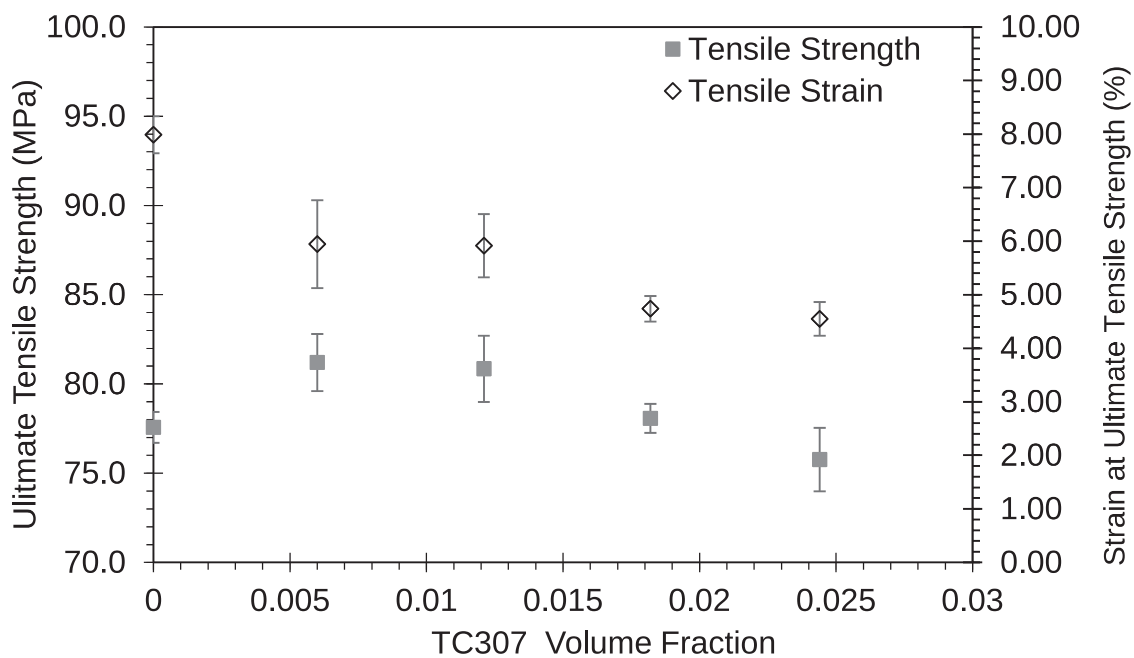

Figure 4-7: Ultimate tensile strength and strain at ultimate tensile strength TC307/epoxy composites

4.1.4 xGnP®-C-300 with Carbon Fiber in Epoxy Tensile Results

Table 4-4 shows the tensile results (mean, standard deviation, and number of samples tested) for the carbon fiber/epoxy and xGnP®-C-300/carbon fiber/epoxy composites. From the data it is clear that adding 1 to 3 wt% xGnP®-C-300 to carbon fiber/epoxy composites did not cause an the axial modulus to change significantly, but caused an increase in the transverse modulus. The carbon fiber dominates in the axial direction, this is expected due to the high axial modulus of the carbon fiber. The epoxy matrix serves to transfer the load between the fibers, this occurs primarily through interfacial shear.

Table 4-4: Tensile Modulus results for xGnP®-C-300/carbon fiber/epoxy composites

Material system	Filler Wt %	Filler Vol %	Axial Modulus (GPa)	Transverse Modulus (GPa)
carbon fiber/epoxy	GNP – 0 CF – 67	GNP – 0 CF – 58	134.29 ± 9.27 n = 6	7.81 ± 0.81 n =5
xGnP®-C-300/carbon fiber/epoxy	GNP – 1 CF – 67	GNP – 0.8 CF – 58	137.5 ± 9.33 n = 15	7.89 ± 0.13 n =3
xGnP®-C-300/carbon fiber/epoxy	GNP – 2 CF – 67	GNP – 1.6 CF – 58	137.0 ± 6.53 n = 15	8.29 ± 0.14 n =3
xGnP®-C-300/carbon fiber/epoxy	GNP – 3 CF – 67	GNP – 2.3 CF – 58	137.1 ± 9.75 n = 11	---

4.2 Nanoindentation Results

4.2.1 xGnP®-M-15 in Epoxy Nanoindentation Results[3]

Typical curves for E and H as a function of indenter penetration are shown in Figure 4-8 for 5 wt % GNP in epoxy. The E and H values reported are the average of E and H determined over the range of indenter penetration from 500 to 1500 nm. Figure 6 shows that hardness is 0.26 GPa. Table I shows a constant hardness value (~0.24–0.26 GPa) for neat epoxy and 1–6 wt % GNP in epoxy.

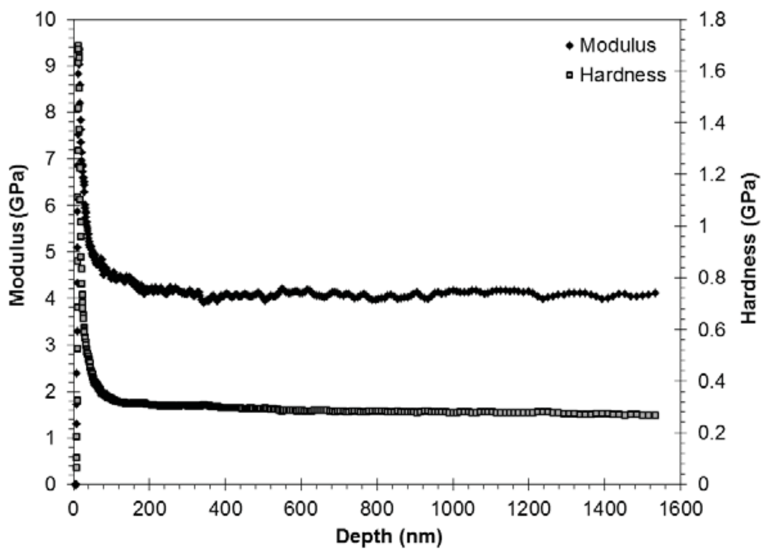

Figure 4-8: Modulus and hardness determined by nanoindentation for 5 wt% xGnP®-M-15 in epoxy

[3] The material contained within this section has been published in the journal *"Journal of Applied Polymer Science."*
Citation:
J. A. King, D. R. Klimek, I. Miskioglu, G. Odegard; "Mechanical Properties of Graphene Nanoplatelet/Epoxy Composites" *Journal of Applied Polymer Science*, Vol 128, No. 6, pp.4217-4223, June 2013.

Figure 4-9 shows the mean modulus as determined by the nanoindentation test. The error bars that are shown represent ±1 SD. Whether or not the Berkovich indenter encounters a GNP rich area or a matrix rich area on the sample will introduce error into this test method. Thus, as the amount of GNP increases, the error bars become larger, because more GNP is present in the sample. Several researchers have shown for polymers and polymer-based composites that modulus as determined by nanoindentation is higher than that reported by macroscopic tensile tests [6-8]. These ratios have been found to be 1.70 for polystyrene and 1.64 for polycarbonate [6]. This difference is likely due to pile up of material around the contact impression and viscoelasticity of the polymer and polymer based composites that is not accounted for by the modulus as determined by the Oliver–Pharr method [6-8]. The mean modulus from nanoindentation for the neat epoxy was 3.61 GPa (see Figure 4-9) when compared with 2.72 GPa (see Table 4-1) from the macroscopic D638 tensile test. Hence, a ratio of 1.33 was seen for our neat epoxy composites. This ratio was then used as a scaling factor for all moduli for the GNP/epoxy composites, and these mean results are also shown in Figure 4. The modulus as determined from nanoindentation showed a similar trend to the tensile modulus determined by a macroscopic method.

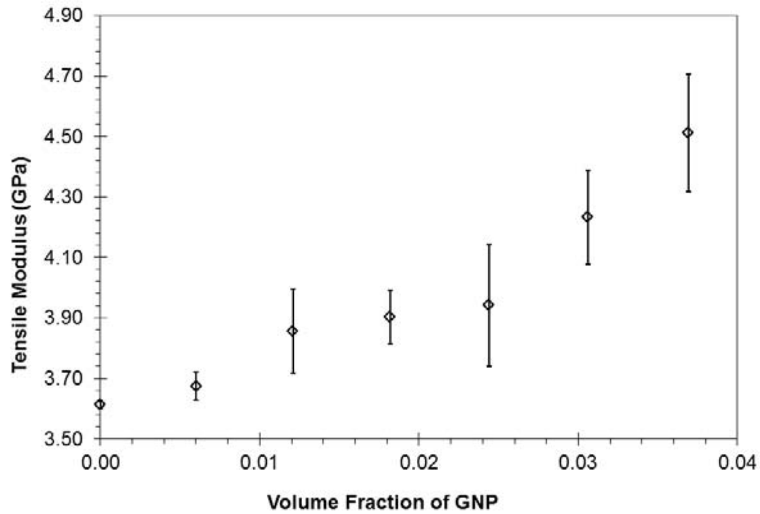

Figure 4-9: Modulus determined by nanoindentation for xGnP®-M-15/epoxy composites

Figure 4-10 shows typical displacement (also called depth) curves as a function of time for 5 wt % (3.1 vol %) GNP in epoxy at varying loads from 2 to 45 mN. Figure 4-10 shows that a steady state creep stage is observed almost as soon as the creep load was reached. Figure 4-11 shows the creep compliance for the 5 wt % GNP in epoxy composite at all loads. The creep compliance calculated varies very little with the loads between 15 and 45 mN in the steady-state creep range. For loads of 2, 5, and 10 mN, the creep compliance increases with load. Figure 4-12 displays the creep compliance results at 2 mN for all the formulations. Figure 4-12 shows that the creep compliance for the neat epoxy is slightly higher than that of the GNP/epoxy composites. This result was also observed for the 5 mN loading. For loadings from 10 to 45 mN, the creep compliance was the same for all formulations (neat epoxy and GNP/epoxy). As an example, Figure 4-13 illustrates the creep compliance results at 25 mN for all the formulations. Tehrani et al. reported for neat epoxy (room temperature cure) and for 3 wt % multiwall carbon nanotube (MWCNT) in epoxy composites similar creep compliance curves tested at 1 mN and 25 °C [9]. At 25 °C and 3 mN, Tehrani et al. observed reduced creep compliance in the MWCNT/epoxy composite as opposed to the neat epoxy [9]. Our results reported in this work indicate that for 10–45 mN, creep compliance is the same for the neat epoxy and GNP/epoxy composites.

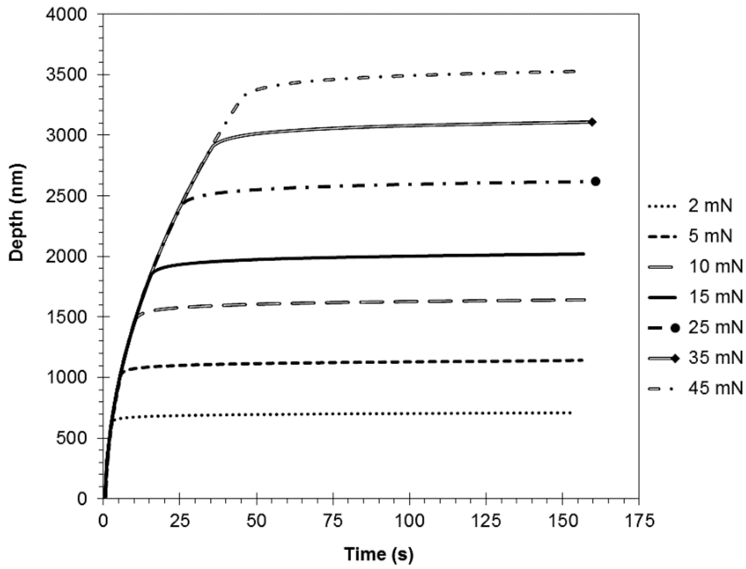

Figure 4-10: Nanoindentation penetration depth curves at various loads for 5 wt% xGnP®-M-15 in epoxy

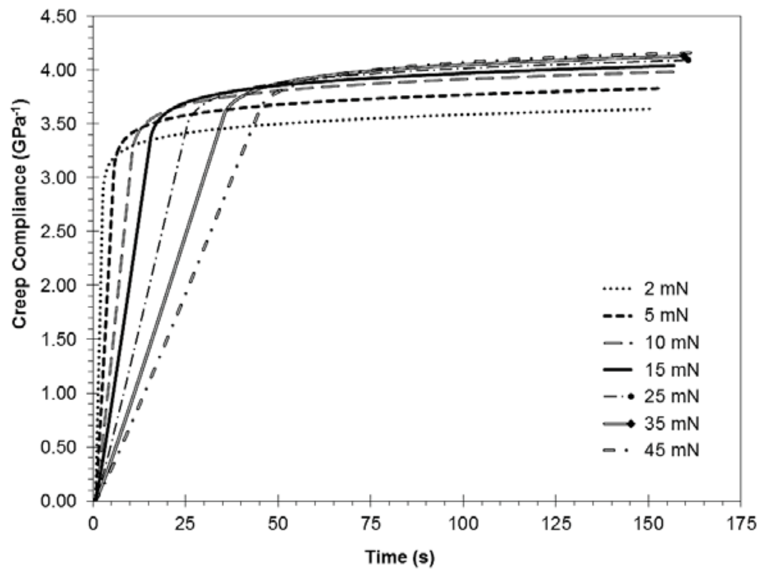

Figure 4-11: Creep compliance for 5 wt% xGnP®-M-15 in epoxy at various loads

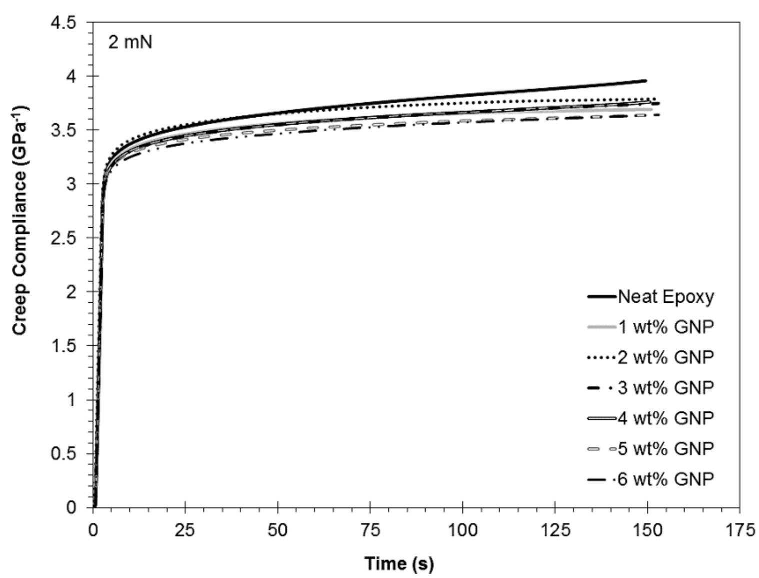

Figure 4-12: Creep compliance at a load of 2 mN for neat epoxy and xGnP®-M-15/epoxy composites

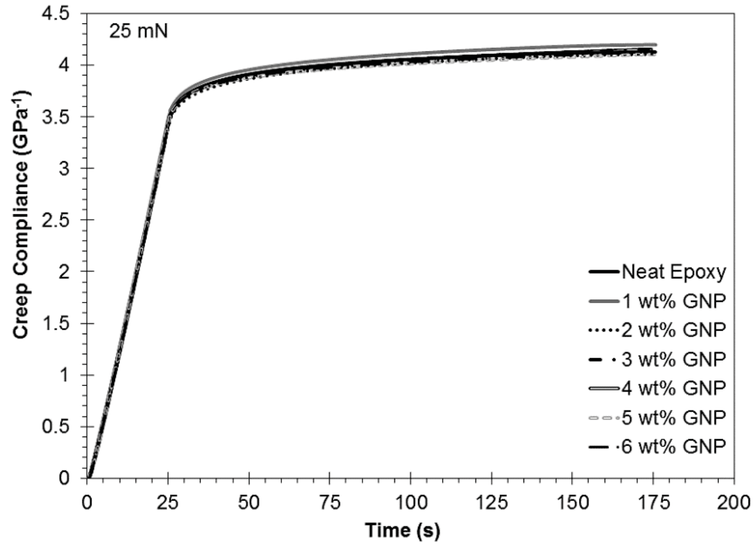

Figure 4-13: Creep compliance at a load of 25 mN for neat epoxy and xGnP®-M-15/epoxy composites

4.2.2 xGnP®-M-5 in Epoxy and xGnP®-C-300 in Epoxy Nanoindentation Results[4]

The modulus (E) and hardness (H) values reported are the average of E and H determined over the range of indenter penetration from 500nm to 1500nm using the CSM method. Table 1 shows a constant hardness value of 0.25 to 0.27 GPa for neat epoxy, 1 to 6wt% xGnP-M-5/epoxy, and 1-6wt% xGnP-C-300/epoxy composites. Typical curves for

[4] The material contained within this section has been published in the journal *"Journal of Composite Materials."*
Citation:
J. A. King, D. R. Klimek, I. Miskioglu, G. Odegard; "Mechanical Properties of Graphene Nanoplatelet/Epoxy Composites" *Journal of Composite Materials*, Vol 49, No. 6, pp.659-668, March 2015.

E and H as a function of indenter penetration have been published previously in King et al [2].

Several researchers have shown for polymers and polymer-based composites that modulus as determined by nanoindentation is higher than that reported by macroscopic tensile tests [6-8]. Tranchida et al. reported that for polystyrene the modulus obtained from nanoindentation was 1.70 times the modulus obtained from macroscopic tensile tests [6]. This difference is likely due to pile-up of material around the contact impression and viscoelasticity of the polymer and polymer-based composites that is not accounted for by the modulus as determined by the Oliver-Pharr Method [6-8]. The mean modulus from nanoindentation for the neat epoxy was 3.61 GPa as compared to 2.72 GPa from the macroscopic tensile test. Hence, the ratio of nanoindentation to macroscopic tensile modulus was found to be 1.33. This ratio was then used as a scaling factor for all moduli for the xGn-M-5/epoxy and xGnP-C-300/epoxy composites and these mean results are also shown in Table 4-5 (standard deviation and number of samples tested are also shown). The modulus as determined from nanoindentation showed a similar trend to the tensile modulus determined by the macroscopic method for all composite systems.

Table 4-5: Modulus as Determined by Nanoindentation for xGnP®-M-5 in Epoxy and xGnP®-C-300 in Epoxy

Formulation	GNP Wt %	GNP Vol %	Nano Modulus (GPa)	Scaled Nano Modulus (MPa)	Hardness (GPa)	Stress Exponent (m) for 25 mN Creep Load
Epoxy	0	0.0	3.61 ± 0.02 n = 16	2.72 ± 0.02 n = 16	0.255 ± 0.003 n = 16	30.45 ± 1.62 n = 36
xGnP®-M-5						
1M5	1	0.60	3.75 ± 0.04 n = 24	2.82 ± 0.04 n =24	0.261 ± 0.005 n = 24	29.14 ± 0.82 n = 36
2M5	2	1.21	3.89± 0.07 n = 24	2.92 ± 0.07 n = 24	0.259 ± 0.006 n = 24	28.77 ± 1.67 n = 36
3M5	3	1.82	3.95 ± 0.11 n = 24	2.97 ± 0.11 n = 24	0.256 ± 0.010 n = 24	30.65 ± 1.32 n = 36
4M5	4	2.44	4.08 ± 0.15 n = 24	3.07 ± 0.15 n =24	0.252 ± 0.013 n = 24	29.85 ± 1.40 n = 36
5M5	5	3.06	4.32 ± 0.15 n = 24	3.25 ± 0.15 n =24	0.254 ± 0.016 n = 24	31.47 ± 1.55 n = 36
6M5	6	3.69	4.45 ± 0.14 n = 24	3.35 ± 0.14 n =24	0.255 ± 0.012 n = 24	30.47 ± 1.20 n = 36
xGnP®-C-300						
1C300	1	0.60	3.65 ± 0.04 n = 35	2.74 ± 0.04 n =35	0.261 ± 0.004 n = 35	27.13 ± 0.43 n = 36
2C300	2	1.21	3.79 ± 0.03 n = 25	2.85 ± 0.03 n =25	0.265 ± 0.007 n = 25	28.47 ± 0.46 n = 36
3C300	3	1.82	3.79 ± 0.03 n = 29	2.85 ± 0.03 n =29	0.263 ± 0.007 n = 29	29.52 ± 0.68 n = 36
4C300	4	2.44	3.83 ± 0.04 n = 30	2.88 ± 0.04 n =30	0.265 ± 0.005 n = 30	28.57 ± 0.56 n = 36
5C300	5	3.06	3.90 ± 0.09 n = 24	2.93 ± 0.09 n =24	0.263 ± 0.007 n = 24	28.50 ± 0.64 n = 36
6C300	6	3.69	3.91 ± 0.08 n = 24	2.94 ± 0.08 n =24	0.267 ± 0.004 n = 24	29.62 ± 0.87 n = 36

Creep tests were conducted at the following loads: 2, 5, 10, 15, 25, 35, and 45 mN. Typical displacement (also called depth) curves as a function of time were previously shown in King et al [2]. At 2 mN, the maximum depth observed was 740nm for neat epoxy and 706nm for 6wt% xGnP-M-5/epoxy and 6wt% xGnP-C-300/epoxy composites (4.6% decrease in the maximum penetration depth). At the highest load of 45 mN, the maximum depth was 3539nm for neat epoxy and 3486nm for 6wt% xGnP-M-5/epoxy and 6wt% xGnP-C-300/epoxy composites (1.5% decrease in maximum penetration depth). Figure 4-14 shows the creep compliance for the 5wt% xGnP-C-300/epoxy composite. The creep compliance varies very little with loads between 15 and 45mN in the steady-state creep range. For loads of 2, 5, and 10 mN, the creep compliance increases with load. A similar creep response was also observed for the neat epoxy, 1 to 4 and 6wt% xGnP-C-300/epoxy, and 1 to 6wt% xGnP-M-5/epoxy composites. As an example, Figure 4-15 shows the creep compliance at the 25mN load for neat epoxy, 2 and 5wt% xGnP-M-5/epoxy, and 2 and 5wt% xGnP-C-300/epoxy composites. These figures show that the steady-state creep compliance remains constant for neat epoxy, 1-6wt% wt% xGnP-C-300/epoxy composites, and 1 to 6wt% xGnP-M-5/epoxy composites.

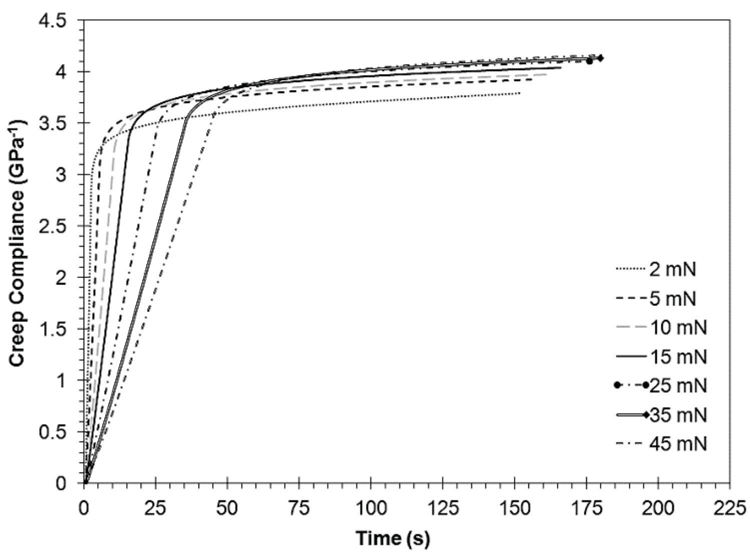

Figure 4-14: Creep Compliance for 5 wt% xGnP®-C-300 in Epoxy at Various Loads

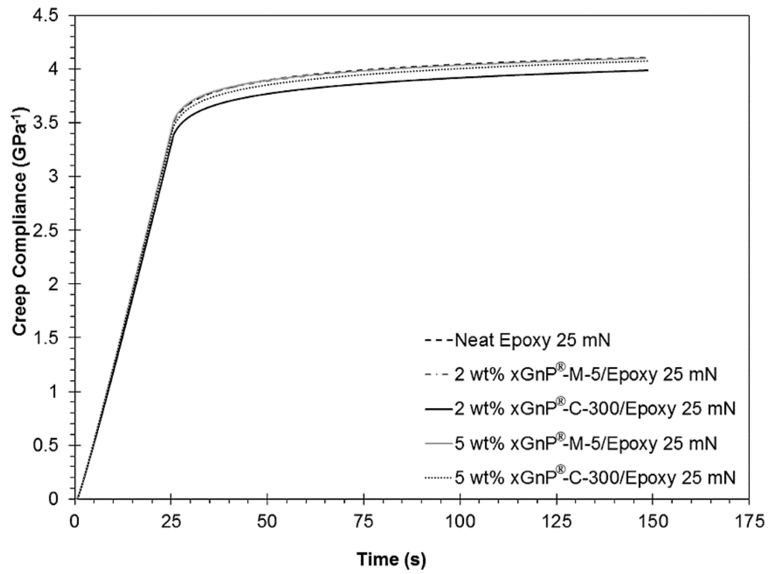

Figure 4-15: Creep Compliance for Neat Epoxy, 2 and 5 wt% xGnP®-C-300 in Epoxy at 25 mN

A simplified relationship between the strain rate ($\dot{\varepsilon}$) and stress (σ) during steady state creep can be given as

$$\dot{\varepsilon} = B\sigma^{m} \qquad (4\text{-}1)$$

Where B and m are constants. Strain rate is calculated from the displacement of the indenter as

$$\dot{\varepsilon} = \left[\frac{(\mathrm{displacement})_i - (\mathrm{displacement})_{i-1}}{(\mathrm{displacement})_i}\right] / (t_i - t_{i-1}) \qquad (4\text{-}2)$$

and stress is

$$\sigma = \frac{P_0}{A(t)} \tag{4-3}$$

where A(t) is the contact area (see Equation 3-2). The calculations for $\dot{\varepsilon}$, σ, and the stress exponent m are performed at the conclusion of the creep test. The stress exponent m can be interpreted as the sensitivity of the material to creep [10]. The stress exponent m was calculated for neat epoxy, 1–6wt% xGnP-C-300/ epoxy composites, and 1 to 6wt% xGnP-M-5/epoxy composites at 2, 5, 10, 15, 25, 35, and 45 mN. The m values in all formulations at all loadings were similar. Table 1 shows the values for m at 25mN for all the formulations. The constant value for stress exponent m agrees with the constant creep compliance curves. In addition, Zandiastashbar et al. reported similar creep results for graphene platelet/epoxy nanocomposites [11].

4.2.3 Asbury TC307 in Epoxy Nanoindentation Results

The modulus (E) and hardness (H) shown in Table 4-6 were determined by taking the average E and H over a penetration depth of 500 nm to 1500 nm as per the continuous stiffness method. Table 4-6 shows a hardness of ~0.26 GPa for neat epoxy and ~0.28 for 4 wt% TC307 in epoxy. Figure 4-16 shows the modulus as determined by nanoindentation as a function of volume fraction of TC307, error bars shown are ± 1 SD.

It has been observed that the modulus as determined by nanoindentation is higher than the modulus as determined by macroscopic tensile test (ASTM D638) for polymer-based composites [6-8]. A possible cause for this difference is pile-up of material around the point of contact. The polymer viscoelasticity is not accounted for in the modulus as determined by Oliver-Pharr method [6-8]. The modulus as determined by nanoindentation for neat epoxy was found to be 3.61 GPa which is 1.33 times greater than that measured by the macroscopic tensile test. This ratio was then applied to the moduli of all formulations and the result is shown in Table 4-6 as 'scaled nano modulus'. The scaled nanoindentation modulus shows the same trend as the macroscopic tensile modulus.

Table 4-6: Nanoindentation Results for TC307 in Epoxy

Material system	**Filler Wt % (Vol %)**	**Nano Modulus (GPa)**	**Scaled Nano Modulus (GPa)**	**Hardness (GPa)**
Neat Epoxy	0 (0.00)	3.61 ± 0.02 n = 16	2.72 ± 0.02 n = 16	0.255 ± 0.003 n = 16
1TC307	1 (0.60)	3.70 ± 0.03 n = 33	2.78 ± 0.03 n = 33	0.273 ± 0.005 n = 33
2TC307	2 (1.21)	3.74 ± 0.07 n = 33	2.81 ± 0.07 n = 33	0.273 ± 0.009 n = 33
3TC307	3 (1.82)	3.81 ± 0.06 n = 34	2.87 ± 0.06 n = 34	0.278± 0.006 n = 34
4TC307	4 (2.44)	3.85 ± 0.07 n = 33	2.89 ± 0.07 n = 33	0.281 ± 0.009 n = 33

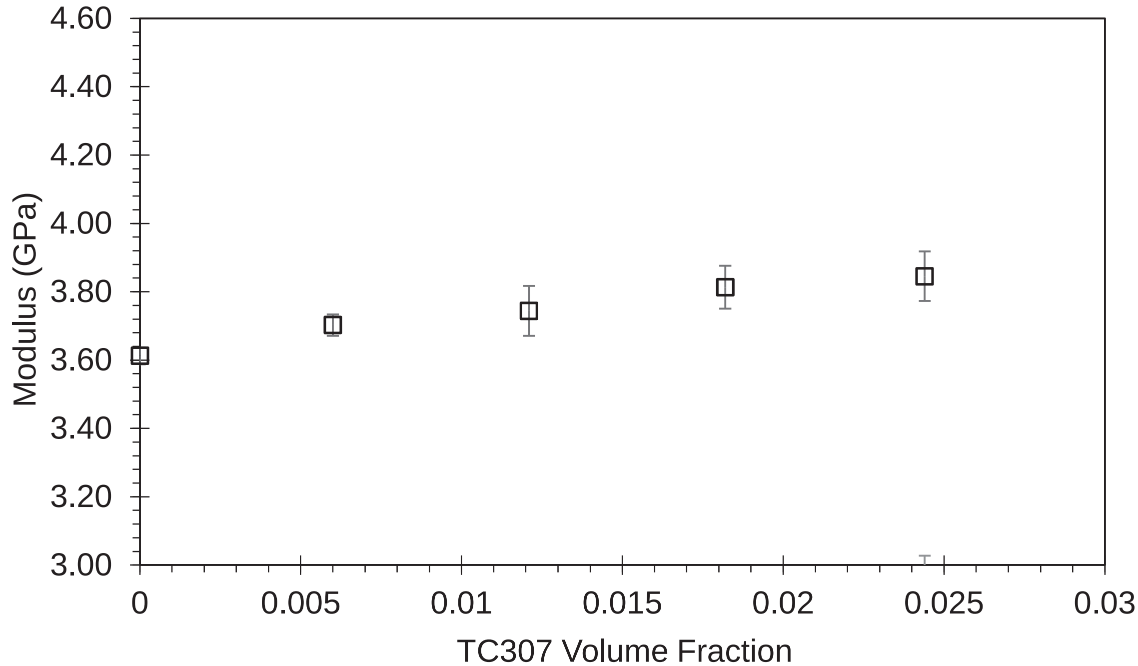

Figure 4-16: Modulus as Determined by Nanoindentation for TC307/Epoxy Composites

4.3 Electrical Resistivity Results

4.3.1 xGnP®-M-15 in Epoxy Electrical Resistivity Results[5]

Figure 4-17 shows the log (electrical resistivity in Ω-cm) as a function of filler volume fraction. All the data points have been plotted in this figure. At low-filler loadings, the electrical resistivity remains similar to that of the pure polymer. Then, at a point called the percolation threshold, the resistivity decreases dramatically over a very

[5] The material contained within this section has been published in the journal *"Journal of Applied Polymer Science."*
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narrow range of filler concentrations [12, 13]. Figure 4-17 illustrates that the percolation threshold occurs at ~0.99 vol % (~ 1.6 wt %) for GNP. Enough formulations were tested to determine the percolation threshold, because this will be discussed later for tensile modulus models.

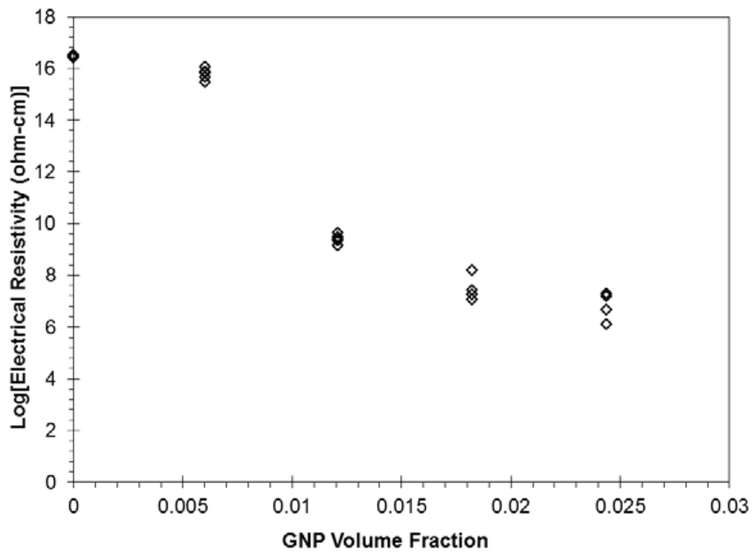

Figure 4-17: Log (electrical resistivity) results for xGnP®-M-15/epoxy composites

The percolation threshold for GNP has been recently modeled using a similar analytical method using the interparticle distance concept [14]. This model postulates that conductive particles can be separated by a distance equal to the electron tunneling distance through the nonconductive matrix and still have conduction between the particles. The model assumes high-aspect ratio disc-like morphology for GNP and uses this assumption to determine cubic elements containing a single GNP and then uses these elements to determine a percolation threshold. The resulting analytical formula for the percolation threshold for GNP/PC composites is given by Equation 4-4 below [14].

$$\phi_C = \frac{27\ \pi D^2 t}{4(D + IPD)^3} \quad (4\text{-}4)$$

In Equation 4-4, ϕ_c is the filler volume fraction at the percolation threshold, 0.0099; t is the thickness of the platelet, 7 m; IPD is the electron tunneling distance, 10 nm for many polymer systems [14]; and D is the diameter of the platelet. Using Equation 4-4, the diameter of the platelet was then calculated to be 15,000 nm (15 μm), which agrees with the vendor literature [3].

4.3.2 xGnP®-M-5 in Epoxy and xGnP®-C-300 in Epoxy Electrical Resistivity Results

Table 4-7 shows the volume electrical resistivity for all formulations for both types of GNP. At low-filler loadings, the electrical resistivity remains similar to that of the pure polymer. Then, at the percolation threshold, the resistivity decreases dramatically over a very narrow range of filler concentrations [12, 13]. The percolation threshold occurs at ~2.53 vol % (~ 4.1 wt %) for xGnP®-M-5 and ~2.09 vol% (~3.4 wt%) for xGnP®-C-300. Enough formulations were tested to determine the percolation threshold, because this will be used later for tensile modulus models. Both of these values match the as received aspect ratio that is used to solve for ϕ_C in Equation 4-4.

Table 4-7: Electrical resistivity results for xGnP®-M-5/epoxy and xGnP®-C-300/epoxy composites

Formulation	Storage Modulus Onset (°C)
Neat Epoxy	$2.88 \times 10^{16} \pm 3.20 \times 10^{15}$ n=6
xGnP®-M-5	
1M5	$2.72 \times 10^{15} \pm 1.02 \times 10^{15}$ n=5
2M5	$4.71 \times 10^{15} \pm 1.86 \times 10^{15}$ n=4
3M5	$9.73 \times 10^{14} \pm 6.50 \times 10^{14}$ n=4
4M5	$3.78 \times 10^{9} \pm 2.55 \times 10^{9}$ n=5
5M5	$6.06 \times 10^{8} \pm 8.88 \times 10^{9}$ n=5
6M5	$2.14 \times 10^{8} \pm 3.36 \times 10^{8}$ n=5
xGnP®-C-300	
1C300	$2.63 \times 10^{16} \pm 1.58 \times 10^{16}$ n=6
2C300	$5.07 \times 10^{15} \pm 2.27 \times 10^{15}$ n=6
3C300	$5.85 \times 10^{15} \pm 1.99 \times 10^{15}$ n=5
4C300	$3.48 \times 10^{8} \pm 6.32 \times 10^{7}$ n=5
5C300	$1.99 \times 10^{8} \pm 1.40 \times 10^{8}$ n=5
6C300	$4.35 \times 10^{7} \pm 6.49 \times 10^{7}$ n=5

4.4 Dynamic Mechanical Analysis (DMA) Results

The glass transition temperature (T_g) for the neat epoxy was found to be about 156 °C (determined by the peak of the tan delta curve). Table 4-8 shows the average temperature for the storage modulus onset, the peak of the loss modulus, and the peak of the tan delta for all formulations of xGnP®-M-15 in epoxy, xGnP®-M-5 in epoxy, and xGnP®-C-300 in epoxy. The storage modulus onset, the peak of the loss modulus and the peak of the tan delta all follow a similar trend. The T_g decreases until it hits the approximate percolation threshold and then begins to increase again, this is true for all three types of GNP.

Table 4-8: DMA results for xGnP®-M-15 in epoxy, xGnP®-M-5 in epoxy and xGnP®-C-300 in epoxy

Formulation	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
Neat Epoxy	139.3 ± 0.5 n=6	149.8 ± 0.3 n=6	155.8 ± 0.4 n=6
xGnP®-M-15			
1M15	131.9 ± 0.8 n=3	145.3 ± 0.3 n=3	153.5 ± 0.5 n=3
2M15	130.2 ± 0.7 n=5	143.6 ± 0.8 n=5	152.6 ± 0.8 n=5
3M15	130.4 ± 0.5 n=3	143.1 ±0.2 n=3	150.4 ± 0.3 n=3
4M15	131.5 ± 0.6 n=3	144.1 ± 0.5 n=3	152.4 ± 0.3 n=3
5M15	137.1 ± 0.3 n=3	149.1 ± 0.4 n=3	156.0 ± 0.4 n=3
6M15	136.0 ± 1.0 n=5	148.4 ± 0.6 n=5	155.9 ± 0.5 n=5
xGnP®-M-5			
1M5	136.2± 0.4 n=3	147.6 ± 0.3 n=3	154.3 ± 0.3 n=3
2M5	134.7 ± 0.5 n=3	145.8 ± 0.2 n=3	153.1 ± 0.4 n=3
3M5	137.4 ± 0.2 n=3	148.14 ± 0.4 n=3	154.7 ± 0.3 n=3
4M5	139.6 ± 0.3 n=3	150.0 ± 0.5 n=3	156.5 ± 0.4 n=3
5M5	140.9 ± 0.1 n=3	150.7 ± 0.3 n=3	157.2 ± 0.4 n=3
6M5	139.3 ± 0.3 n=3	149.8 ± 0.1 n=3	156.4 ± 0.1 n=3
xGnP®-C-300			
1C300	132.2 ± 0.4 n=3	145.3 ± 0.2 n=3	152.7 ± 0.3 n=3
2C300	120.6 ± 0.5 n=3	134.8 ± 0.1 n=3	143.4 ± 0.2 n=3
3C300	132.5 ± 0.6 n=3	145.1 ± 0.1 n=3	152.7 ± 0.4 n=3
4C300	135.0 ± 0.9 n=3	147.4 ± 0.3 n=3	154.9 ± 0.3 n=3
5C300	133.0 ± 0.7 n=3	145.6 ± 0.1 n=3	153.3 ± 0.3 n=3
6C300	132.8 ± 0.5 n=3	145.4 ± 0.1 n=3	153.4 ± 0.2 n=3
TC307			
1TC307	132.7 ± 0.6 n=4	146.2 ± 0.4 n=4	154.0 ± 0.3 n=4
2TC307	134.6 ± 0.4 n=4	148.3 ± 0.4 n=4	155.7 ± 0.3 n=4
3TC307	132.4 ± 0.5 n=4	145.5 ± 0.0 n=4	153.3 ± 0.1 n=4
4TC307	135.4 ± 0.3 n=4	148.8 ± 0.1 n=4	156.4 ± 0.1 n=4

4.5 Differential Scanning Calorimeter (DSC) Results

The glass transition temperature (T_g) for the neat epoxy was found to be about 143 °C. Table 4-9 shows the average glass transition temperature for all formulations of xGnP®-M-15 in epoxy. The T_g follows a similar trend as the storage modulus onset, peak of the loss modulus and peak of the tan delta for the DMA test. The T_g decreases until it hits the approximate percolation threshold and then begins to increase again.

Table 4-9: DSC results for xGnP®-M-15 in epoxy

Formulation	T_g (°C)
Neat Epoxy	143.3 ± 0.7 n=7
1M15	145.3 ± 1.5 n=8
2M15	143.3 ± 1.0 n=8
3M15	138.7 ± 2.4 n=8
4M15	144.3 ± 1.0 n=8
5M15	145.2 ± 1.9 n=8
6M15	145.6 ± 2.4 n=8

4.6 Microscopy Results

4.6.1 xGnP®-M-15 in Epoxy Microscopy Results[6]

Figure 4-18 shows the random dispersion of 5 wt % GNP (see white nanoplatelet geometry) in epoxy. Figure 4-19 shows the FESEM image of a tensile fracture surface for the 5 wt % GNP in epoxy composite. This figure clearly shows the platelet shape of the GNP coming out of the fracture surface (z direction). The epoxy is seen in the ''x–y plane'' of Figure 4-19.

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Citation:
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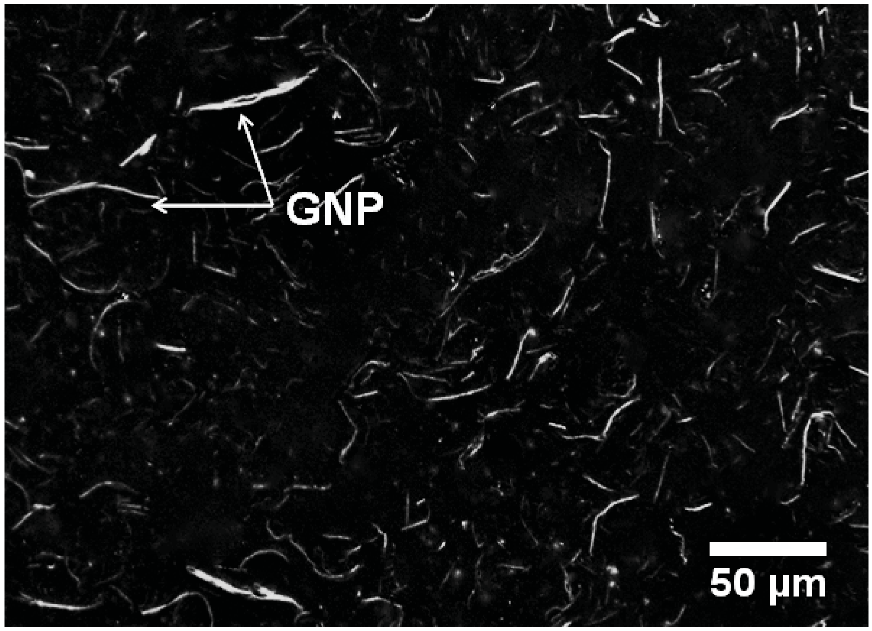

Figure 4-18: Optical Microscope micrograph of 5 wt% graphene nanoplatelets in epoxy

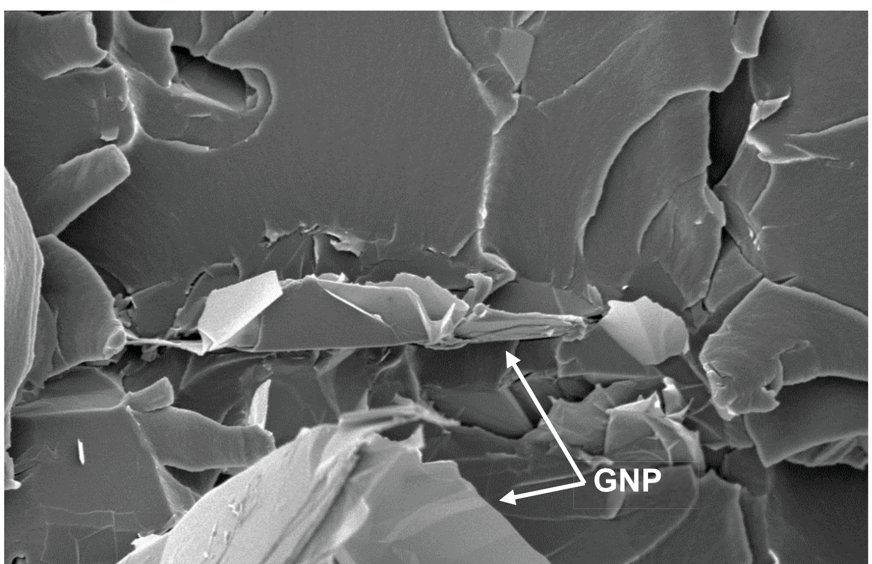

Figure 4-19: Field emission scanning electron microscope micrograph of 5 wt% graphene nanoplatelets in epoxy

4.6.2 xGnP®-M-5 in Epoxy and xGnP®-C-300 in Epoxy Microscopy Results[7]

Figure 4-20 shows the random dispersion of 5wt% xGnP®-M-5 (see white nanoplatelet geometry) in epoxy. Figure 4-21 shows the FESEM image of a tensile fracture surface for the 5wt% xGnP®-M-5 in epoxy composite. This figure clearly shows the platelet shape of the GNP coming out of the fracture surface (z direction). Figure 4-22 shows the random dispersion of 4wt% xGnP®-C-300 in epoxy. Figure 4-23 shows the FESEM image of a tensile fracture surface for the 4wt% xGnP®-C-300 in epoxy. As expected, the xGnP®-C-300 flake (~2 mm) is smaller than the xGnP®-M-5 (~5 mm) flake shown in Figure 4-21.

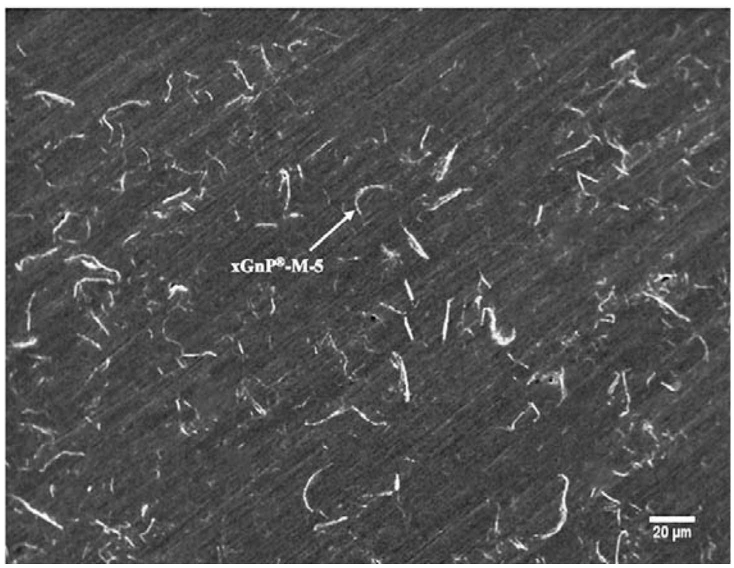

Figure 4-20: Environmental scanning electron microscope micrograph of 5 wt% xGnP®-M-5 in epoxy

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[7] The material contained within this section has been published in the journal *"Journal of Composite Materials."*
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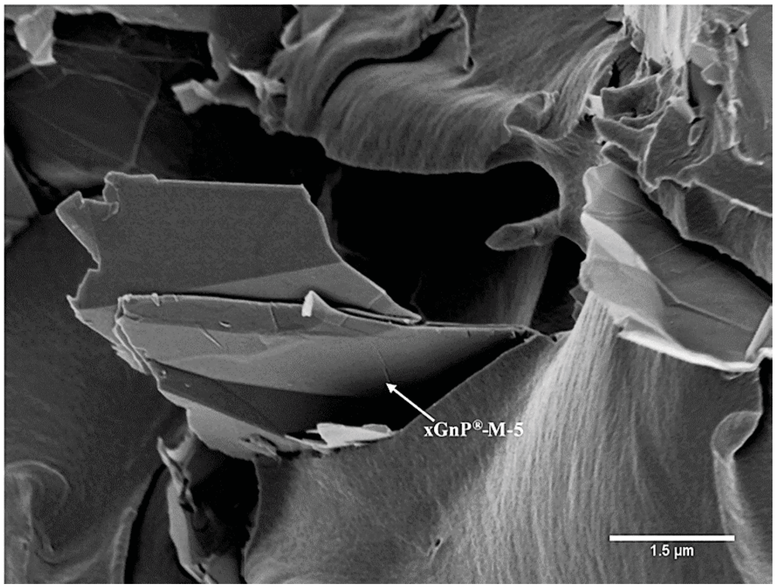

Figure 4-21: Field emission scanning electron microscope micrograph of 5 wt% xGnP®-M-5 in epoxy

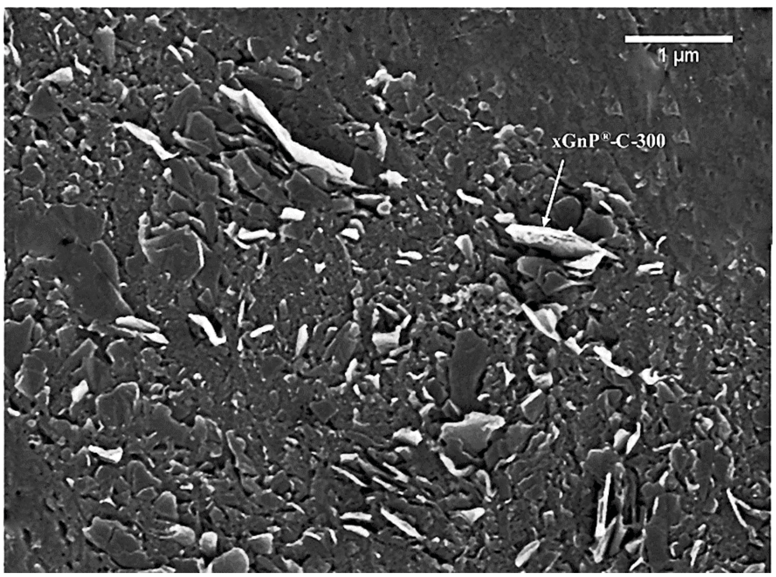

Figure 4-22: Environmental scanning electron microscope micrograph of 4 wt% xGnP®-C-300 in epoxy

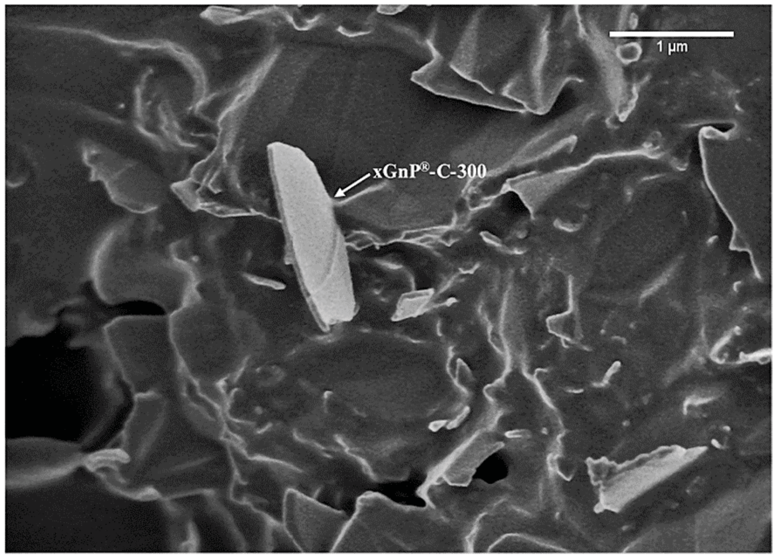

Figure 4-23: Field emission scanning electron microscope micrograph of 4 wt% xGnP®-C-300 in epoxy

4.6.3 xGnP®-C-300 with Continuous Carbon Fiber in Epoxy Microscopy Results

Samples were cut to view the transverse tensile fracture surface. Figure 4-24 shows the transverse tensile fracture of 2 wt% xGnP®-C-300/68 wt% carbon fiber/ 30 wt% epoxy composite. This figure clearly shows the xGnP®-C-300 on top of a carbon fiber.

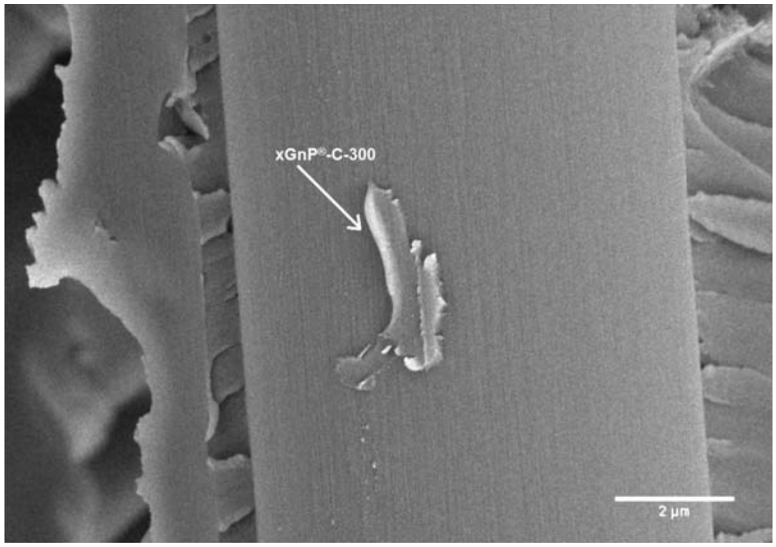

Figure 4-24: Field Emission Scanning Electron Microscope Image of the Fracture Surface of xGnP®-C-300/Carbon Fiber/Epoxy Composite

4.6.4 Asbury TC307 in Epoxy Microscopy Results

Figure 4-25 shows a FESEM image of a tensile fracture surface for 4 wt% TC307 in epoxy. This figure appears to shows a three-dimensional random arrangement of TC307 in epoxy. Figure 4-26 is also a FESEM image of the same fracture surface using a higher magnification. This figure shows the platelet shape of GNP protruding out of the fracture surface (z-direction).

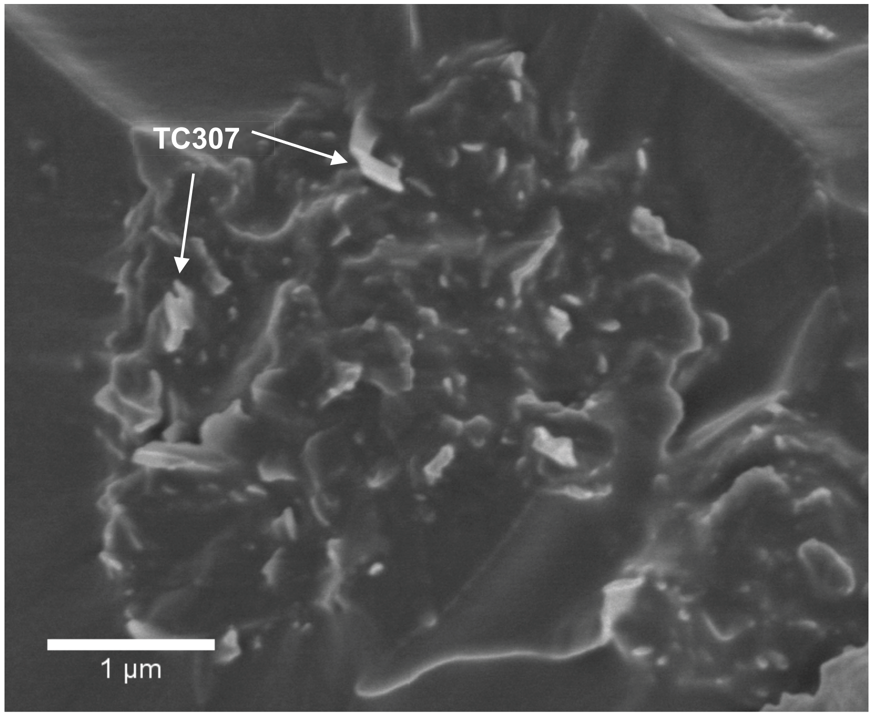

Figure 4-25: Field Emission Scanning Electron Micrograph of 4 wt% TC307 in Epoxy

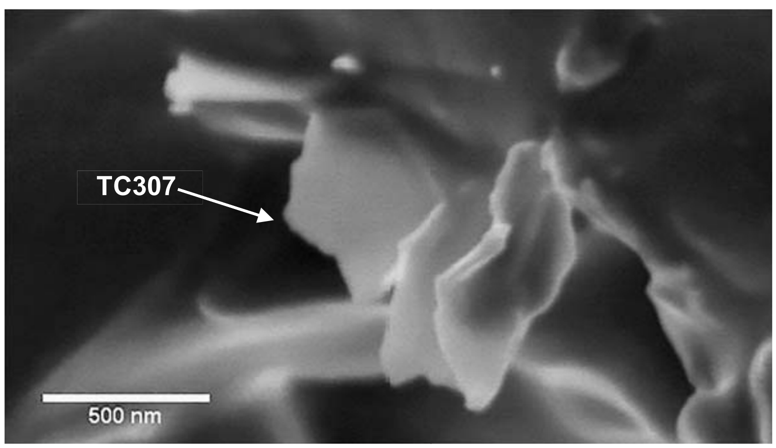

Figure 4-26: Field Emission Scanning Electron Micrograph of 4 wt% TC307 in Epoxy at Higher Magnification

4.7 References

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5 Halpin-Tsai Modulus Modeling

The Halpin–Tsai model predicts the tensile modulus of composite materials using the aspect ratio and volume fraction of the filler as well as the tensile moduli of the matrix and filler. For unidirectional, discontinuous filler composites, the Halpin–Tsai model predicts the composite tensile modulus in both the longitudinal direction and the transverse direction using Equations 5-1 and 5-2 shown below:

$$\frac{E_L}{E_M} = \frac{1 + \xi\eta_L V_f}{1 - \eta_L V_f} \tag{5-1}$$

$$\frac{E_T}{E_M} = \frac{1 + 2\eta_T V_f}{1 - \eta_T V_f} \tag{5-2}$$

where E_L is the longitudinal composite tensile modulus, E_T is the transverse composite tensile modulus, E_M is the tensile modulus of the matrix, L/d is the filler aspect ratio, V_f is the volume fraction of filler, and ξ is the filler shape factor [1-4]. The parameters η_L and η_T are given in Equations 5-3 and 5-4 shown below:

$$\eta_L = \frac{\left(\frac{E_f}{E_M}\right) - 1}{\left(\frac{E_f}{E_M}\right) + \xi} \tag{5-3}$$

$$\eta_T = \frac{\left(\frac{E_f}{E_M}\right) - 1}{\left(\frac{E_f}{E_M}\right) + 2} \tag{5-4}$$

where E_f is the tensile modulus of the filler [6-9]. Equations 5-5 and 5-6 are used for the two-dimensional (2D) random orientation of fillers and the three-dimensional (3D) random orientation of fillers are shown below:

$$E_C = \frac{3}{8}E_L + \frac{5}{8}E_T \quad \text{2D Randomly oriented filler} \qquad (5\text{-}5)$$

$$E_C = \frac{1}{5}E_L + \frac{4}{5}E_T \quad \text{3D Randomly oriented filler} \qquad (5\text{-}6)$$

where E_C is the composite tensile modulus [2,3].

For all formulations, E_M, the tensile modulus of the matrix was measured experimentally to be 2.72 GPa. To model the GNP/epoxy system, filler information is needed. Graphene sheets have a tensile modulus of 1060 GPa in the plane of the sheet [5]. GNP is made up of multiple sheets stacked on each other. When tensile loads are transferred to the GNP particles from the polymer, the van der Waals's dispersion bonding between layers is likely to fail before graphitic carbon–carbon bonding within the sheets fails, leading to further exfoliation of the particle. Hence, for the Halpin–Tsai model, the tensile modulus of GNP was equal to the modulus of exfoliation in the graphite c-axis (through-the-plane) of 36.5 GPa [6]. For platelets, the filler shape factor, ξ, is equal to 0.667 (L/d) [7]. In prior work, Kalaitzidou et al. used the Halpin–Tsai model with ξ is equal to 0.667 (L/d) to successfully model the tensile modulus of GNP/polypropylene composites that were produced by extrusion and then injection molding [8].

5.1 Halpin-Tsai Model for xGnP®-M-15 in Epoxy

Figure 5-1 shows the tensile modulus, the scaled nanoindentation modulus, the 2D and 3D Halpin-Tsai Models for xGnP®-M-15. The L/d ratio used was equal to 2143 (length = 15,000 nm and thickness = 7 nm, same as ‘as received’). The 2D Halpin–Tsai model fits the experimental data well. It is likely that during composite fabrication, a random 2D filler orientation was obtained (see Figure 4-18). This figure shows a random dispersion of the GNP in the x-y plane. The GNP flakes are sticking out in the z plane.

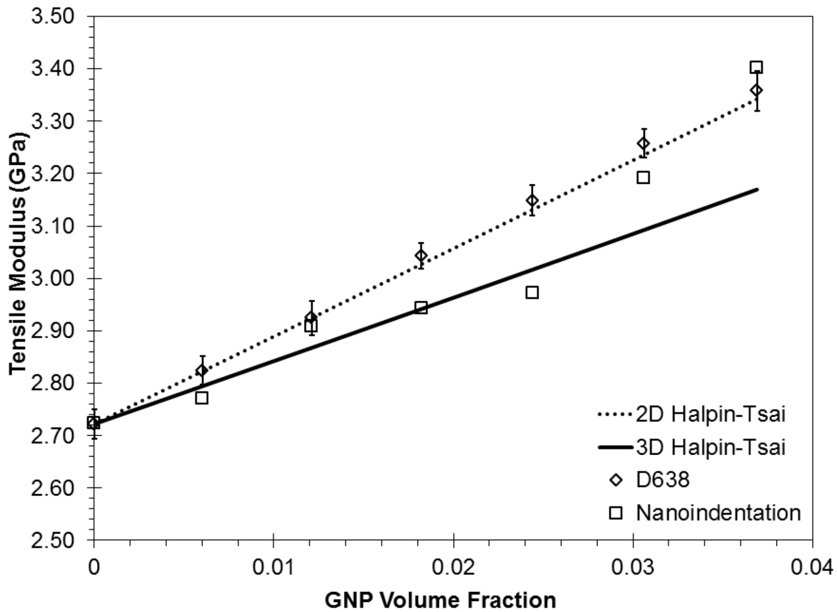

Figure 5-1: Tensile Modulus, Scaled Nanoindentation Modulus, and Halpin-Tsai Models for xGnP®-M-15 in Epoxy

5.2 Halpin-Tsai Model for xGnP®-M-5 in Epoxy

Figure 5-2 shows the tensile modulus, the scaled nanoindentation modulus, the 2D and 3D Halpin-Tsai Models for xGnP®-M-15. The L/d ratio used was equal to 714.3 (length = 5,000 nm and thickness = 7 nm, same as ‘as received’). The 2D Halpin–Tsai model fits the experimental data well. It is likely that during composite fabrication, a random 2D filler orientation was obtained (see Figure 4-20). This figures also shows a random dispersion in the x-y plane, with the GNP sticking out in the z direction.

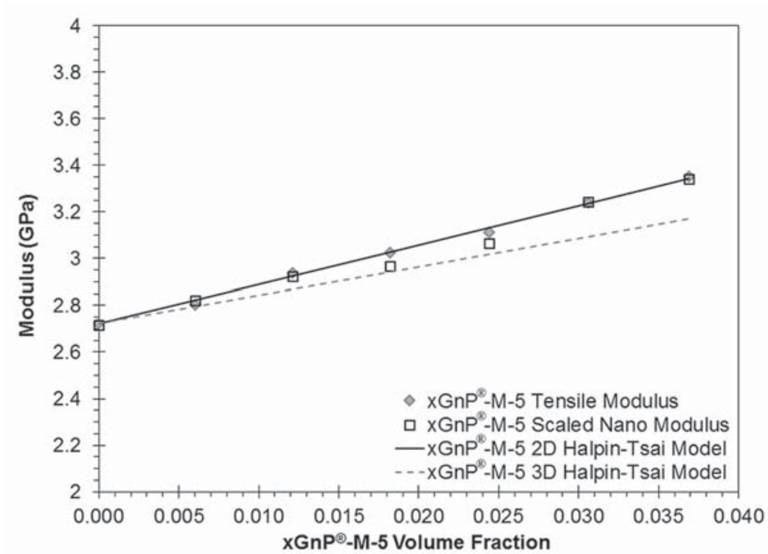

Figure 5-2: Tensile Modulus, Scaled Nanoindentation Modulus, and Halpin-Tsai Models for xGnP®-M-5 in Epoxy

5.3 Halpin-Tsai Model for xGnP®-C-300 in Epoxy

Figure 5-3 shows the tensile modulus, the scaled nanoindentation modulus, the 2D and 3D Halpin-Tsai Models for xGnP®-C-300 in epoxy. The L/d ratio used was equal to 1000 (length = 2,000 nm and thickness = 2 nm, same as 'as received'). The 3D Halpin–Tsai model fits the experimental data well. It is likely that during composite fabrication, a random 3D filler orientation was obtained (see Figure 4-22). This figure shows a random dispersion in all three planes (x-y-z).

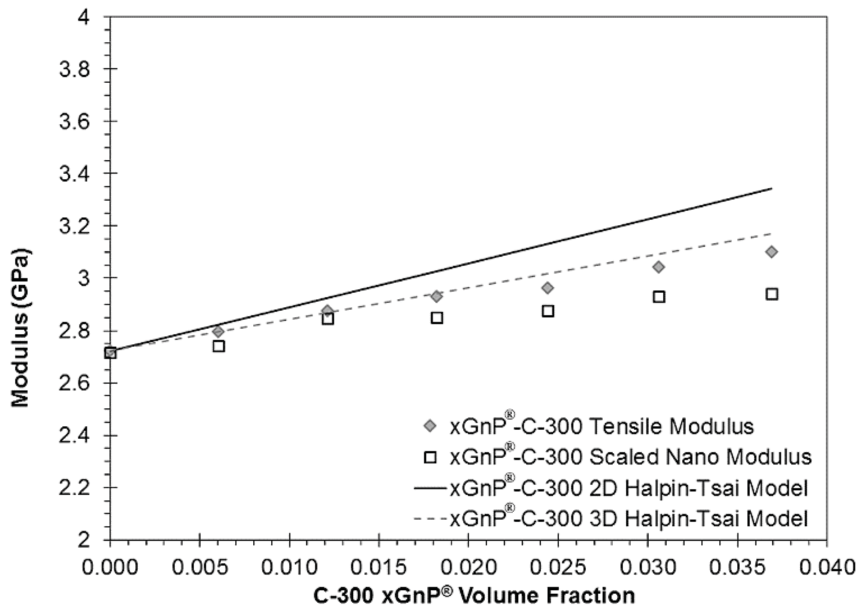

Figure 5-3: Tensile Modulus, Scaled Nanoindentation Modulus, and Halpin-Tsai Models for xGnP®-C-300 in Epoxy

5.4 Halpin-Tsai Model for TC307 in Epoxy

Figure 5-4 shows the tensile modulus, the scaled nanoindentation modulus, the 2D and 3D Halpin-Tsai Models for TC307 in epoxy. The L/d ratio used was equal to 500 (length = 1,000 nm and thickness = 2 nm, same as 'as received'). The 3D Halpin–Tsai model fits the experimental data well. It is likely that during composite fabrication, a random 3D filler orientation was obtained (see Figure 4-25). This figure also shows a random dispersion in three-dimensions, thus visually confirming the 3D Halpin-Tsai model.

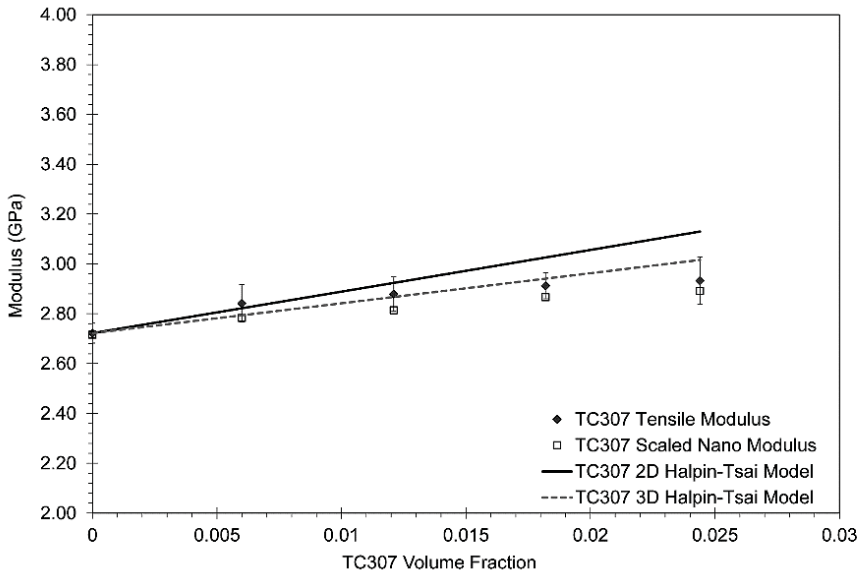

Figure 5-4: Tensile Modulus, Scaled Nanoindentation Modulus, and Halpin-Tsai Models for TC307 in Epoxy

5.5 References

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6 Conclusions and Future Work

6.1 Tensile Properties

6.1.1 Neat Epoxy and GNP/Epoxy Composite Tensile Properties

Adding carbon fillers to a polymer will often change the mechanical properties. It was observed that the addition of any type of GNP will increase the tensile modulus. The modulus for the neat epoxy was measured to be ~2.72 GPa. The addition of 6 wt% (3.69 vol%) xGnP®-M-15 in epoxy increased the tensile modulus to ~3.36 GPa. 6 wt% (3.69 vol%) xGnP®-M-5 in epoxy was very close to xGnP®-M-15 with a modulus of ~3.35 GPa. Where 6 wt% (3.69 vol%) xGnP®-C-300 in epoxy only increased the modulus to ~3.10 GPa. The maximum amount of TC307 added to epoxy was 4 wt% (2.44 vol%), this increased the tensile modulus to ~2.93 GPa.

The stress and strain for the xGnP®-M-15 in epoxy and xGnP®-M-5 in epoxy decreased rapidly as more GNP was added. The stress and strain for xGnP®-C-300 and TC307 stays constant until about 4 wt% and then begins to decrease slowly as more GNP is added. The ultimate tensile stress and strain at ultimate tensile stress for neat epoxy was measured to be ~77.6 MPa and ~8.0% respectively. For 4 wt% (2.44 vol %) xGnP®-M-15 in epoxy, stress was measured to be ~41.9 MPa with ~2.2% strain. For 4 wt% (2.44 vol %) xGnP®-M-5 in epoxy, stress was found to be ~43.9 MPa, and strain was ~2.3%. For 4 wt% (2.44 vol %) xGnP®-C-300 in epoxy, strength was measured to be ~75.8 MPa and ~4.6% (closer to neat epoxy than the M grade GNPs). For 4 wt% (2.44 vol %) TC307 in epoxy, ~75.8 MPa was found for stress and ~4.6% for strain.

The two M-grade GNPs showed very similar properties to one another, the only difference between the two M-grades was the platelet diameter. The larger one (xGnP®-M-15) had a diameter of ~3 times that of the than the smaller (xGnP®-M-5). The xGnP®-C-300 and the TC307 showed similar properties to one another. The size, shape, and surface area for the two GNPs were very similar, it was expected to get similar tensile properties.

6.1.2 Continuous Carbon Fiber/Epoxy and Continuous Carbon Fiber/xGnP®-C-300/Epoxy Composite Tensile Properties

For the xGnP®-C-300 with continuous carbon fiber in epoxy composites, the xGnP®-C-300 did not appear to have an effect on the tensile modulus in the axial direction, but increased the tensile modulus in the transverse direction. The axial modulus for the unidirectional continuous carbon fiber/epoxy composite was found to be 134 ± 9 GPa. For the 3 wt% xGnP®-C-300 with unidirectional continuous carbon fiber/epoxy, the modulus was measured to be 137 ± 9 GPa. The continuous carbon fiber tensile properties dominate over the GNP/epoxy tensile properties in the axial direction. The transverse modulus increases from 7.81 GPa for CCF/epoxy to 8.29 GPa for 2 wt% xGnP®-C-300 with CCF/epoxy.

6.1.3 Tensile Property Summary

Based on the tensile properties obtained for all four types of GNP added to epoxy, the optimal GNP to use would be the xGnP®-C-300 or TC307. These GNPs had approximately the same effect on the tensile properties of the composite. It is recommended that 2 wt% of either xGnP®-C-300 or TC307 be added to epoxy. At this filler loading the strength and strain of the composite are still close to that of the neat epoxy, and the tensile modulus increases slightly. This suggests that the load transfer between the GNP and the epoxy is better than the two M-grade GNPs.

6.2 Nanoindentation Properties for Neat Epoxy and GNP/Epoxy Composites

The modulus as determined by nanoindentation for all four types of GNP was 1.33 times greater than the macroscopic tensile modulus. This was thus used as a scaling factor, a similar trend has been noticed for most polymer composites. It is likely due to pile-up of material around the indenter tip, the Oliver-Pharr method does not take into account the viscoelasticity of polymer based materials. The modulus measured by nanoindentation for the neat epoxy before scaling was measured to be ~3.61 GPa. 6 wt% xGnP®-M-15 in epoxy had a modulus of ~4.44 GPa. The modulus measured for 6 wt% xGnP®-M-5 in epoxy was ~4.45 GPa. For 6 wt% xGnP®-C-300 in epoxy the modulus was found to be ~3.91 GPa. And for 4 wt% TC307 in epoxy ~3.85 GPa was measured. The modulus as determine by nanoindentation for all the GNPs showed a similar trend to the modulus as determined by macroscopic tensile tests.

6.3 Electrical Resistivity Properties for Neat Epoxy and GNP/Epoxy Composites

Electrical resistivity (ER) tests were conducted for all three types of XG Sciences Inc GNPs. This data was useful for finding the percolation threshold (the point at which the electrical resistivity decreases dramatically). The percolation threshold equation is a function of L/d (the aspect ratio), the aspect ratio is used in the Halpin-Tsai model. The neat epoxy has an ER of 2.88×10^{16} Ω-cm. The ER for 2 wt% xGnP®-M-15 in epoxy dropped to 2.61×10^{9} Ω-cm, the percolation threshold for xGnP®-M-15 was approximated to be ~0.99 vol% (~1.6 wt%). The ER for 4 wt% xGnP®-M-5 in epoxy dropped to 3.78×10^{9} Ω-cm. The percolation threshold was approximated to be ~ 2.53 vol % (~ 4.1 wt %) for xGnP®-M-5 and ~2.09 vol% (~3.4 wt%) for xGnP®-C-300. For 4 wt% xGnP®-C-300 in epoxy the ER dropped to 3.48×10^{8} Ω-cm. The approximated percolation threshold matched the filler loading that produced a drop in the electrical resistivity of the composite for all three types of GNP. Since the approximated percolation threshold matched the actual percolation threshold, the vendor given aspect ratios were used for the Halpin-Tsai model.

6.4 Glass Transition Temperature (T_g) for Neat Epoxy and GNP/Epoxy Composites

The glass transition temperature is the transition of a polymer from the “glassy” state to the “rubbery” state. A composite in the “rubbery state is more pliable than one in the “glassy” state, this makes the composite less brittle. The glass transition temperature for xGnP®-M-15 in epoxy was measured two different ways, DSC and DMA tests. The T_g from DMA for neat epoxy (determined from the peak of the tan delta curve) was found to be 155.8 °C ± 0.4 °C (n=6). At around the percolation threshold (~3 wt%) the T_g for xGnP®-M-15 in epoxy drops to about 150.4 °C ± 0.3 °C (n=3). The T_g after this point increased and 155.9 °C ± 0.5 °C (n=5) at 6 wt% xGnP®-M-15 in epoxy. This same trend was observed with the T_g determined by DSC. The neat epoxy T_g was measured to be 143.3 °C ± 0.7 °C (n=7) and decreases to 138.7 °C ± 2.4 °C (n=8) at 3 wt% xGnP®-M-15 in epoxy. It then increased to 145.6 °C ± 2.4 °C (n=8) for 6 wt% xGnP®-M-15 in epoxy.

DMA was used to find the T_g for xGnP®-M-5 in epoxy and xGnP®-C-300 in epoxy. For xGnP®-M-5 in epoxy T_g drops to 153.1 °C ± 0.4 °C (n=3), this occurs at 2 wt% and it increases to 156.4 °C ± 0.1 °C (n=3) for 6 wt% xGnP®-M-5 in epoxy. For xGnP®-C-300 in epoxy at 2 wt%, the T_g decreases to 143.4 °C ± 0.2 °C (n=3). The T_g increased to 153.4 °C ± 0.2 °C (n=3) for 6 wt% xGnP®-C-300 in epoxy. Since 2 wt% xGnP®-C-300 in epoxy has the lowest glass transition temperature, it is the optimum formulation.

6.5 Halpin-Tsai Modeling for Neat Epoxy and GNP/Epoxy Composites

The Halpin-Tsai model is an existing model that takes into account the tensile properties and volume fractions of the matrix and the filler material. It also accounts for the composite filler geometry. A modified shape factor was used to predict the modulus behavior of a platelet shaped filler geometry. For the two larger GNPs (xGnP®-M-15 and xGnP®-M-5), the two-dimensional randomly oriented filler Halpin-Tsai model fit the best. By looking at images of the GNP in epoxy it was apparent that a 2D dispersion was obtained. Since the platelet diameter is much larger than the thickness it was expected to see this 2D dispersion.

For the two smaller GNPs (xGnP®-C-300 and TC307) the platelet diameter is closer to the platelet thickness and it is believed that a 3D dispersion was obtained. This is visible through looking at images of the two types of GNP in epoxy. Thus, a three-dimensional randomly oriented Halpin-Tsai model fit the data the best.

6.6 Overall Conclusions

Four types of GNP were added to an epoxy matrix to produce polymer composites. The composites were tested for mechanical properties. A different mixing method was developed for each of the four types of GNP to ensure that good dispersion was achieved. Tensile properties were measured and the fracture surface produced was imaged to see the dispersion in the composite. From tensile testing results, 2 wt% of xGnP®-C-300 or TC307 in epoxy is recommended. This formulation increases the composite tensile modulus without sacrificing the strength and strain dramatically.

When xGnP®-C-300 is added to a unidirectional continuous carbon fiber composite, there is no change in the composite tensile modulus. The carbon fiber dominates the tensile properties for a unidirectional carbon fiber composite.

6.7 Recommendations for Future Work

This project showed that adding GNP to an epoxy can increase the tensile modulus, it would be beneficial to see if the GNP has a similar effect on a different aerospace epoxy system. There are also many other different types of GNP available from XG Sciences Inc., Asbury Carbons, and others. Different surface treatments on the GNP may allow for chemical bonding between the Epoxy and the GNP. If the chemical bond created between the GNP and epoxy is strong then load transfer between the filler and the matrix will occur.

Load transfer between the GNP and epoxy should be investigated. It is important to have good load transfer between the filler and the matrix, this allows for enhanced mechanical properties and not just increased stiffness. If the load transfer is not good then the filler is like a void space in the epoxy and will create a weak point in the composite. For GNP with a higher specific surface area, it appears as though the load transfer between the GNP and epoxy is better.

The surface area of the platelets seems to play in important role in the tensile properties. The larger the surface area, the better the ultimate tensile stress and strain at ultimate tensile stress of the composite. Only by having a controlled set of tests (hold diameter and thickness constant but change surface areas) can the effect of surface area on the tensile properties be determined. Specifically XG Sciences Inc. has an H-Grade GNP, the dimensions for the H-Grade are similar to the M-Grade the only difference is the surface area (~60 m^2/g for H-Grade). 1-6 wt% of xGnP®-H-15 and xGnP®-H-5 in EPON 862/EPIKURE Curing Agent W should be fabricated and tested.

All of the mechanical properties were determined at room temperature. It is not likely that these composites would be used at ambient conditions. For aircrafts, they are usually exposed to high temperatures. It is recommended that the tensile properties at elevated temperatures (just below and above the T_g) be measured to determine if the composite can withstand higher temperatures.

For carbon fiber composites, stiffness only tells part of the mechanical story. Fracture toughness testing can tell more about the failure mechanisms associated with a CCF/epoxy composite. It is important to ensure that there is good adhesion between the carbon fiber and the epoxy matrix. Delamination is one of the main modes of failure for a continuous carbon fiber composite. It is recommended that fracture toughness tests be conducted to better understand the failure mechanisms for the continuous carbon fiber/epoxy composites.

Appdenix A: Tensile Results

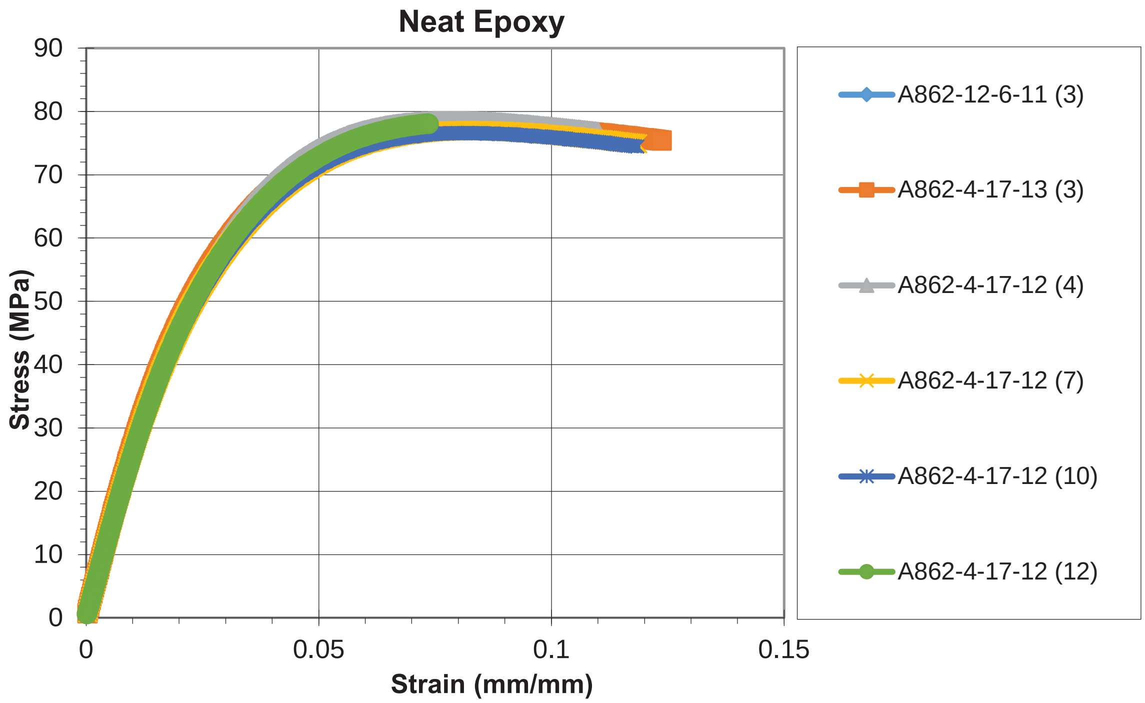


Figure A-1: Tensile Results for Neat Epoxy

Table A-1: Tensile Results for Neat Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-12-6-11	3	76.998	8.013	76.986	8.189	2.717
A862-4-17-12	3	77.854	8.180	75.559	12.209	2.757
A862-4-17-12	4	78.900	7.898	77.279	10.858	2.782
A862-4-17-12	7	76.976	8.272	74.873	11.852	2.710
A862-4-17-12	10	76.591	8.201	74.569	11.701	2.691
A862-4-17-12	12	78.065	7.335	78.065	7.335	2.678
Average		**77.56**	**7.98**	**76.22**	**10.36**	**2.72**
Standard Deviation		**0.86**	**0.35**	**1.42**	**2.08**	**0.04**
Count		**6**	**6**	**6**	**6**	**6**

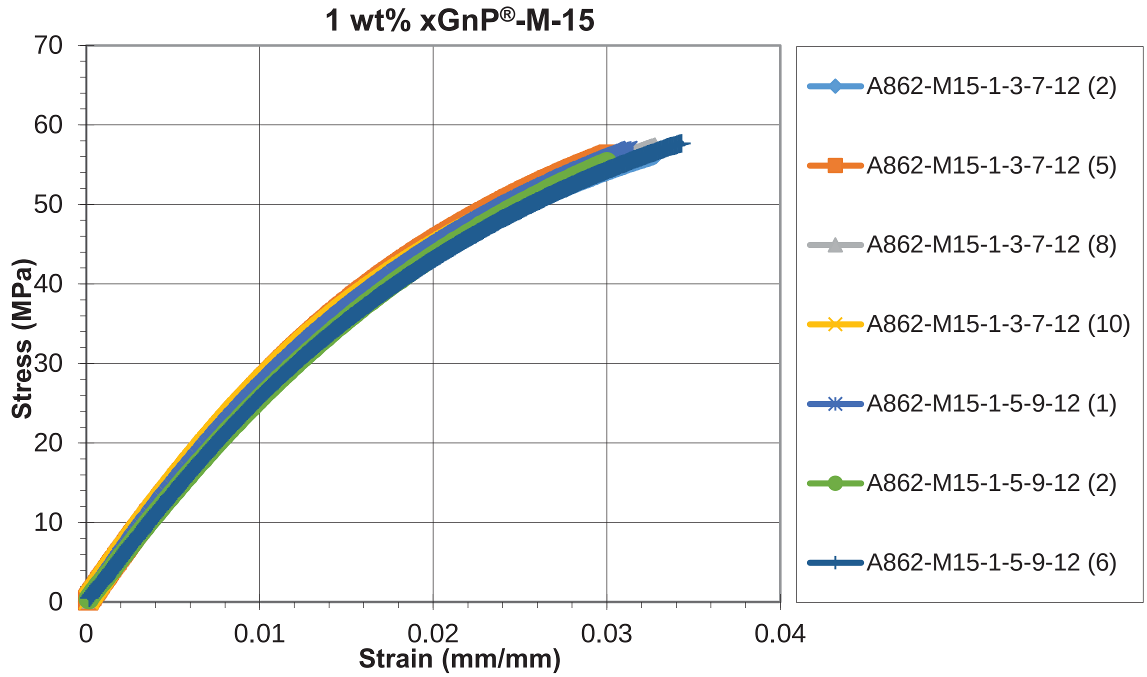


Figure A-2: Tensile Results for 1 wt% xGnP®-M-15 in Epoxy

Table A-2: Tensile Results for 1 wt% xGnP®-M-15 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (MPa)
A862-M15-1-3-7-12	2	56.001	3.282	56.001	3.282	2.842
A862-M15-1-3-7-12	5	56.244	3.010	56.244	3.010	2.829
A862-M15-1-3-7-12	8	57.461	3.278	57.461	3.278	2.802
A862-M15-1-3-7-12	10	55.000	3.007	55.000	3.007	2.869
A862-M15-1-5-9-12	1	57.118	3.138	57.118	3.138	2.863
A862-M15-1-5-9-12	2	55.279	3.001	55.279	3.001	2.791
A862-M15-1-5-9-12	6	57.676	3.430	57.676	3.430	2.773
Average		**56.40**	**3.16**	**56.40**	**3.16**	**2.82**
Standard Deviation		**1.05**	**0.17**	**1.05**	**0.17**	**0.04**
Count		**7**	**7**	**7**	**7**	**7**

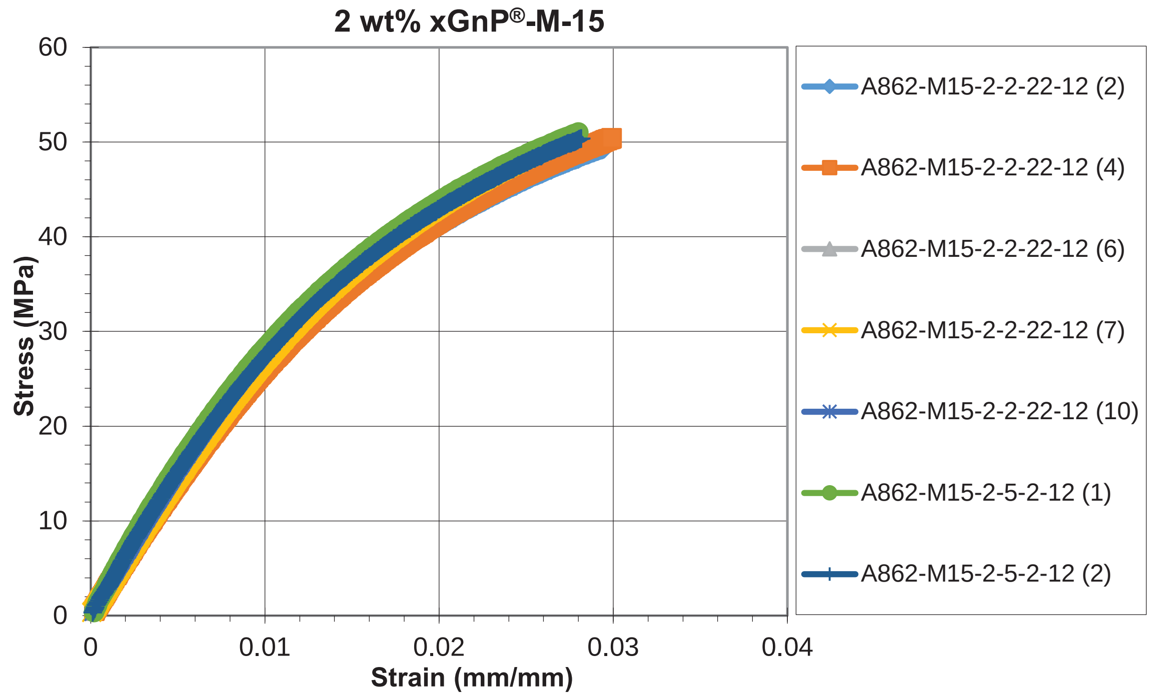


Figure A-3: Tensile Results for 2 wt% xGnP®-M-15 in Epoxy

Table A-3: Tensile Results for 2 wt% xGnP®-M-15 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M15-2-2-22-12	2	49.046	2.946	49.046	2.946	2.889
A862-M15-2-2-22-12	4	50.384	2.991	50.384	2.991	2.899
A862-M15-2-2-22-12	6	49.505	2.616	49.505	2.616	2.862
A862-M15-2-2-22-12	7	48.838	2.561	48.838	2.561	2.914
A862-M15-2-2-22-12	10	50.130	2.704	50.130	2.704	2.971
A862-M15-2-5-2-12	1	50.999	2.799	50.999	2.799	2.973
A862-M15-2-5-2-12	2	50.383	2.816	50.383	2.816	2.962
Average		**49.90**	**2.78**	**49.90**	**2.78**	**2.92**
Standard Deviation		**0.79**	**0.16**	**0.79**	**0.16**	**0.04**
Count		**7**	**7**	**7**	**7**	**7**

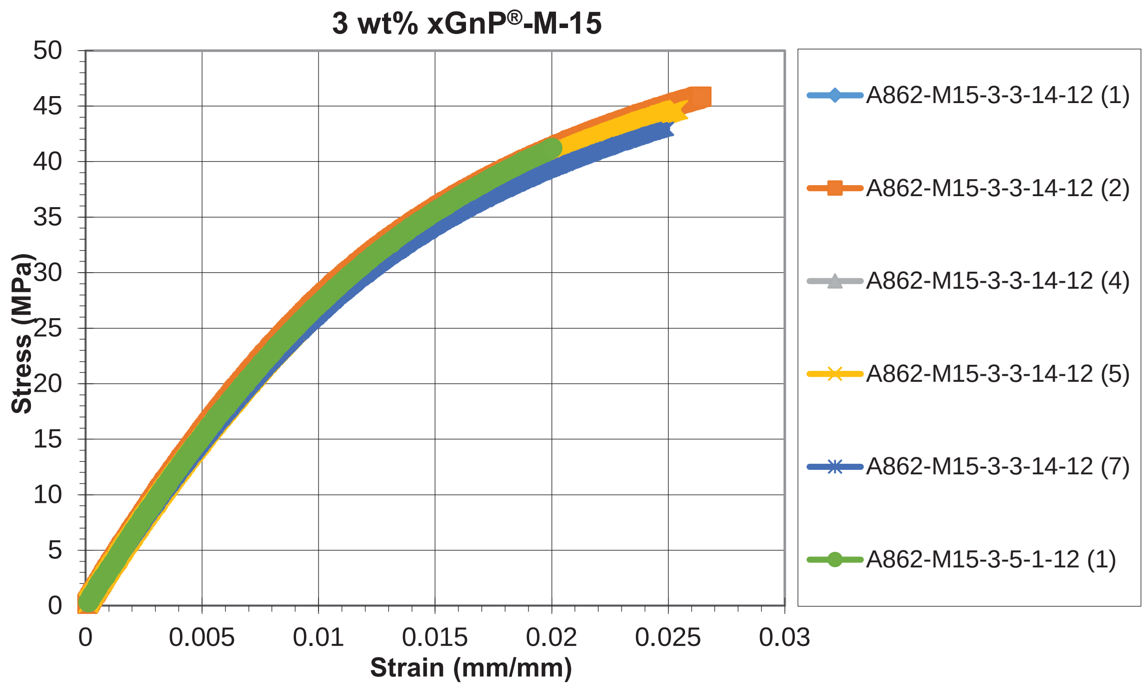


Figure A-4: Tensile Results for 3 wt% xGnP®-M-15 in Epoxy

Table A-4: Tensile Results for 3 wt% xGnP®-M-15 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M15-3-3-14-12	1	40.724	2.096	40.724	2.096	3.054
A862-M15-3-3-14-12	2	45.829	2.642	45.829	2.642	3.074
A862-M15-3-3-14-12	4	43.314	2.384	43.314	2.384	3.008
A862-M15-3-3-14-12	5	44.690	2.544	44.690	2.544	3.043
A862-M15-3-3-14-12	7	42.955	2.495	42.955	2.495	3.004
A862-M15-3-5-1-12	1	41.217	2.002	41.217	2.002	3.076
Average		**43.12**	**2.36**	**43.12**	**2.36**	**3.04**
Standard Deviation		**1.96**	**0.26**	**1.96**	**0.26**	**0.03**
Count		**6**	**6**	**6**	**6**	**6**

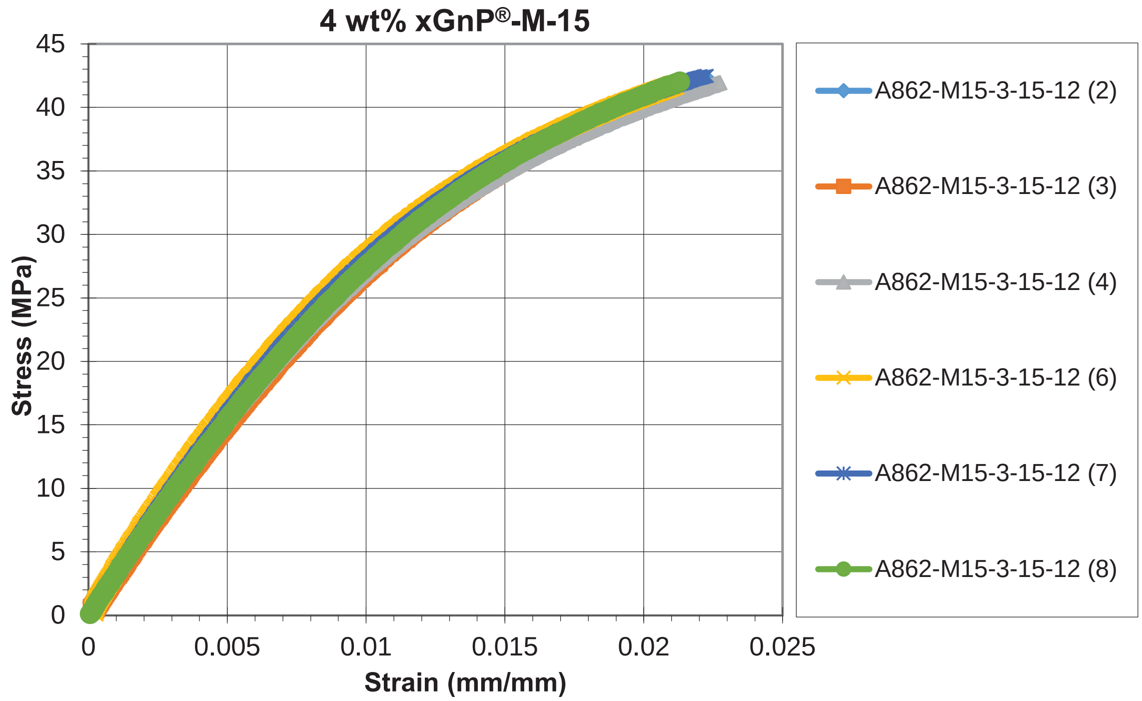


Figure A-5: Tensile Results for 4 wt% xGnP®-M-15 in Epoxy

Table A-5: Tensile Results for 4 wt% xGnP®-M-15 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (MPa)
A862-M15-4-3-15-12	2	42.289	2.230	42.289	2.230	3.169
A862-M15-4-3-15-12	3	41.170	2.096	41.170	2.096	3.104
A862-M15-4-3-15-12	4	41.961	2.274	41.961	2.274	3.124
A862-M15-4-3-15-12	6	41.743	2.111	41.743	2.111	3.193
A862-M15-4-3-15-12	7	42.469	2.226	42.469	2.226	3.180
A862-M15-4-3-15-12	8	42.016	2.130	42.016	2.130	3.124
Average		**41.94**	**2.18**	**41.94**	**2.18**	**3.15**
Standard Deviation		**0.46**	**0.07**	**0.46**	**0.07**	**0.04**
Count		**6**	**6**	**6**	**6**	**6**

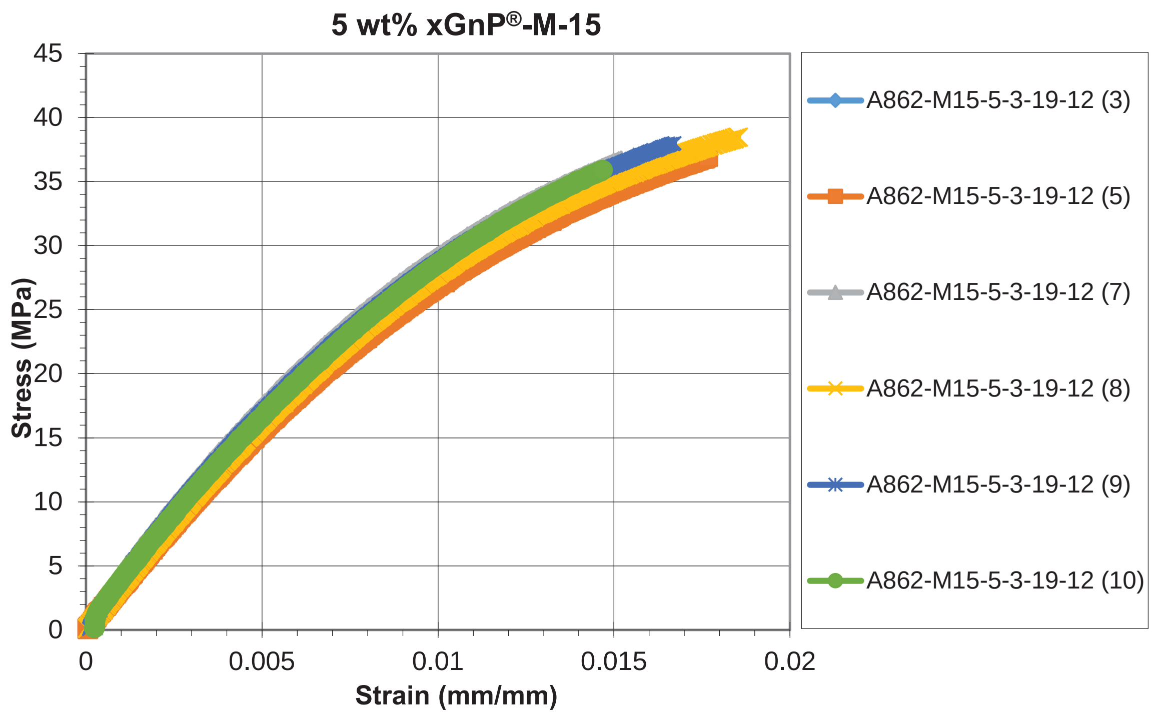


Figure A-6: Tensile Results for 5 wt% xGnP®-M-15 in Epoxy

Table A-6: Tensile Results for 5 wt% xGnP®-M-15 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M15-5-3-19-12	3	36.797	1.626	36.797	1.626	3.238
A862-M15-5-3-19-12	5	36.940	1.767	36.940	1.767	3.197
A862-M15-5-3-19-12	7	36.818	1.521	36.818	1.521	3.291
A862-M15-5-3-19-12	8	38.478	1.853	38.478	1.853	3.266
A862-M15-5-3-19-12	9	38.060	1.674	38.060	1.674	3.278
A862-M15-5-3-19-12	10	35.902	1.468	35.902	1.468	3.275
Average		**37.17**	**1.65**	**37.17**	**1.65**	**3.26**
Standard Deviation		**0.94**	**0.15**	**0.94**	**0.15**	**0.03**
Count		**6**	**6**	**6**	**6**	**6**

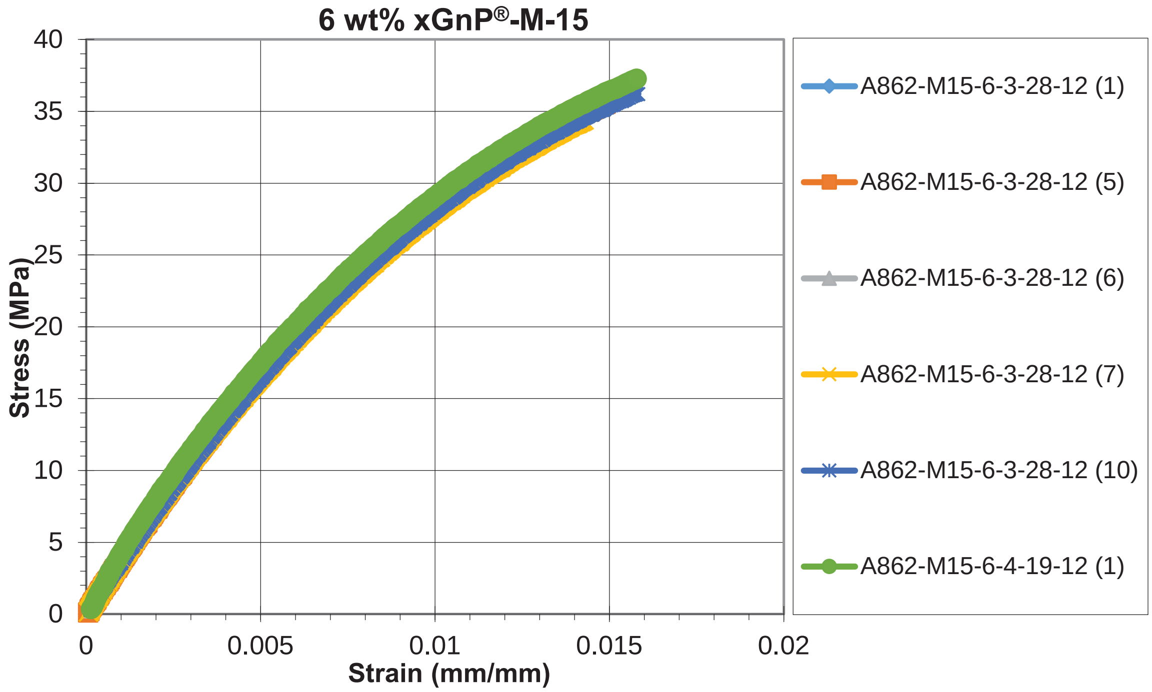


Figure A-7: Tensile Results for 6 wt% xGnP®-M-15 in Epoxy

Table A-7: Tensile Results for 6 wt% xGnP®-M-15 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M15-6-3-28-12	1	33.728	1.369	33.728	1.369	3.284
A862-M15-6-3-28-12	5	35.621	1.491	35.621	1.491	3.376
A862-M15-6-3-28-12	6	35.888	1.497	35.888	1.497	3.385
A862-M15-6-3-28-12	7	34.405	1.428	34.405	1.428	3.313
A862-M15-6-3-28-12	10	36.205	1.583	36.205	1.583	3.373
A862-M15-6-4-19-12	1	37.267	1.578	37.267	1.578	3.413
Average		**35.52**	**1.49**	**35.52**	**1.49**	**3.36**
Standard Deviation		**1.27**	**0.08**	**1.27**	**0.08**	**0.05**
Count		**6**	**6**	**6**	**6**	**6**

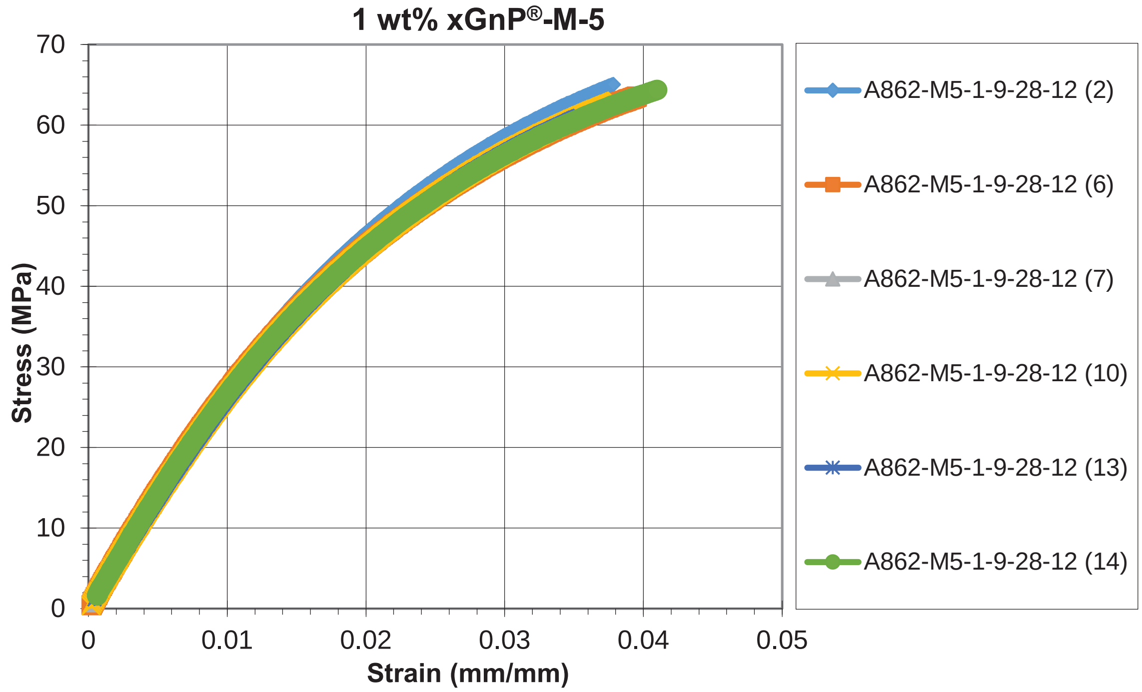


Figure A-8: Tensile Results for 1 wt% xGnP®-M-5 in Epoxy

Table A-8: Tensile Results for 1 wt% xGnP®-M-5 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M5-1-9-28-12	2	65.072	3.783	65.072	3.783	2.907
A862-M5-1-9-28-12	6	63.537	3.951	63.537	3.951	2.824
A862-M5-1-9-28-12	7	62.614	3.660	62.614	3.660	2.771
A862-M5-1-9-28-12	10	62.980	3.809	62.980	3.809	2.787
A862-M5-1-9-28-12	13	61.167	3.541	61.167	3.541	2.734
A862-M5-1-9-28-12	14	64.375	4.100	64.375	4.100	2.803
Average		**63.29**	**3.81**	**63.29**	**3.81**	**2.80**
Standard Deviation		**1.38**	**0.20**	**1.38**	**0.20**	**0.06**
Count		**6**	**6**	**6**	**6**	**6**

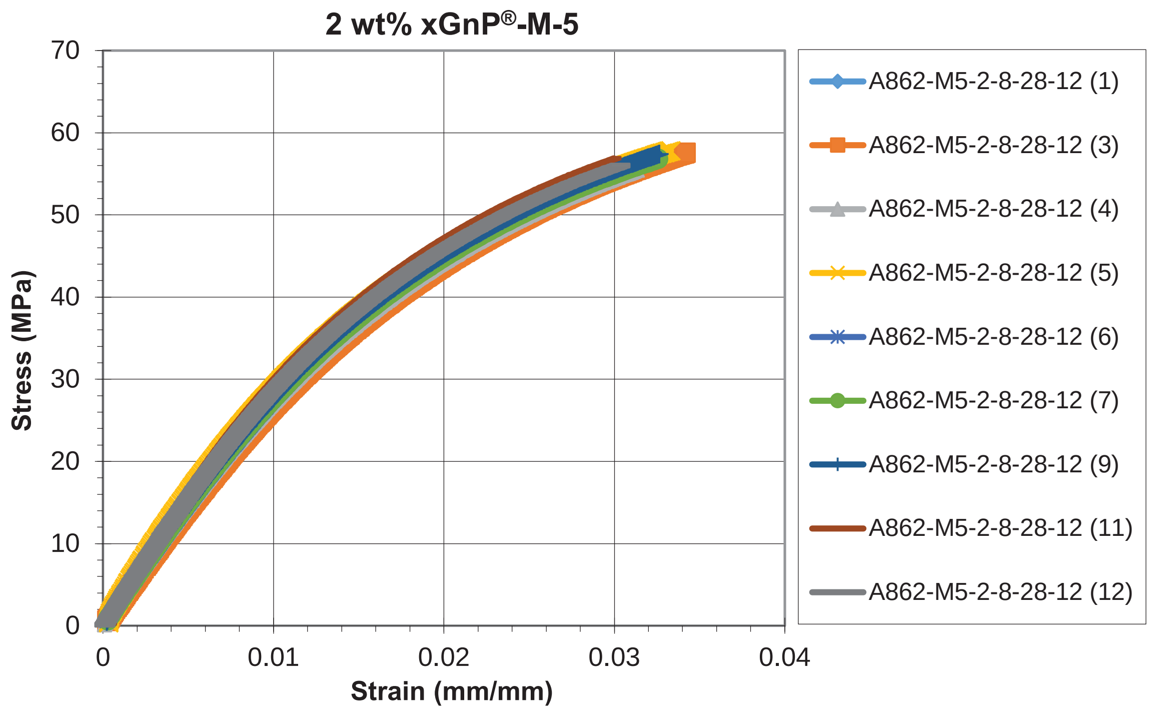


Figure A-9: Tensile Results for 2 wt% xGnP®-M-5 in Epoxy

Table A-9: Tensile Results for 2 wt% xGnP®-M-5 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M5-2-8-28-12	1	56.471	3.115	56.471	3.115	2.953
A862-M5-2-8-28-12	3	57.562	3.418	57.562	3.418	2.874
A862-M5-2-8-28-12	4	55.470	3.135	55.470	3.135	2.915
A862-M5-2-8-28-12	5	57.814	3.332	57.814	3.332	2.942
A862-M5-2-8-28-12	6	56.470	3.152	56.470	3.152	2.931
A862-M5-2-8-28-12	7	56.832	3.257	56.832	3.257	2.942
A862-M5-2-8-28-12	9	57.419	3.265	57.419	3.265	2.951
A862-M5-2-8-28-12	11	57.285	3.054	57.285	3.054	2.987
A862-M5-2-8-28-12	12	56.004	3.034	56.004	3.034	2.961
Average		**56.81**	**3.20**	**56.81**	**3.20**	**2.94**
Standard Deviation		**0.78**	**0.13**	**0.78**	**0.13**	**0.03**
Number		**9**	**9**	**9**	**9**	**9**

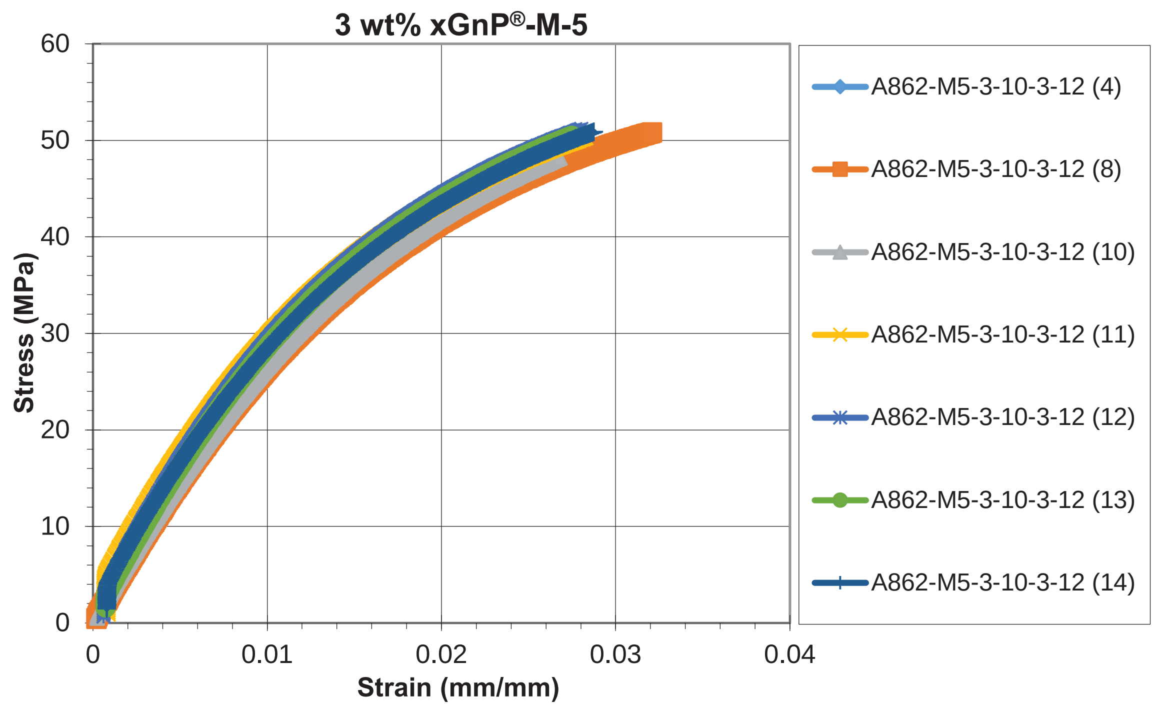


Figure A-10: Tensile Results for 3 wt% xGnP®-M-5 in Epoxy

Table A-10: Tensile Results for 3 wt% xGnP®-M-5 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M5-3-10-3-12	4	48.540	2.8	48.540	2.773	3.016
A862-M5-3-10-3-12	8	50.809	3.2	50.809	3.208	3.023
A862-M5-3-10-3-12	10	48.165	2.7	48.165	2.682	2.986
A862-M5-3-10-3-12	11	50.479	2.8	50.479	2.818	3.037
A862-M5-3-10-3-12	12	51.173	2.8	51.173	2.803	3.045
A862-M5-3-10-3-12	13	50.457	2.7	50.457	2.747	3.039
A862-M5-3-10-3-12	14	50.883	2.9	50.883	2.875	3.035
Average		**50.07**	**2.84**	**50.07**	**2.84**	**3.03**
Standard Deviation		**1.20**	**0.17**	**1.20**	**0.17**	**0.02**
Number		**7**	**7**	**7**	**7**	**7**

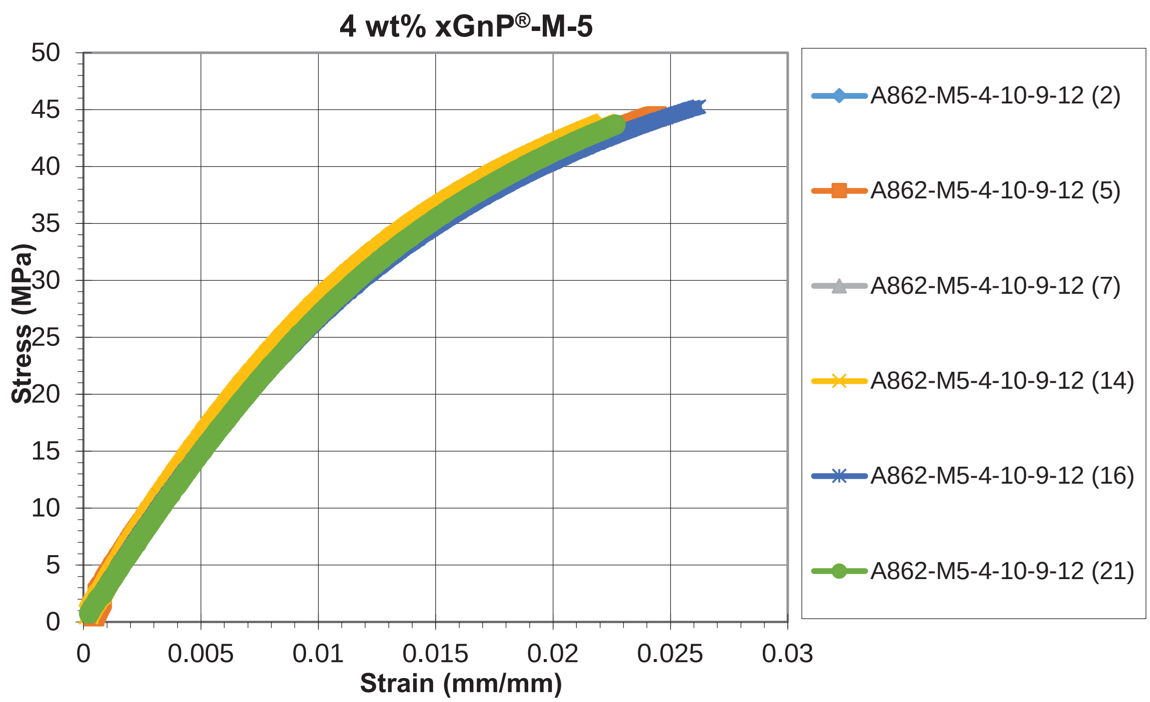


Figure A-11: Tensile Results for 4 wt% xGnP®-M-5 in Epoxy

Table A-11: Tensile Results for 4 wt% xGnP®-M-5 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M5-4-10-9-12	2	43.139	2.193	43.139	2.193	3.136
A862-M5-4-10-9-12	5	44.412	2.440	44.412	2.440	3.069
A862-M5-4-10-9-12	7	43.224	2.276	43.224	2.276	3.146
A862-M5-4-10-9-12	14	43.804	2.223	43.804	2.223	3.194
A862-M5-4-10-9-12	16	45.263	2.622	45.263	2.622	3.087
A862-M5-4-10-9-12	21	43.667	2.266	43.667	2.266	3.023
Average		**43.92**	**2.34**	**43.92**	**2.34**	**3.11**
Standard Deviation		**0.80**	**0.16**	**0.80**	**0.16**	**0.06**
Count		**6**	**6**	**6**	**6**	**6**

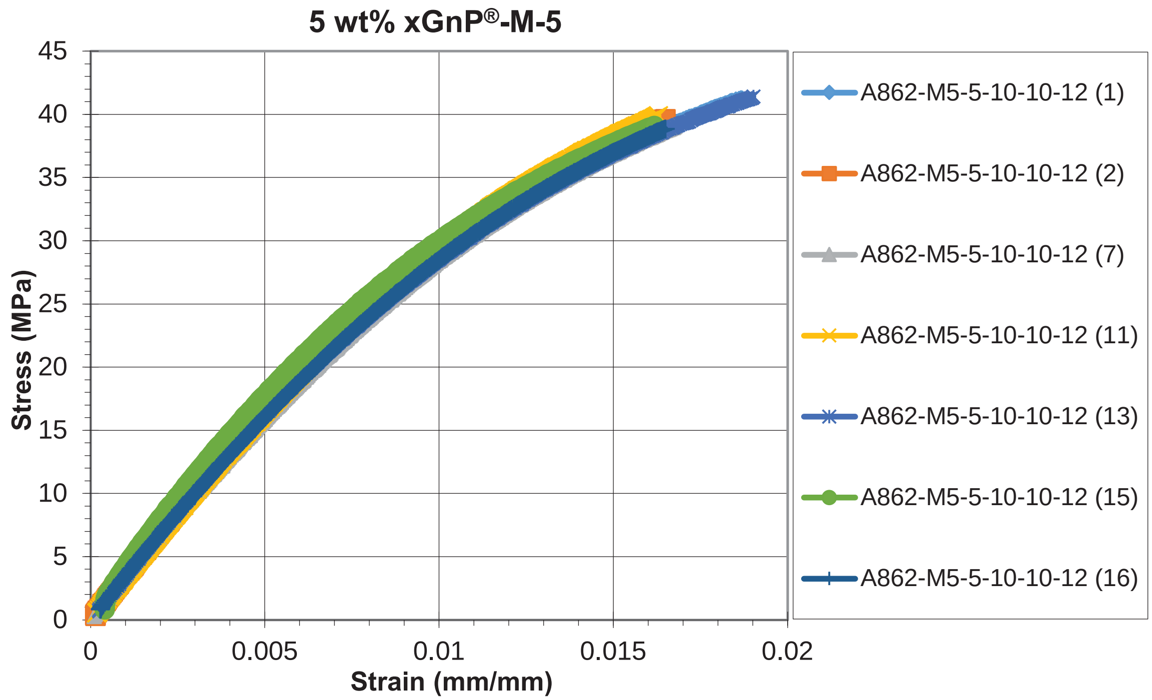


Figure A-12: Tensile Results for 5 wt% xGnP®-M-5 in Epoxy

Table A-12: Tensile Results for 5 wt% xGnP®-M-5 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-M5-5-10-10-12	1	41.304	1.866	41.304	1.866	3.252
A862-M5-5-10-10-12	2	39.607	1.650	39.607	1.650	3.280
A862-M5-5-10-10-12	7	39.034	1.676	39.034	1.676	3.161
A862-M5-5-10-10-12	11	39.886	1.630	39.886	1.630	3.282
A862-M5-5-10-10-12	13	41.401	1.902	41.401	1.902	3.208
A862-M5-5-10-10-12	15	39.088	1.618	39.088	1.618	3.275
A862-M5-5-10-10-12	16	38.857	1.649	38.857	1.649	3.215
Average		**39.88**	**1.71**	**39.88**	**1.71**	**3.24**
Standard Deviation		**1.06**	**0.12**	**1.06**	**0.12**	**0.05**
Count		**7**	**7**	**7**	**7**	**7**

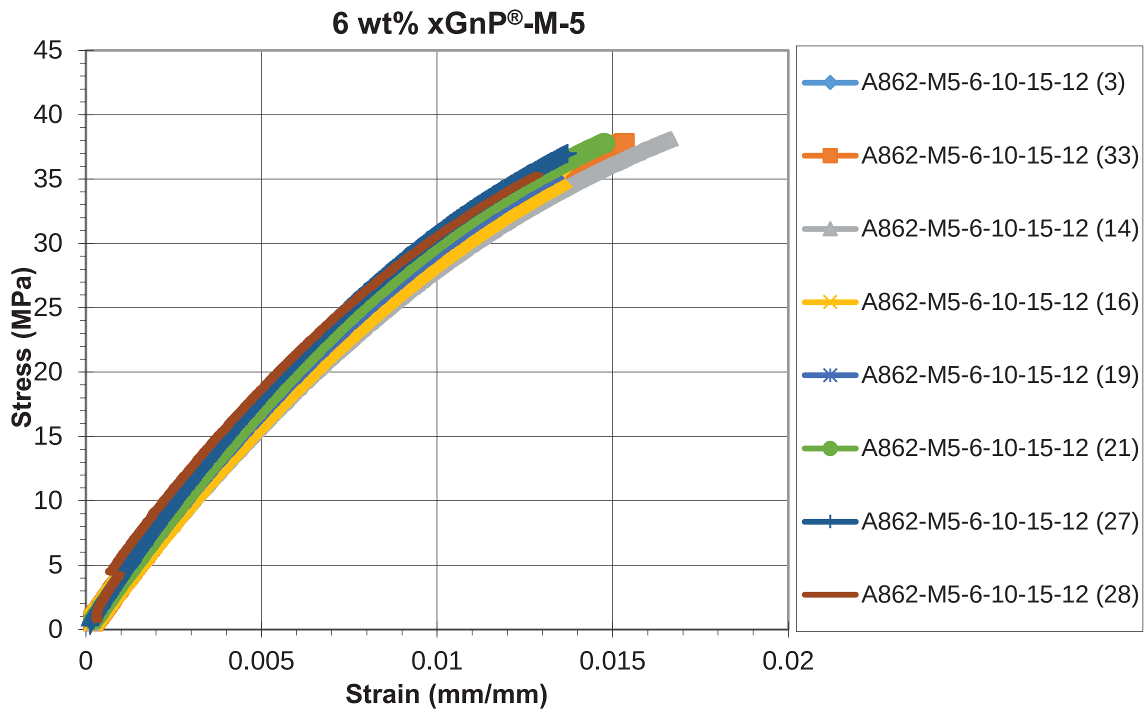


Figure A-13: Tensile Results for 6 wt% xGnP®-M-5 in Epoxy

Table A-13: Tensile Results for 6 wt% xGnP®-M-5 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (MPa)
A862-M5-6-10-15-12	3	34.104	1.294	34.104	1.294	3.305
A862-M5-6-10-15-12	13	37.783	1.534	37.783	1.534	3.330
A862-M5-6-10-15-12	14	38.144	1.667	38.144	1.667	3.241
A862-M5-6-10-15-12	16	35.081	1.359	35.081	1.359	3.304
A862-M5-6-10-15-12	19	35.515	1.343	35.515	1.343	3.366
A862-M5-6-10-15-12	21	37.782	1.474	37.782	1.474	3.421
A862-M5-6-10-15-12	27	37.096	1.378	37.096	1.378	3.480
A862-M5-6-10-15-12	28	35.726	1.304	35.726	1.304	3.357
Average		**36.40**	**1.42**	**36.40**	**1.42**	**3.35**
Standard Deviation		**1.49**	**0.13**	**1.49**	**0.13**	**0.07**
Count		**8**	**8**	**8**	**8**	**8**

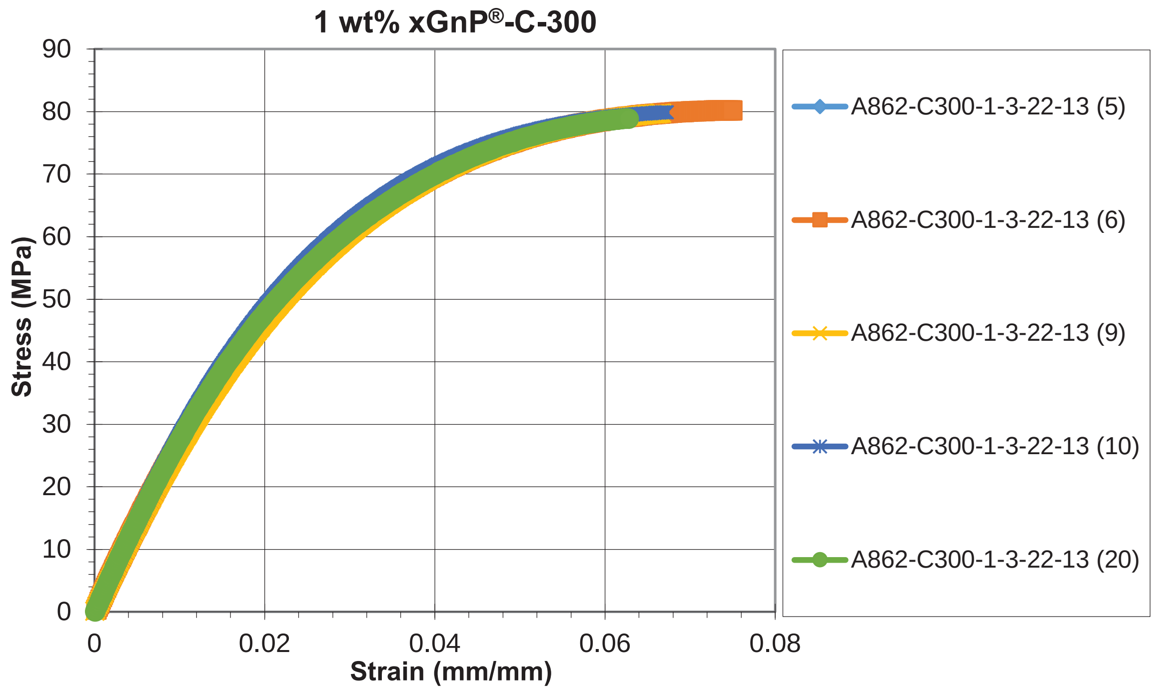


Figure A-14: Tensile Results for 1 wt% xGnP®-C-300 in Epoxy

Table A-14: Tensile Results for 1 wt% xGnP®-C-300 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-C300-1-3-22-13	5	78.989	6.540	78.989	6.540	2.738
A862-C300-1-3-22-13	6	80.209	7.448	80.209	7.448	2.804
A862-C300-1-3-22-13	7	80.006	6.738	80.006	6.738	2.825
A862-C300-1-3-22-13	9	79.739	6.666	79.739	6.666	2.781
A862-C300-1-3-22-13	10	79.892	6.705	79.892	6.705	2.861
A862-C300-1-3-22-13	20	78.931	6.262	78.931	6.262	2.800
Average		**79.63**	**6.73**	**79.63**	**6.73**	**2.80**
Standard Deviation		**0.54**	**0.39**	**0.54**	**0.39**	**0.04**
Count		**6**	**6**	**6**	**6**	**6**

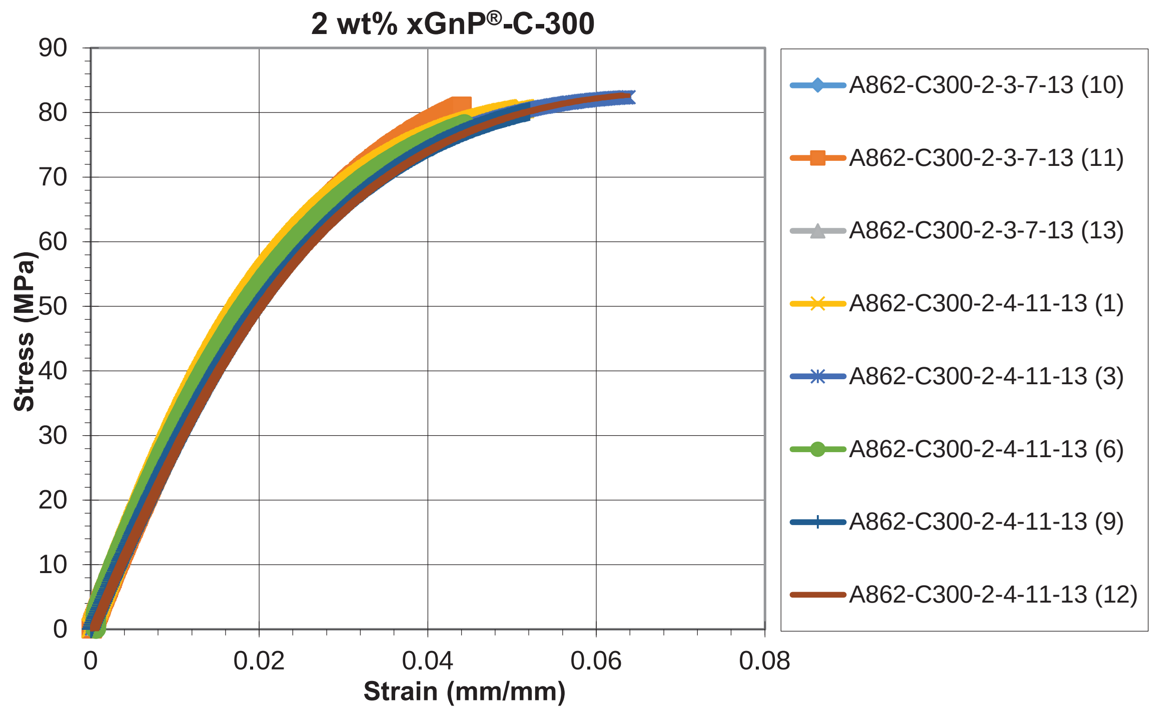


Figure A-15: Tensile Results for 2 wt% xGnP®-C-300 in Epoxy

Table A-15: Tensile Results for 2 wt% xGnP®-C-300 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-C300-2-3-7-13	10	80.361	4.327	80.361	4.327	2.804
A862-C300-2-3-7-13	11	80.898	4.399	80.898	4.399	2.816
A862-C300-2-3-7-13	13	79.099	4.461	79.099	4.461	2.836
A862-C300-2-4-11-13	1	80.621	5.148	80.621	5.148	2.975
A862-C300-2-4-11-13	3	82.373	6.342	82.373	6.342	2.860
A862-C300-2-4-11-13	6	78.184	4.435	78.184	4.435	2.990
A862-C300-2-4-11-13	9	80.233	5.236	80.233	5.236	2.871
A862-C300-2-4-11-13	12	82.655	6.296	82.655	6.296	2.870
Average		80.55	5.08	80.55	5.08	2.88
Standard Deviation		1.50	0.84	1.50	0.84	0.07
Count		8	8	8	8	8

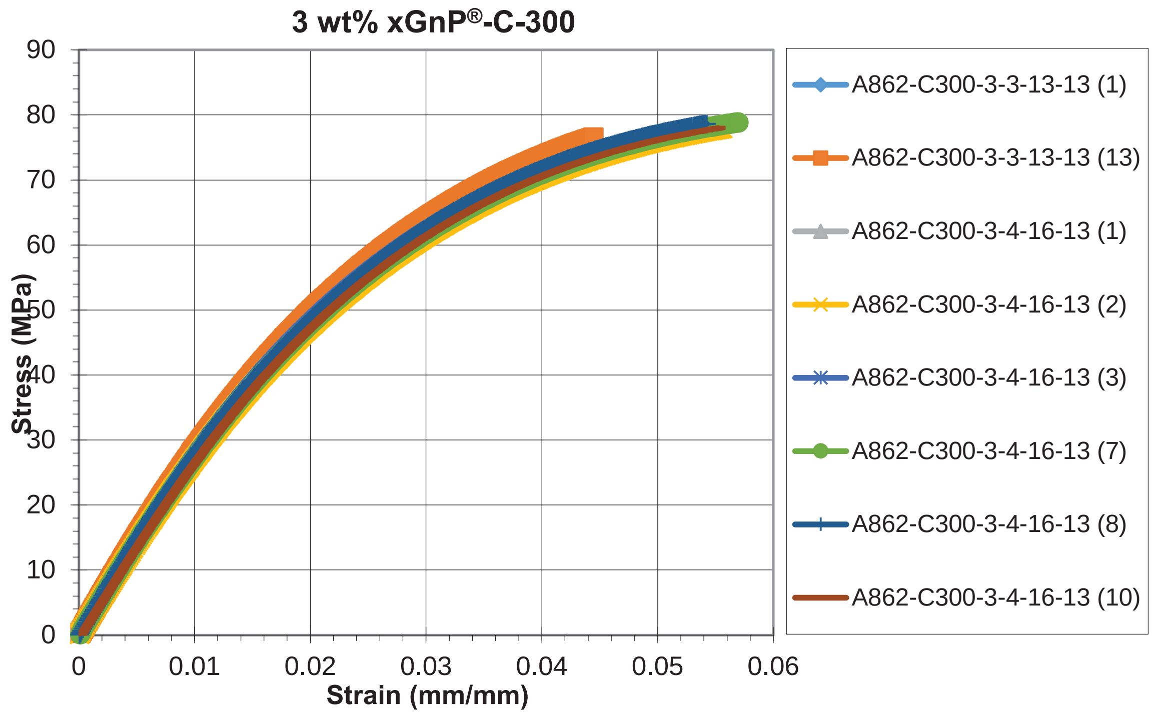


Figure A-16: Tensile Results for 3 wt% xGnP®-C-300 in Epoxy

Table A-16: Tensile Results for 3 wt% xGnP®-C-300 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-C300-3-3-13-13	1	76.509	4.436	76.509	4.436	3.099
A862-C300-3-3-13-13	13	76.535	4.445	76.535	4.445	3.013
A862-C300-3-4-16-13	1	78.457	5.469	78.457	5.469	2.970
A862-C300-3-4-16-13	2	77.887	5.565	77.887	5.565	2.835
A862-C300-3-4-16-13	3	78.253	5.300	78.253	5.300	2.917
A862-C300-3-4-16-13	7	78.844	5.699	78.844	5.699	2.875
A862-C300-3-4-16-13	8	78.739	5.456	78.739	5.456	2.924
A862-C300-3-4-16-13	10	78.321	5.642	78.321	5.642	2.820
Average		**77.94**	**5.25**	**77.94**	**5.25**	**2.93**
Standard Deviation		**0.93**	**0.52**	**0.93**	**0.52**	**0.09**
Number		**8**	**8**	**8**	**8**	**8**

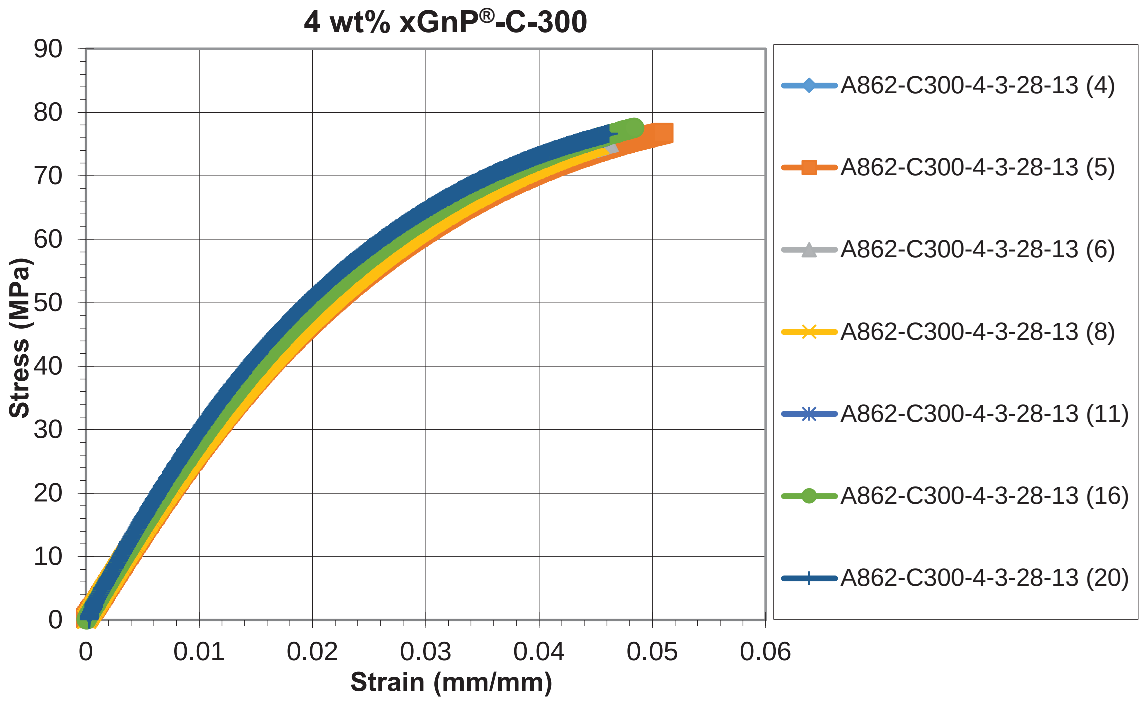


Figure A-17: Tensile Results for 4 wt% xGnP®-C-300 in Epoxy

Table A-17: Tensile Results for 4 wt% xGnP®-C-300 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-C300-4-3-28-13	4	73.910	4.217	73.910	4.217	2.991
A862-C300-4-3-28-13	5	76.747	5.095	76.747	5.095	2.766
A862-C300-4-3-28-13	6	74.937	4.637	74.937	4.637	3.000
A862-C300-4-3-28-13	8	74.934	4.537	74.934	4.537	2.865
A862-C300-4-3-28-13	11	75.810	4.562	75.810	4.562	3.086
A862-C300-4-3-28-13	16	77.557	4.840	77.557	4.840	2.943
A862-C300-4-3-28-13	20	76.632	4.612	76.632	4.612	3.100
Average		**75.79**	**4.64**	**75.79**	**4.64**	**2.96**
Standard Deviation		**1.27**	**0.27**	**1.27**	**0.27**	**0.12**
Number		**7**	**7**	**7**	**7**	**7**

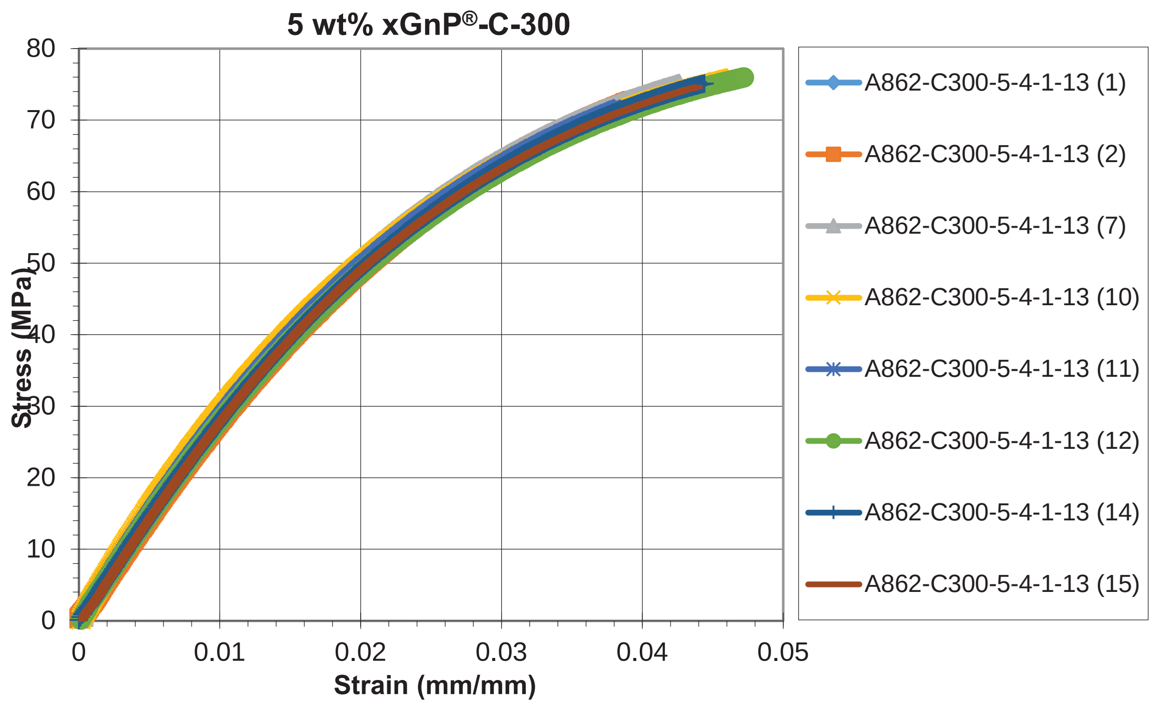


Figure A-18: Tensile Results for 5 wt% xGnP®-C-300 in Epoxy

Table A-18: Tensile Results for 5 wt% xGnP®-C-300 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-C300-5-4-1-13	1	71.597	3.84	71.597	3.84	3.0715
A862-C300-5-4-1-13	2	72.679	3.93	72.679	3.93	2.9547
A862-C300-5-4-1-13	7	75.442	4.26	75.442	4.26	3.1686
A862-C300-5-4-1-13	10	75.918	4.66	75.918	4.66	3.2026
A862-C300-5-4-1-13	11	71.979	3.84	71.979	3.84	3.0867
A862-C300-5-4-1-13	12	75.984	4.72	75.984	4.72	2.9747
A862-C300-5-4-1-13	14	75.193	4.45	75.193	4.45	2.92
A862-C300-5-4-1-13	15	75.098	4.46	75.098	4.46	2.96
Average		**74.24**	**4.27**	**74.24**	**4.27**	**3.04**
Standard Deviation		**1.83**	**0.36**	**1.83**	**0.36**	**0.11**
Count		**8**	**8**	**8**	**8**	**8**

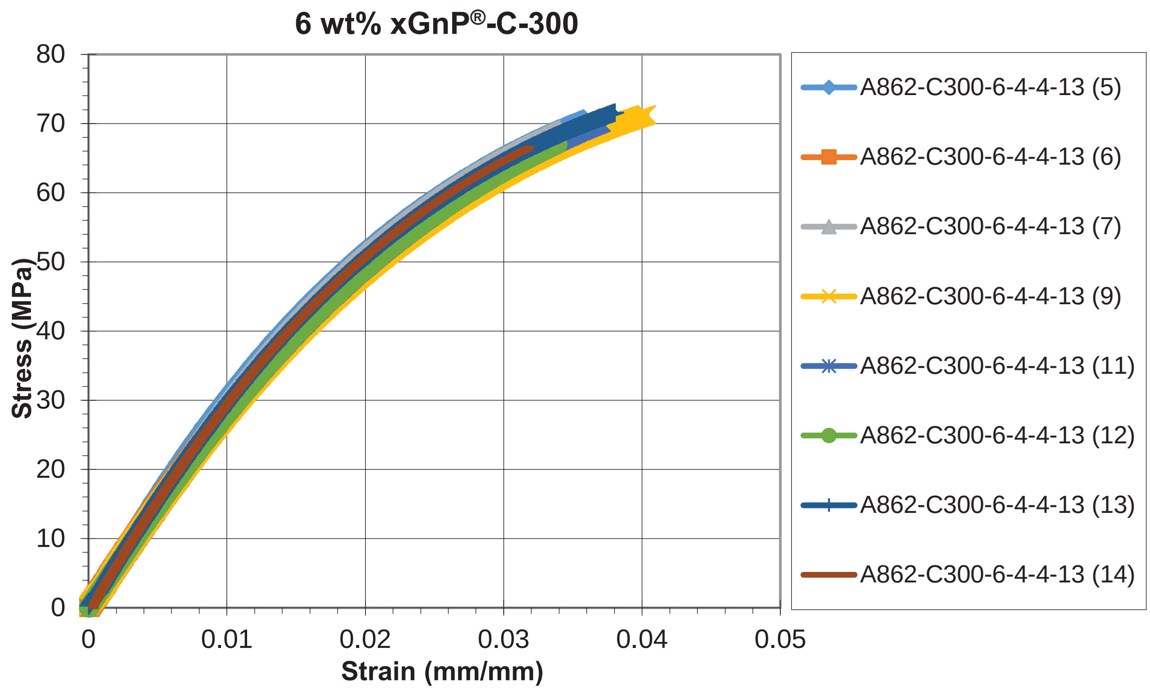


Figure A-19: Tensile Results for 6 wt% xGnP®-C-300 in Epoxy

Table A-19: Tensile Results for 6 wt% xGnP®-C-300 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-C300-6-4-4-13	5	70.973	3.576	70.973	3.576	3.302
A862-C300-6-4-4-13	6	67.176	3.348	67.176	3.348	2.998
A862-C300-6-4-4-13	7	69.391	3.400	69.391	3.400	3.295
A862-C300-6-4-4-13	9	71.270	4.033	71.270	4.033	2.978
A862-C300-6-4-4-13	11	69.814	3.723	69.814	3.723	2.940
A862-C300-6-4-4-13	12	67.038	3.387	67.038	3.387	2.970
A862-C300-6-4-4-13	13	71.574	3.803	71.574	3.803	3.121
A862-C300-6-4-4-13	14	67.020	3.221	67.020	3.221	3.201
Average		**69.28**	**3.56**	**69.28**	**3.56**	**3.10**
Standard Deviation		**1.96**	**0.27**	**1.96**	**0.27**	**0.15**
Count		**8**	**8**	**8**	**8**	**8**

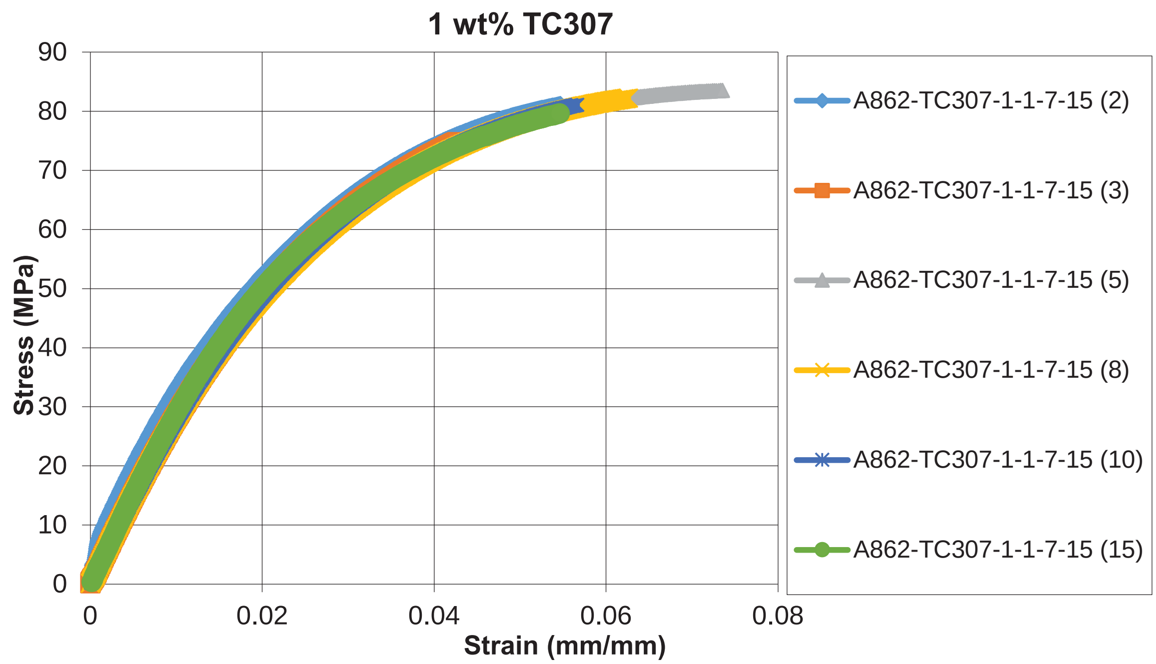


Figure A-20: Tensile Results for 1 wt% TC307 in Epoxy

Table A-20: Tensile Results for 1 wt% TC307 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-TC307-1-1-7-15	2	81.283	5.719	81.283	5.719	2.9528
A862-TC307-1-1-7-15	3	79.200	5.047	79.200	5.047	2.8246
A862-TC307-1-1-7-15	5	83.530	7.399	83.530	7.399	2.7576
A862-TC307-1-1-7-15	8	82.270	6.294	82.270	6.294	2.8231
A862-TC307-1-1-7-15	10	81.170	5.693	81.170	5.693	2.7821
A862-TC307-1-1-7-15	15	79.715	5.492	79.715	5.492	2.9091
Average		81.19	5.94	81.19	5.94	2.84
Standard Deviation		1.60	0.82	1.60	0.82	0.08
Count		6	6	6	6	6

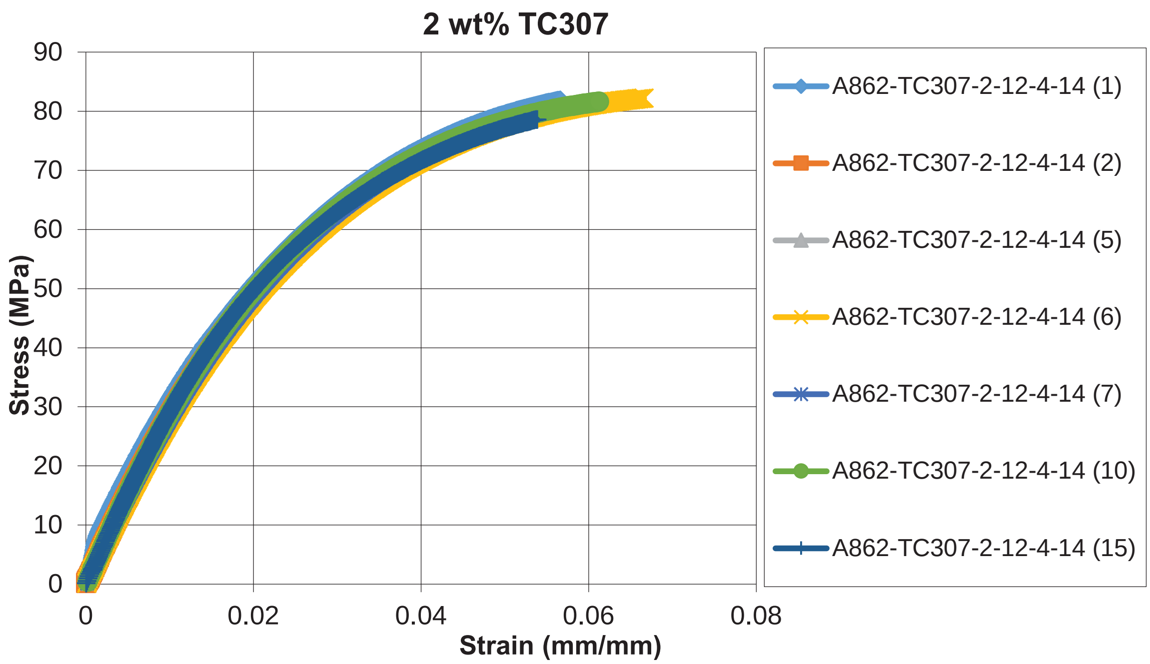


Figure A-21: Tensile Results for 2 wt% TC307 in Epoxy

Table A-21: Tensile Results for 2 wt% TC307 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-TC307-2-12-4-14	1	82.237	5.917	82.237	5.917	2.807
A862-TC307-2-12-4-14	2	77.648	4.894	77.648	4.894	2.886
A862-TC307-2-12-4-14	5	82.008	6.213	82.008	6.213	2.870
A862-TC307-2-12-4-14	6	82.244	6.704	82.244	6.704	2.823
A862-TC307-2-12-4-14	7	81.351	6.085	81.351	6.085	2.826
A862-TC307-2-12-4-14	10	81.706	6.166	81.706	6.166	2.960
A862-TC307-2-12-4-14	1	82.237	5.417	78.734	5.417	2.982
Average		**80.85**	**5.91**	**80.85**	**5.91**	**2.88**
Standard Deviation		**1.87**	**0.59**	**1.87**	**0.59**	**0.07**
Number		**7**	**7**	**7**	**7**	**7**

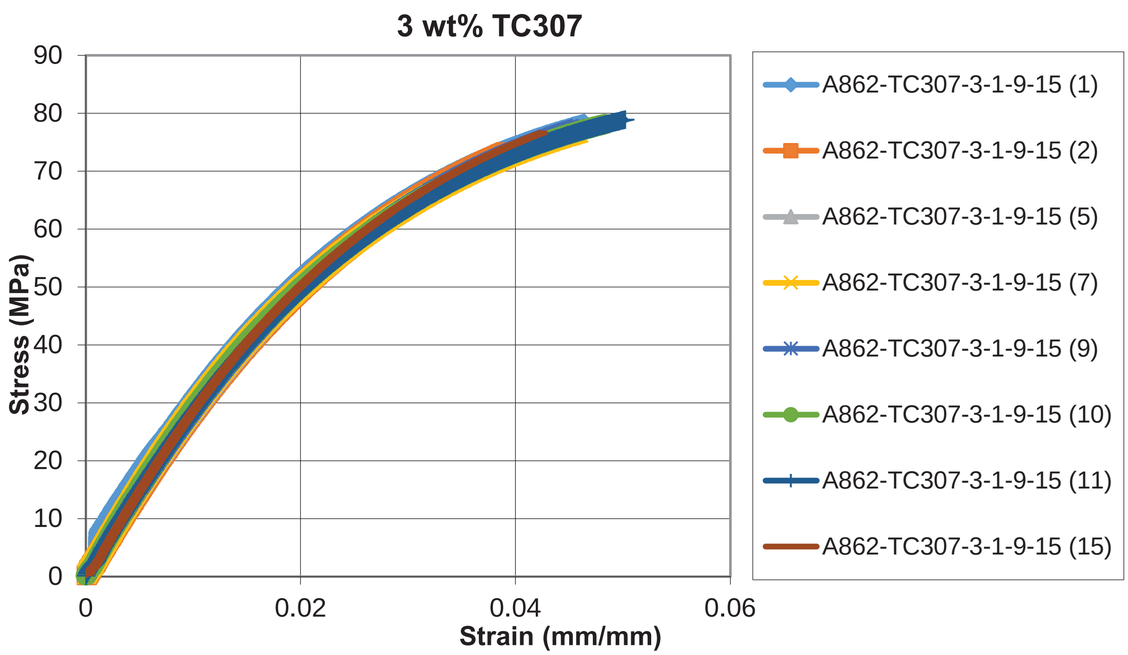


Figure A-22: Tensile Results for 3 wt% TC307 in Epoxy

Table A-22: Tensile Results for 3 wt% TC307 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-TC307-3-1-9-15	1	79.039	4.94	79.039	4.94	2.886
A862-TC307-3-1-9-15	2	78.367	4.68	78.367	4.68	2.903
A862-TC307-3-1-9-15	5	78.095	4.86	78.095	4.86	2.839
A862-TC307-3-1-9-15	7	76.717	4.61	76.717	4.61	2.974
A862-TC307-3-1-9-15	9	77.977	4.60	77.977	4.60	2.884
A862-TC307-3-1-9-15	10	78.259	4.85	78.259	4.85	2.973
A862-TC307-3-1-9-15	11	79.023	5.05	79.023	5.05	2.874
A862-TC307-3-1-9-15	15	77.137	4.30	77.137	4.30	2.969
Average		**78.08**	**4.74**	**78.08**	**4.74**	**2.91**
Standard Deviation		**0.82**	**0.24**	**0.82**	**0.24**	**0.05**
Count		**8**	**8**	**8**	**8**	**8**

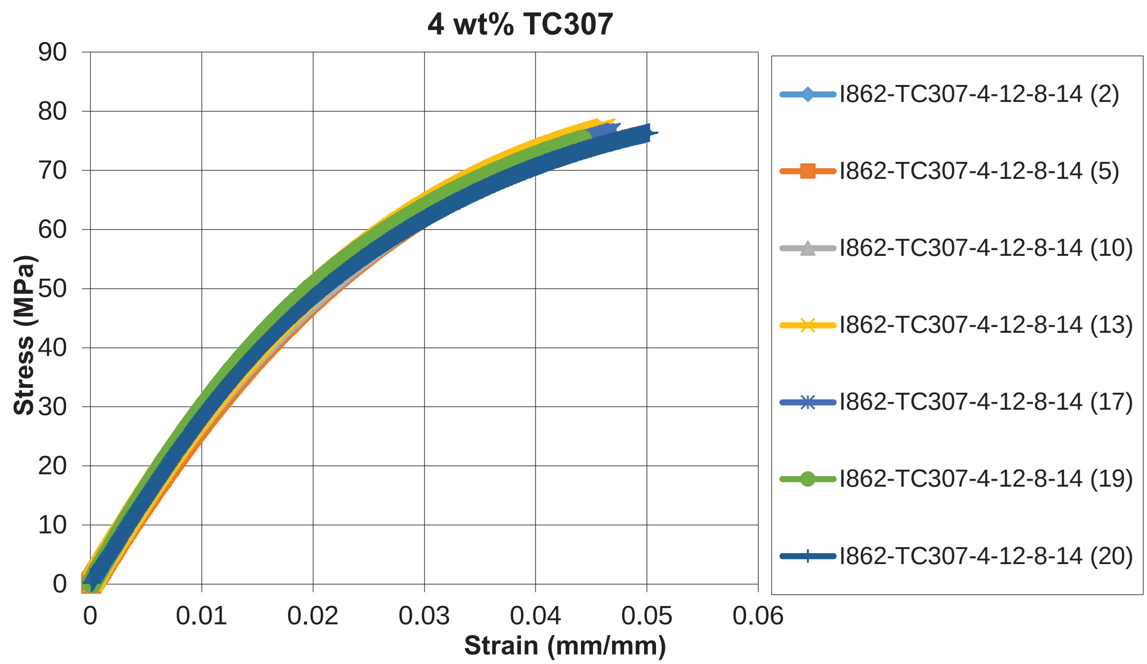


Figure A-23: Tensile Results for 4 wt% TC307 in Epoxy

Table A-23: Tensile Results for 4 wt% TC307 in Epoxy

Sample	No.	Ultimate Tensile Stress (MPa)	Strain at Ultimate Tensile Stress (%)	Tensile Fracture Stress (MPa)	Strain at Tensile Fracture Stress (%)	Tensile Modulus (GPa)
A862-TC307-2-12-4-14	2	76.719	4.508	76.719	4.508	2.914
A862-TC307-2-12-4-14	5	75.668	4.460	75.668	4.460	2.828
A862-TC307-2-12-4-14	10	72.036	4.015	72.036	4.015	2.838
A862-TC307-2-12-4-14	13	77.183	4.630	77.183	4.630	2.964
A862-TC307-2-12-4-14	17	76.997	4.729	76.997	4.729	3.039
A862-TC307-2-12-4-14	19	75.282	4.444	75.282	4.444	3.068
A862-TC307-2-12-4-14	20	76.471	5.046	76.471	5.046	2.879
Average		**75.77**	**4.55**	**75.77**	**4.55**	**2.93**
Standard Deviation		**1.78**	**0.31**	**1.78**	**0.31**	**0.09**
Number		**7**	**7**	**7**	**7**	**7**

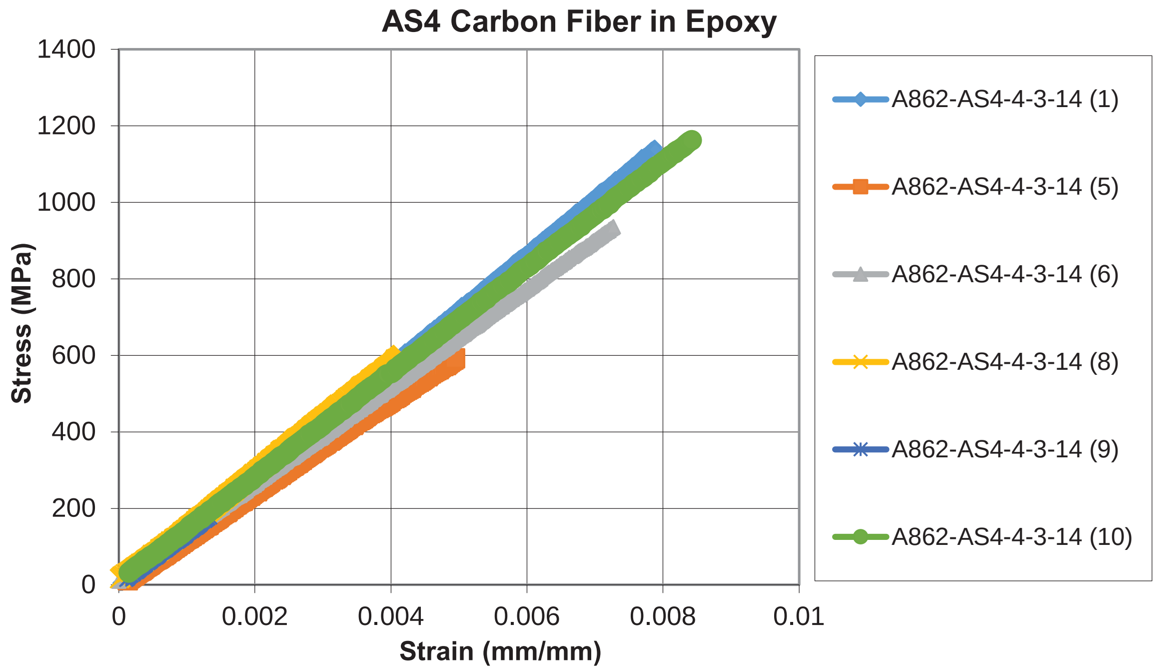


Figure A-24: Tensile Results for AS4 Carbon Fiber in Epoxy

Table A-24: Tensile Results for AS4 Carbon Fiber in Epoxy

Sample	No.	Tensile Modulus (GPa)
A862-AS4-4-3-14	1	144.847
A862-AS4-4-3-14	5	120.89
A862-AS4-4-3-14	6	127.263
A862-AS4-4-3-14	8	143.104
A862-AS4-4-3-14	9	132.278
A862-AS4-4-3-14	10	137.053
Average		**134.24**
Standard Deviation		**9.27**
Number		**6**

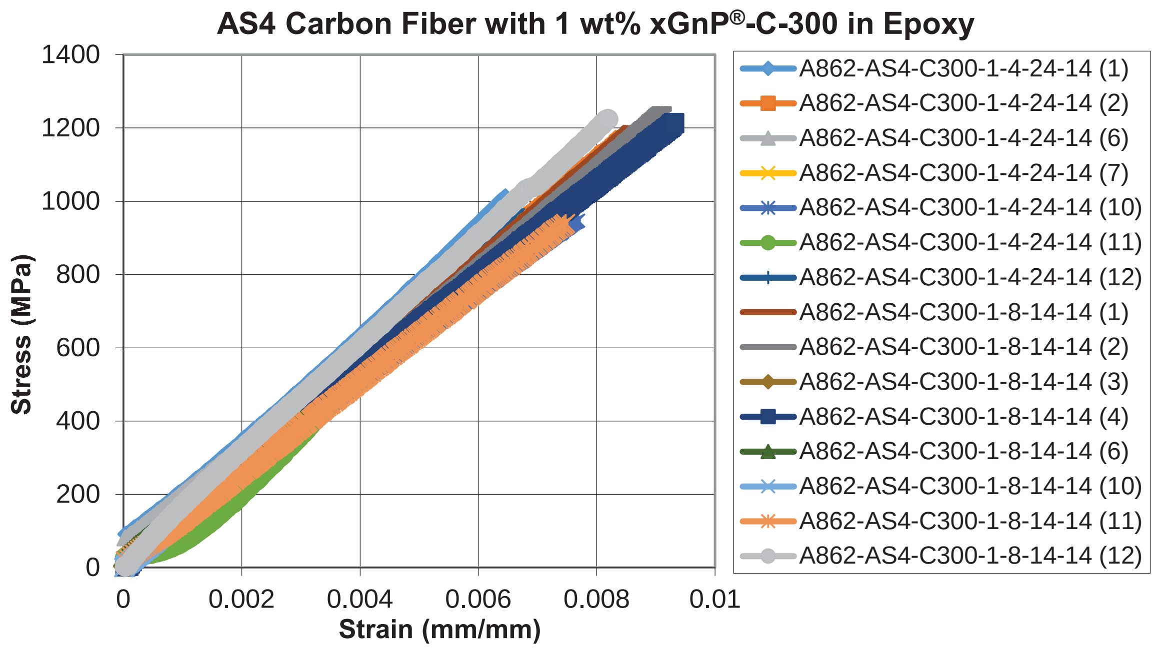


Figure A-25: Tensile Results for 1 wt% xGnP®-C-300 with AS4 Carbon Fiber in Epoxy

Table A-25: Tensile Results for 1 wt% xGnP®-C-300 with AS4 Carbon Fiber in Epoxy

Sample	No.	Tensile Modulus (GPa)
A862-AS4-C300-1-4-24-14	1	144.359
A862-AS4-C300-1-4-24-14	2	136.662
A862-AS4-C300-1-4-24-14	6	123.901
A862-AS4-C300-1-4-24-14	7	135.517
A862-AS4-C300-1-4-24-14	10	123.261
A862-AS4-C300-1-4-24-14	11	153.901
A862-AS4-C300-1-4-24-14	12	147.364
A862-AS4-C300-1-8-14-14	1	140.661
A862-AS4-C300-1-8-14-14	2	136.581
A862-AS4-C300-1-8-14-14	3	133.940
A862-AS4-C300-1-8-14-14	4	129.347
A862-AS4-C300-1-8-14-14	6	136.521
A862-AS4-C300-1-8-14-14	10	146.469
A862-AS4-C300-1-8-14-14	11	125.681
A862-AS4-C300-1-8-14-14	12	147.647
Average		**137.45**
Standard Deviation		**9.33**
Number		**15**

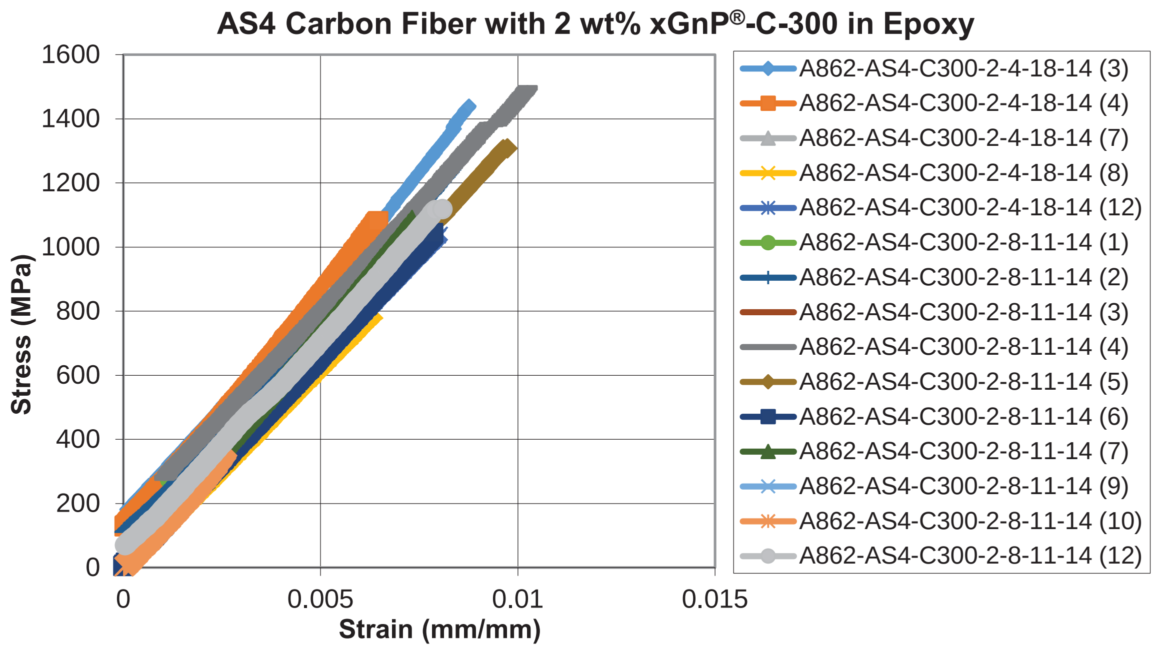


Figure A-26: Tensile Results for 2 wt% xGnP®-C-300 with AS4 Carbon Fiber in Epoxy

Table A-26: Tensile Results for 2 wt% xGnP®-C-300 with AS4 Carbon Fiber in Epoxy

Sample	No.	Tensile Modulus (GPa)
A862-AS4-C300-2-4-18-14	3	144.101
A862-AS4-C300-2-4-18-14	4	148.060
A862-AS4-C300-2-4-18-14	7	142.403
A862-AS4-C300-2-4-18-14	8	125.484
A862-AS4-C300-2-4-18-14	12	128.794
A862-AS4-C300-2-8-11-14	1	137.983
A862-AS4-C300-2-8-11-14	2	138.185
A862-AS4-C300-2-8-11-14	3	133.256
A862-AS4-C300-2-8-11-14	4	132.438
A862-AS4-C300-2-8-11-14	5	132.908
A862-AS4-C300-2-8-11-14	6	132.537
A862-AS4-C300-2-8-11-14	7	145.506
A862-AS4-C300-2-8-11-14	9	142.301
A862-AS4-C300-2-8-11-14	10	138.923
A862-AS4-C300-2-8-11-14	12	132.816
Average		**137.046**
Standard Deviation		**6.53**
Number		**15**

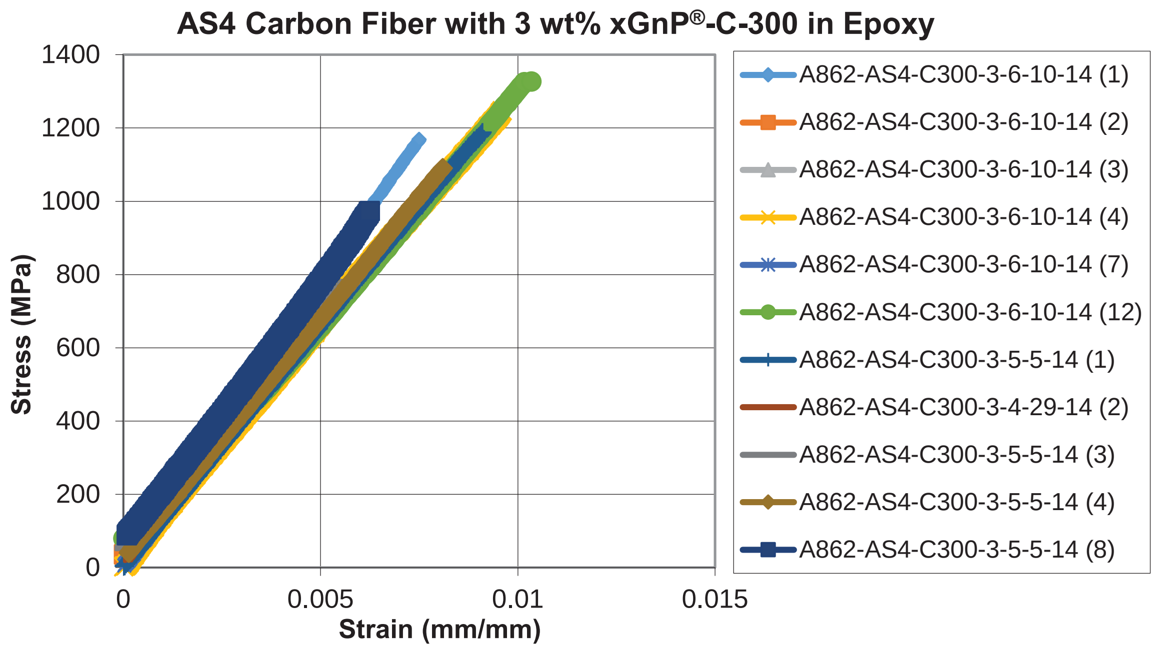


Figure A-27: Tensile Results for 3 wt% xGnP®-C-300 with AS4 Carbon Fiber in Epoxy

Table A-27: Tensile Results for 3 wt% xGnP®-C-300 with AS4 Carbon Fiber in Epoxy

Sample	No.	Tensile Modulus (GPa)
A862-AS4-C300-3-6-10-14	1	149.417
A862-AS4-C300-3-6-10-14	2	148.111
A862-AS4-C300-3-6-10-14	3	131.460
A862-AS4-C300-3-6-10-14	4	129.898
A862-AS4-C300-3-6-10-14	7	142.461
A862-AS4-C300-3-6-10-14	12	122.110
A862-AS4-C300-3-5-5-14	1	131.016
A862-AS4-C300-3-5-5-14	2	150.472
A862-AS4-C300-3-5-5-14	3	128.631
A862-AS4-C300-3-5-5-14	4	131.955
A862-AS4-C300-3-5-5-14	8	142.267
Average		**137.073**
Standard Deviation		**9.75**
Number		**11**

Appdenix B: Nanoindentation Results

***Table B-1**: Nanoindentation Averages Between 500 and 1500 nm Depth for Neat Epoxy*

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.6021	0.0499	0.2579	0.0032
2	3.6090	0.0553	0.2521	0.0022
3	3.5960	0.0567	0.2586	0.0049
4	3.6054	0.0567	0.2556	0.0021
5	3.5958	0.0548	0.2554	0.0023
6	3.6466	0.0557	0.2629	0.0048
7	3.6063	0.0510	0.2533	0.0022
8	3.6040	0.0642	0.2542	0.0027
9	3.5531	0.0362	0.2518	0.0016
10	3.6174	0.0620	0.2570	0.0029
11	3.6495	0.0701	0.2541	0.0035
12	3.6547	0.0567	0.2500	0.0021
13	3.6042	0.0542	0.2544	0.0022
14	3.6113	0.0554	0.2583	0.0036
15	3.6299	0.0465	0.2509	0.0015
16	3.6125	0.0512	0.2542	0.0031
Average	**3.61**	**---**	**0.255**	**---**
Std. Dev.	**0.02**	**---**	**0.003**	**---**
Count	**16**	**16**	**16**	**16**

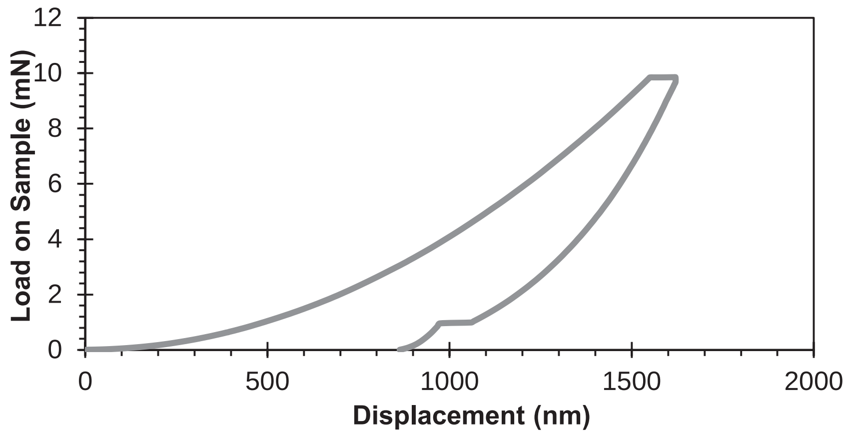


Figure B-1: Load vs. Displacement curve for Neat Epoxy Test 16

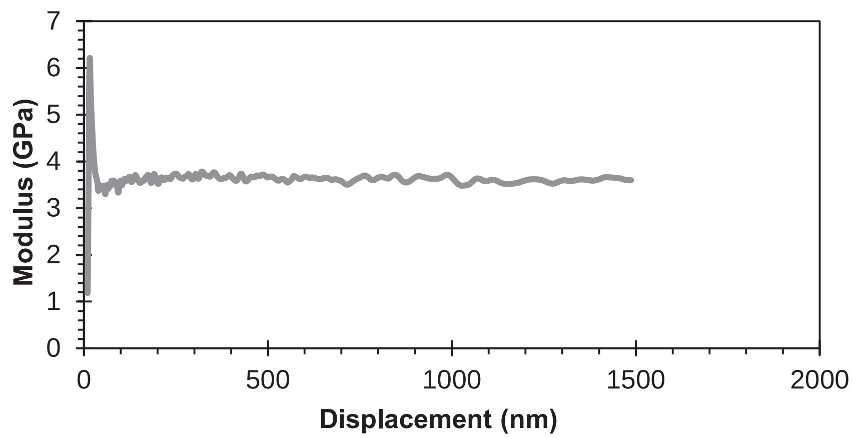


Figure B-2*: *Modulus vs. Displacement curve for Neat Epoxy Test 16

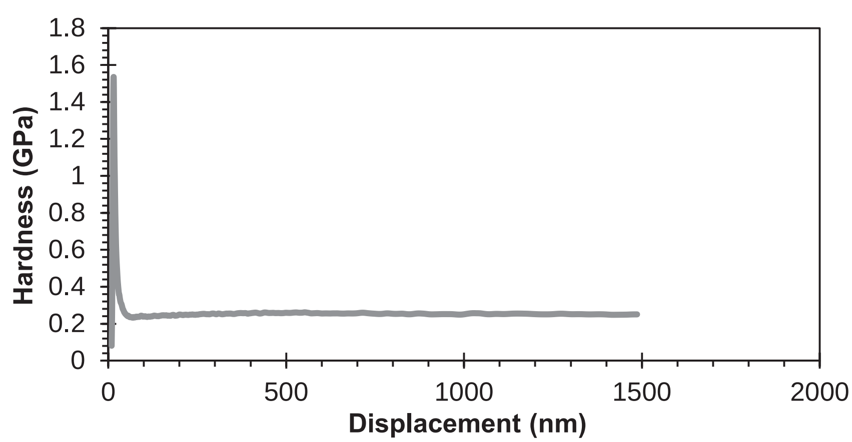


Figure B-3: Hardness vs. Displacement curve for Neat Epoxy Test 16

Table B-2: Nanoindentation Averages Between 500 and 1500 nm Depth for 1 wt% xGnP®-M-15 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
2	3.7515	0.0717	0.2647	0.0086
3	3.6896	0.0649	0.2635	0.0059
7	3.8334	0.0823	0.2598	0.0029
10	3.7979	0.0731	0.2555	0.0027
13	3.7343	0.0735	0.2676	0.0059
15	3.6954	0.0494	0.2578	0.0022
16	3.8412	0.0577	0.2716	0.0023
17	3.7092	0.0588	0.2638	0.0037
20	3.7000	0.0595	0.2621	0.0043
21	3.7377	0.0519	0.2647	0.0037
23	3.8076	0.0752	0.2558	0.0022
26	3.7485	0.0573	0.2661	0.0021
29	3.7551	0.0549	0.2655	0.0029
31	3.8318	0.0456	0.2734	0.0025
34	3.7121	0.0593	0.2557	0.0029
35	3.7437	0.0553	0.2591	0.0051
36	3.7957	0.0678	0.2648	0.0034
37	3.7519	0.0587	0.2690	0.0069
39	3.7250	0.0518	0.2557	0.0039
40	3.7429	0.0449	0.2663	0.0039
42	3.8244	0.0826	0.2772	0.0064
43	3.6823	0.0445	0.2552	0.0025
45	3.7100	0.0969	0.2391	0.0030
46	3.7566	0.0939	0.2573	0.0035
47	3.6548	0.0513	0.2555	0.0022
50	3.7396	0.0566	0.2659	0.0033
51	3.7119	0.0604	0.2597	0.0035
52	3.8312	0.0430	0.2750	0.0072
Mean	**3.75**	**---**	**0.262**	**---**
Std Dev	**0.05**	**---**	**0.008**	**---**
Count	**28**	**28**	**28**	**28**

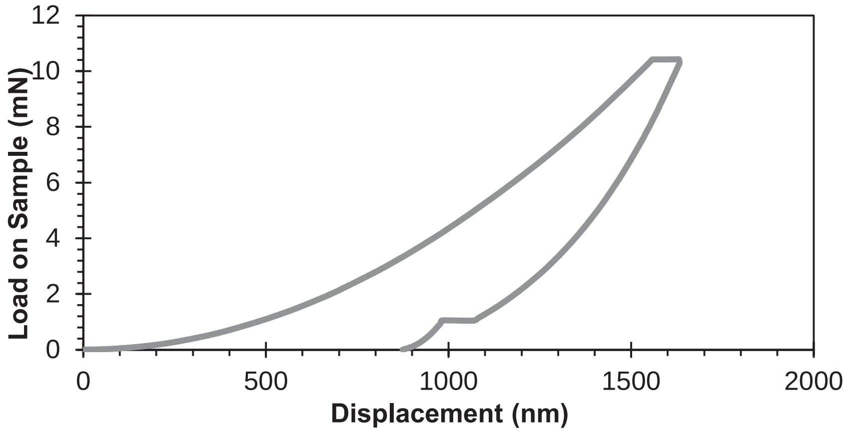


Figure B-4: Load vs. Displacement curve for 1 wt% xGnP®-M-15 in Epoxy Test 16

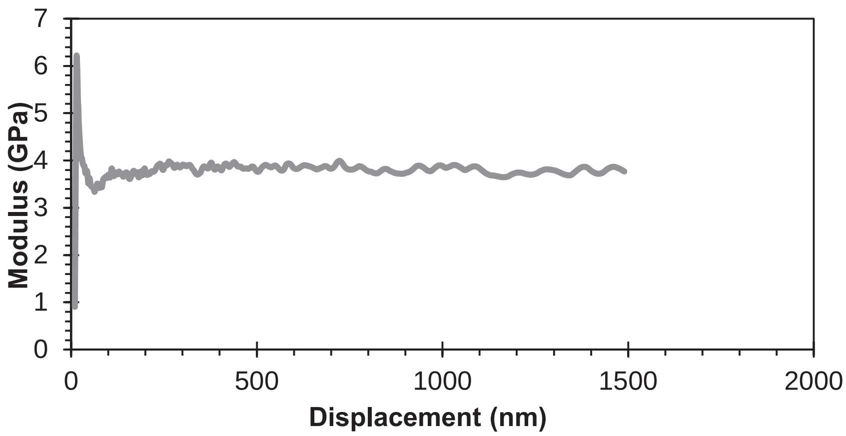


Figure B-5: Modulus vs. Displacement curve for 1 wt% xGnP®-M-15 in Epoxy Test 16

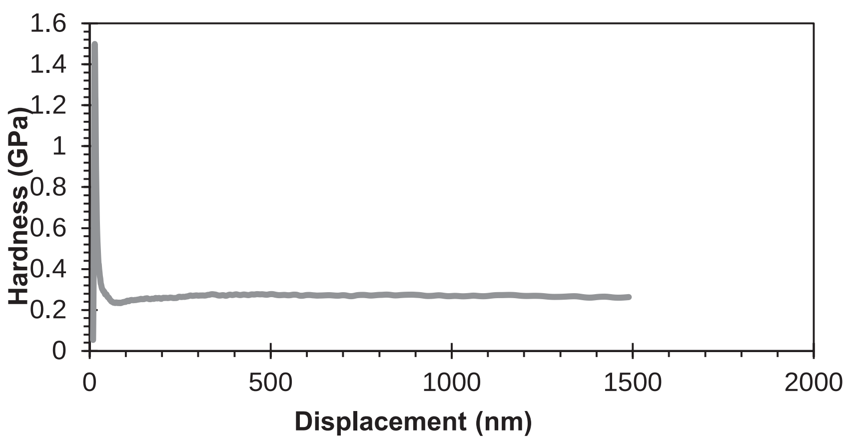


Figure B-6: Hardness vs. Displacement curve 1 wt% xGnP®-M-15 in Epoxy Test 16

Table B-3: Nanoindentation Averages Between 500 and 1500 nm Depth for 2 wt% xGnP®-M-15 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.7333	0.0405	0.2325	0.0094
5	3.7643	0.0670	0.2571	0.0039
6	3.7372	0.0604	0.2566	0.0053
8	4.0044	0.0620	0.2596	0.0023
9	4.0137	0.0612	0.2786	0.0046
13	3.8790	0.0871	0.2652	0.0047
23	3.9598	0.0655	0.2574	0.0016
25	3.9367	0.1068	0.2525	0.0075
29	3.8653	0.0641	0.2313	0.0076
33	3.9276	0.0835	0.2389	0.0062
36	3.8161	0.0680	0.2424	0.0040
37	3.9291	0.0597	0.2564	0.0023
38	3.9886	0.0603	0.2645	0.0032
40	4.0002	0.1087	0.2639	0.0060
41	3.9691	0.0789	0.2608	0.0053
42	3.9908	0.0630	0.2675	0.0036
43	3.9642	0.0689	0.2574	0.0069
44	3.7499	0.1185	0.2166	0.0134
46	3.9399	0.0767	0.2438	0.0108
47	3.9336	0.0594	0.2449	0.0070
48	3.7338	0.1108	0.2280	0.0137
49	3.9682	0.0601	0.2547	0.0040
50	3.9191	0.0875	0.2384	0.0137
51	3.8861	0.0606	0.2614	0.0040
Mean	**3.90**	**---**	**0.251**	**---**
Std Dev	**0.09**	**---**	**0.015**	**---**
Count	**24**	**24**	**24**	**24**

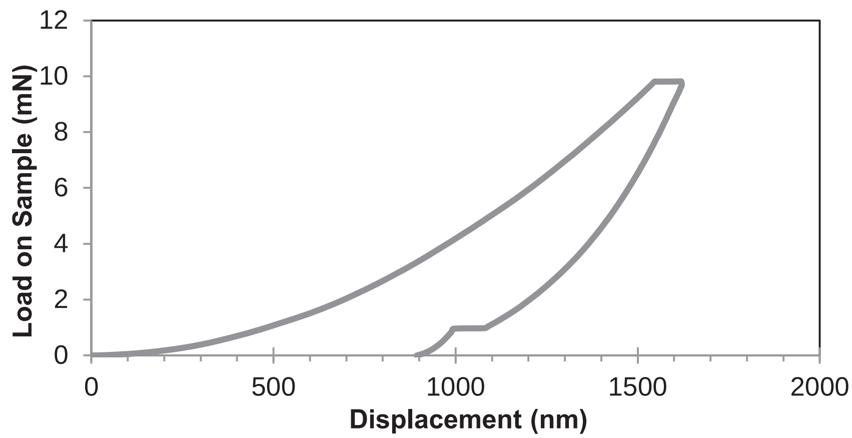


Figure B-7: Load vs. Displacement curve for 2 wt% xGnP®-M-15 in Epoxy Test 6

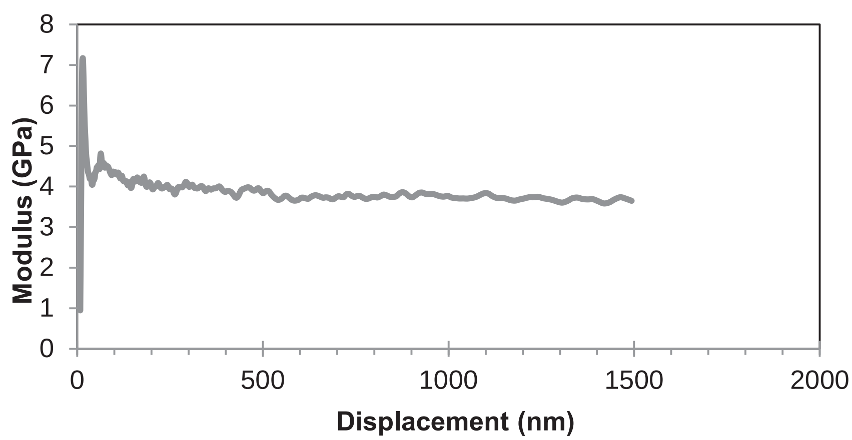


Figure B-8: Modulus vs. Displacement curve for 2 wt% xGnP®-M-15 in Epoxy Test 6

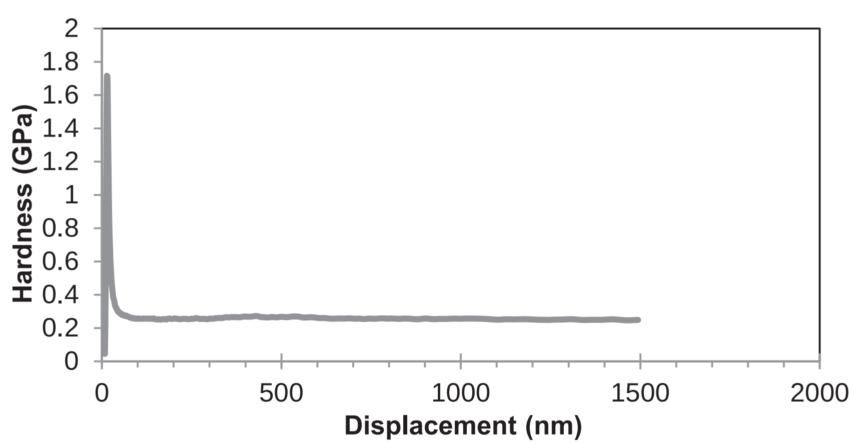


Figure B-9: Hardness vs. Displacement curve 2 wt% xGnP®-M-15 in Epoxy Test 6

Table B-4: Nanoindentation Averages Between 500 and 1500 nm Depth for 3 wt% xGnP®-M-15 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
4	4.1321	0.0688	0.2336	0.0029
8	3.9474	0.0911	0.2291	0.0022
11	4.1595	0.0769	0.2745	0.0081
12	4.0522	0.0940	0.2670	0.0080
15	4.1636	0.0709	0.2486	0.0075
18	3.9821	0.0664	0.2656	0.0075
19	4.1566	0.0650	0.2747	0.0122
22	4.1279	0.0518	0.2911	0.0112
24	4.1511	0.0845	0.2595	0.0093
25	3.9612	0.0514	0.2769	0.0060
26	3.9516	0.1239	0.2503	0.0037
28	3.9229	0.0629	0.2594	0.0069
30	4.0410	0.0670	0.2525	0.0035
33	4.0726	0.0790	0.2688	0.0082
34	3.9586	0.0863	0.2543	0.0032
38	3.9646	0.0674	0.2690	0.0041
39	3.9696	0.0511	0.2705	0.0073
41	4.0447	0.0437	0.2665	0.0018
42	3.9581	0.0745	0.2510	0.0043
43	4.0768	0.0826	0.2604	0.0108
44	4.0330	0.0666	0.2761	0.0102
47	3.9367	0.0583	0.2719	0.0048
48	3.9161	0.0561	0.2603	0.0062
51	3.9962	0.0646	0.2699	0.0084
Mean	**4.03**	**---**	**0.263**	**---**
Std Dev	**0.08**	**---**	**0.014**	**---**
Count	**24**	**24**	**24**	**24**

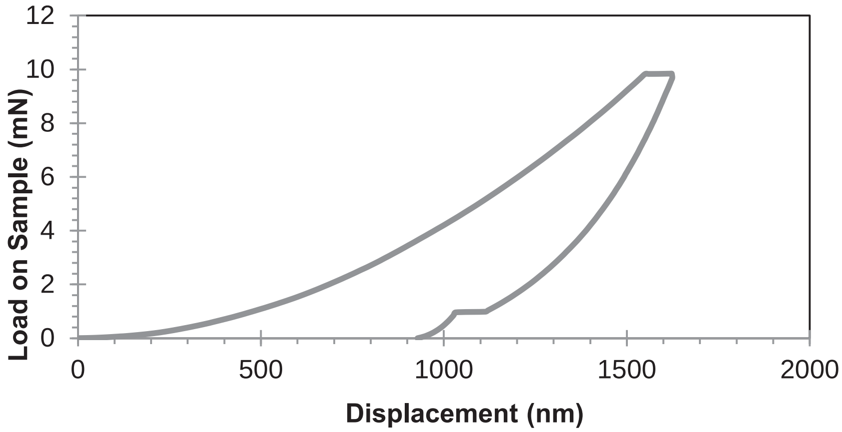


Figure B-10: Load vs. Displacement curve for 3 wt% xGnP®-M-15 in Epoxy Test 15

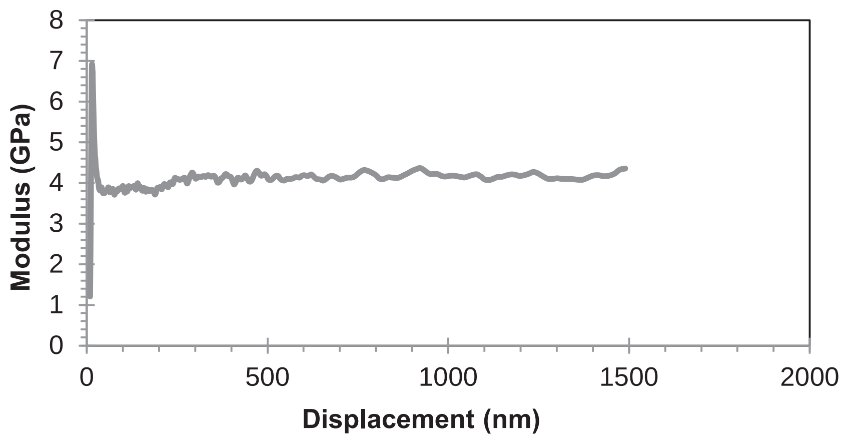


Figure B-11: Modulus vs. Displacement curve for 3 wt% xGnP®-M-15 in Epoxy Test 15

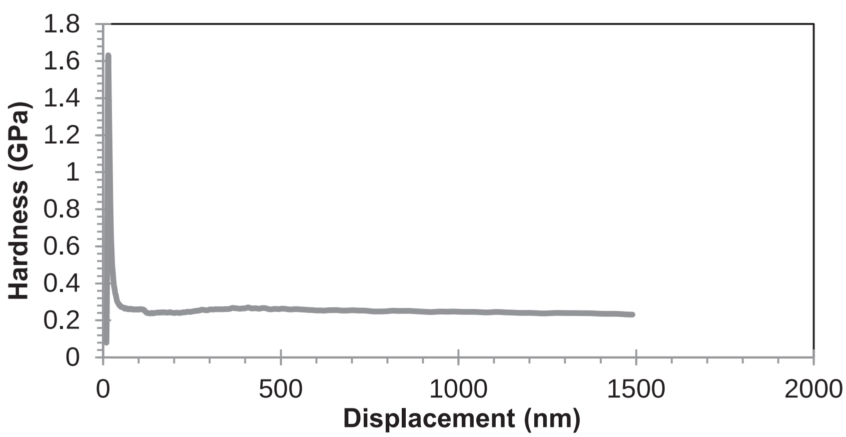


Figure B-12: Hardness vs. Displacement curve 3 wt% xGnP®-M-15 in Epoxy Test 15

Table B-5: Nanoindentation Averages Between 500 and 1500 nm Depth for 4 wt% xGnP®-M-15 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
2	4.4609	0.0485	0.2090	0.0027
3	4.1866	0.0497	0.2347	0.0012
7	3.9969	0.0785	0.2444	0.0030
10	4.4360	0.0676	0.2290	0.0065
12	4.5263	0.1226	0.2399	0.0041
13	3.9860	0.0650	0.2647	0.0052
16	4.2842	0.1022	0.2414	0.0035
20	4.1050	0.0779	0.2161	0.0146
23	4.0215	0.0574	0.2565	0.0071
29	3.9922	0.0457	0.2516	0.0066
31	4.1114	0.0543	0.2469	0.0094
33	3.9774	0.0474	0.2709	0.0052
35	4.4175	0.0723	0.2402	0.0087
42	3.9818	0.0656	0.2641	0.0069
43	3.9863	0.0627	0.2661	0.0040
46	3.9764	0.0641	0.2699	0.0057
49	4.0506	0.0489	0.2590	0.0038
50	4.4769	0.1315	0.2509	0.0019
51	3.9995	0.0623	0.2607	0.0039
56	4.0076	0.0592	0.2714	0.0016
59	4.5152	0.0822	0.2595	0.0033
60	4.5241	0.1659	0.2599	0.0030
62	4.0560	0.0725	0.2656	0.0059
63	4.1370	0.0830	0.2582	0.0084
66	4.1357	0.0644	0.2680	0.0035
67	4.0085	0.0723	0.2649	0.0093
Mean	**4.17**	**---**	**0.252**	**---**
Std Dev	**0.21**	**---**	**0.017**	**---**
Count	**26**	**26**	**26**	**26**

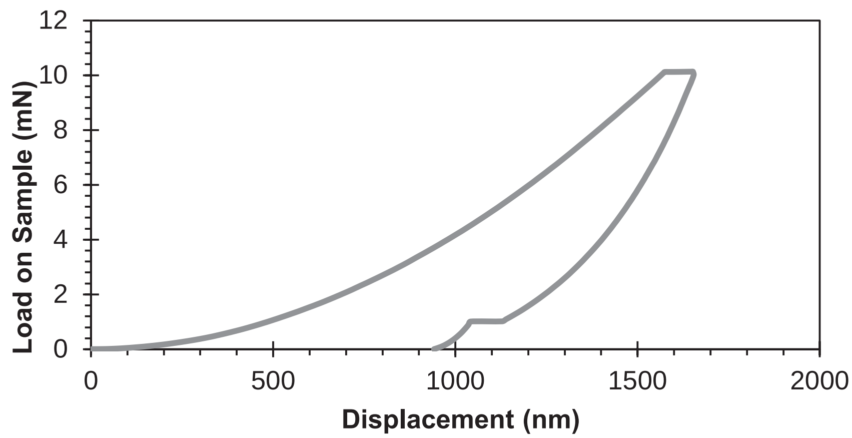


Figure B-13: Load vs. Displacement curve for 4 wt% xGnP®-M-15 in Epoxy Test 29

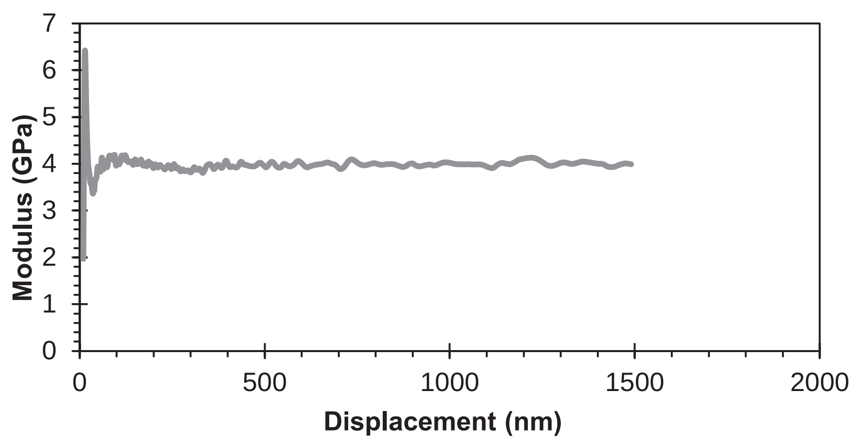


Figure B-14: Modulus vs. Displacement curve for 4 wt% xGnP®-M-15 in Epoxy Test 29

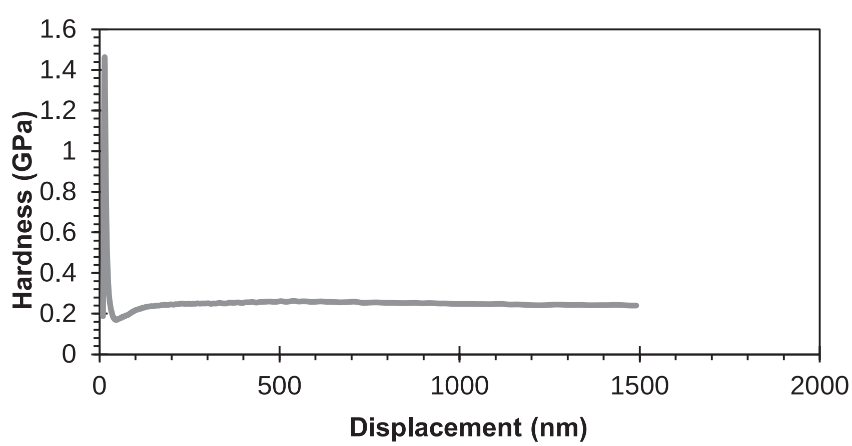


Figure B-15: Hardness vs. Displacement curve 4 wt% xGnP®-M-15 in Epoxy Test 29

Table B-6: Nanoindentation Averages Between 500 and 1500 nm Depth for 5 wt% xGnP®-M-15 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
3	4.5986	0.1462	0.2776	0.0092
4	4.3252	0.0543	0.2570	0.0036
6	4.2933	0.0813	0.2591	0.0078
8	4.3528	0.0435	0.2589	0.0041
9	4.1655	0.1682	0.2443	0.0024
11	4.0861	0.0589	0.2812	0.0054
13	4.0650	0.1111	0.2622	0.0054
16	4.3298	0.0588	0.2514	0.0056
17	4.5582	0.1144	0.2339	0.0064
27	4.3044	0.0751	0.2606	0.0048
28	4.2106	0.0767	0.2565	0.0068
29	4.1950	0.1303	0.2525	0.0034
30	4.3151	0.1071	0.2599	0.0031
32	4.4128	0.1417	0.2540	0.0071
35	4.3599	0.2252	0.2594	0.0099
36	4.0779	0.1449	0.2663	0.0074
41	4.4275	0.0962	0.2409	0.0067
42	4.2676	0.1694	0.2209	0.0200
43	4.4628	0.1103	0.2641	0.0035
44	4.2379	0.0814	0.2537	0.0043
45	4.5075	0.2410	0.2537	0.0034
46	4.2218	0.1227	0.2613	0.0051
50	4.2896	0.1252	0.2677	0.0083
52	4.1670	0.0556	0.2611	0.0044
Mean	**4.30**	**---**	**0.257**	**---**
Std Dev	**0.14**	**---**	**0.013**	**---**
Count	**24**	**24**	**24**	**24**

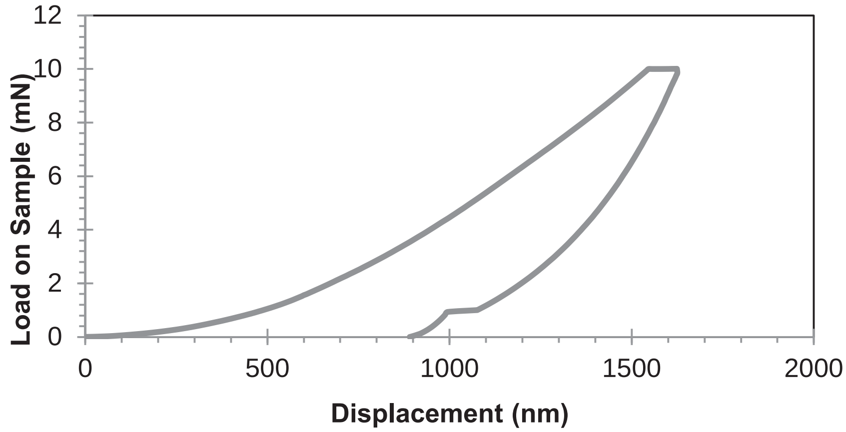


Figure B-16: Load vs. Displacement curve for 5 wt% xGnP®-M-15 in Epoxy Test 13

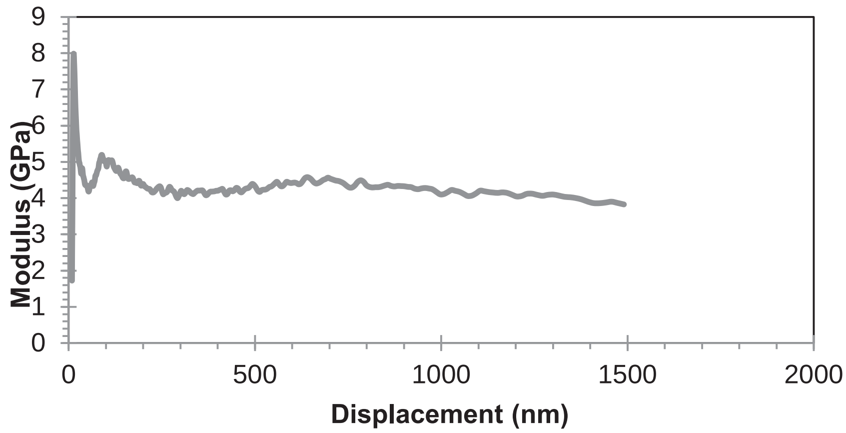


Figure B-17: Modulus vs. Displacement curve for 5 wt% xGnP®-M-15 in Epoxy Test 13

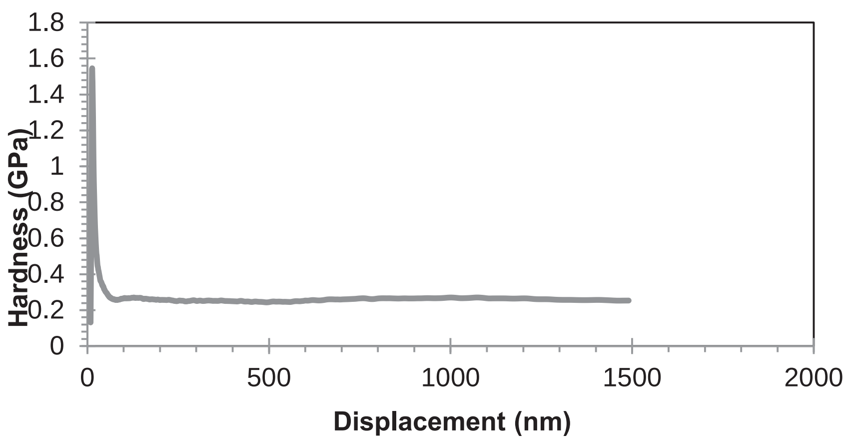


Figure B-18: Hardness vs. Displacement curve 5 wt% xGnP®-M-15 in Epoxy Test 13

Table B-7: Nanoindentation Averages Between 500 and 1500 nm Depth for 6 wt% xGnP®-M-15 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
12	4.6885	0.1412	0.2374	0.0042
13	4.5265	0.0948	0.2693	0.0121
14	4.3266	0.0874	0.2401	0.0045
17	4.2921	0.1027	0.2472	0.0032
20	4.5124	0.0668	0.2701	0.0017
29	4.4587	0.0750	0.2385	0.0036
32	4.4128	0.1560	0.2390	0.0063
33	4.4180	0.2779	0.2565	0.0103
34	4.2569	0.1005	0.2670	0.0049
35	4.4733	0.0758	0.2656	0.0112
37	4.4523	0.1277	0.2623	0.0040
38	4.3739	0.0894	0.2693	0.0065
39	4.2221	0.0850	0.2652	0.0085
42	4.6196	0.1825	0.2589	0.0036
44	4.5049	0.0827	0.2531	0.0018
45	4.6817	0.0847	0.2686	0.0090
46	4.2799	0.0934	0.2714	0.0044
47	4.6403	0.1154	0.2621	0.0027
56	4.5913	0.1084	0.2682	0.0067
60	4.5126	0.1242	0.2687	0.0078
62	4.2329	0.0824	0.2627	0.0040
64	4.6169	0.0895	0.2645	0.0089
66	4.2466	0.1245	0.2722	0.0064
68	4.2044	0.1540	0.2438	0.0027
Mean	**4.44**	**---**	**0.259**	**---**
Std Dev	**0.16**	**---**	**0.012**	**---**
Count	**24**	**24**	**24**	**24**

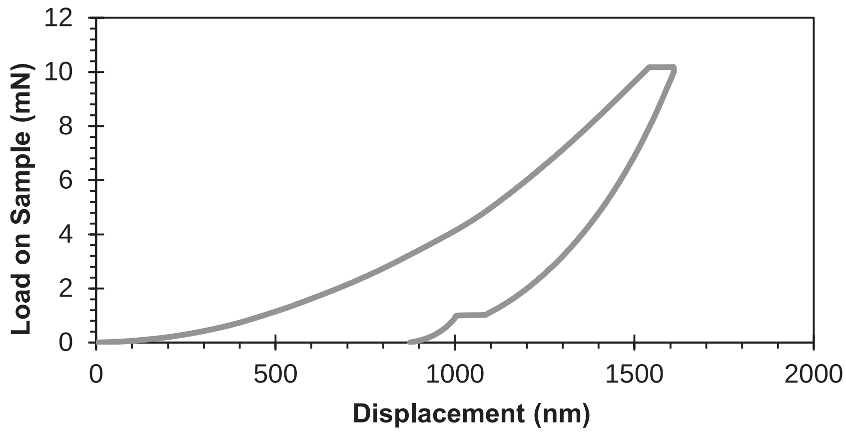


Figure B-19: Load vs. Displacement curve for 6 wt% xGnP®-M-15 in Epoxy Test 32

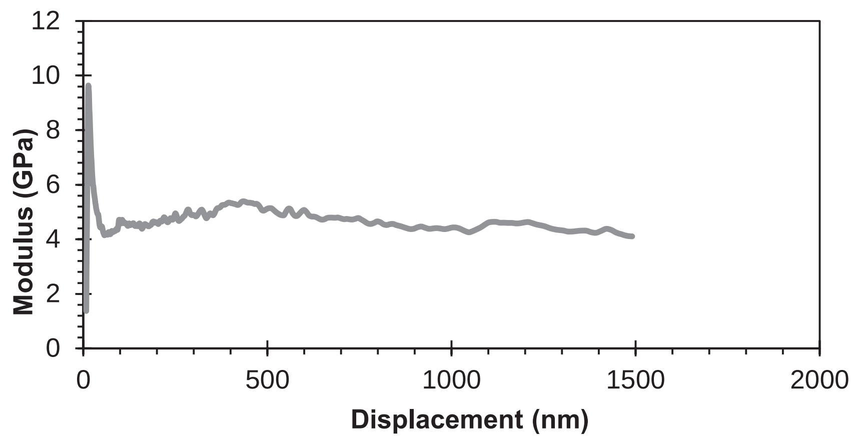


Figure B-20: Modulus vs. Displacement curve for 6 wt% xGnP®-M-15 in Epoxy Test 32

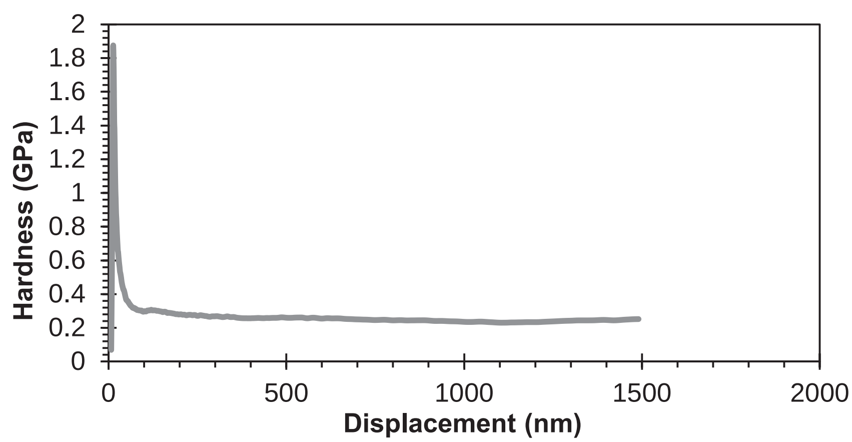


Figure B-21: Hardness vs. Displacement curve 6 wt% xGnP®-M-15 in Epoxy Test 32

Table B-8: Nanoindentation Averages Between 500 and 1500 nm Depth for 1 wt% xGnP®-M-5 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
4	3.7624	0.0704	0.2634	0.0063
7	3.7480	0.0616	0.2638	0.0055
21	3.7002	0.0536	0.2619	0.0056
26	3.7902	0.1522	0.2592	0.0023
29	3.6961	0.0748	0.2638	0.0053
31	3.7501	0.0588	0.2537	0.0081
39	3.7846	0.1071	0.2466	0.0049
41	3.7407	0.0609	0.2593	0.0022
42	3.7722	0.0502	0.2641	0.0059
45	3.7248	0.0524	0.2621	0.0037
46	3.7492	0.0608	0.2628	0.0029
47	3.7500	0.0679	0.2697	0.0057
49	3.7074	0.0571	0.2625	0.0040
51	3.7385	0.0485	0.2634	0.0038
53	3.8458	0.0664	0.2640	0.0052
55	3.7215	0.0533	0.2566	0.0020
56	3.7186	0.0568	0.2601	0.0025
57	3.8032	0.0557	0.2637	0.0045
61	3.7945	0.0516	0.2610	0.0025
62	3.7172	0.0515	0.2584	0.0023
63	3.6965	0.0516	0.2602	0.0026
66	3.7982	0.0504	0.2598	0.0020
69	3.7955	0.0896	0.2533	0.0020
71	3.7151	0.0443	0.2605	0.0024
Mean	**3.75**	**---**	**0.261**	**---**
Std Dev	**0.04**	**---**	**0.005**	**---**
Count	**24**	**24**	**24**	**24**

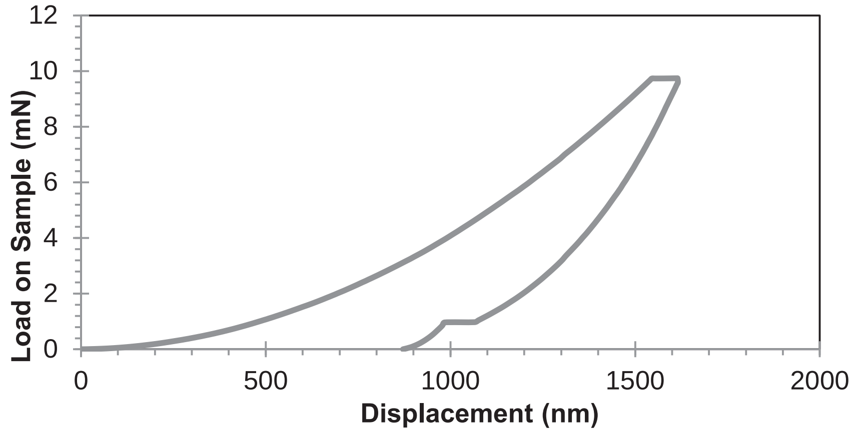


Figure B-22: Load vs. Displacement curve for 1 wt% xGnP®-M-5 in Epoxy Test 31

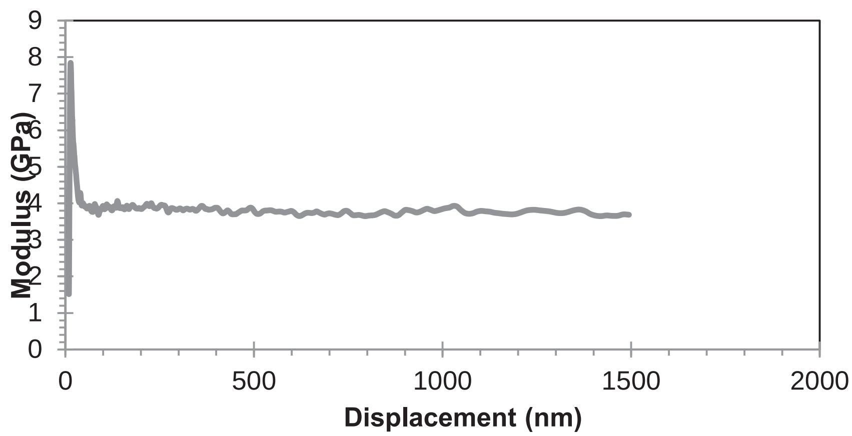


Figure B-23: Modulus vs. Displacement curve for 1 wt% xGnP®-M-5 in Epoxy Test 31

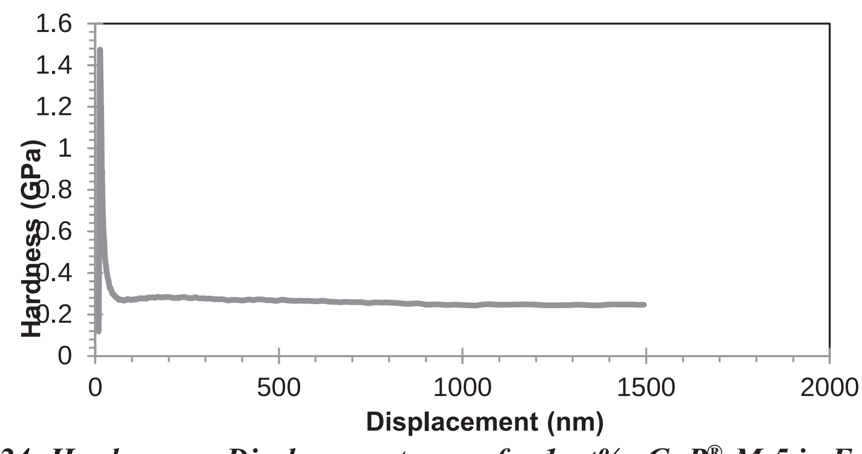


Figure B-24: Hardness vs. Displacement curve for 1 wt% xGnP®-M-5 in Epoxy Test 31

Table B-9: Nanoindentation Averages Between 500 and 1500 nm Depth for 2 wt% xGnP®-M-5 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.8159	0.0509	0.2555	0.0025
2	3.8409	0.0675	0.2544	0.0024
8	3.8699	0.0671	0.2578	0.0037
11	4.0319	0.1107	0.2497	0.0172
13	3.9116	0.0613	0.2603	0.0090
14	3.8206	0.0718	0.2606	0.0042
17	3.8613	0.0714	0.2562	0.0043
18	3.9688	0.1510	0.2568	0.0058
19	3.8269	0.0524	0.2534	0.0082
21	3.9716	0.0715	0.2519	0.0053
22	3.8216	0.0667	0.2598	0.0037
23	3.9145	0.0595	0.2645	0.0050
25	3.8307	0.0746	0.2545	0.0039
26	3.8618	0.0571	0.2687	0.0045
34	3.8745	0.1035	0.2606	0.0066
35	3.8529	0.0773	0.2499	0.0067
43	3.8703	0.0496	0.2589	0.0019
54	3.8639	0.0808	0.2763	0.0072
61	3.8515	0.0752	0.2626	0.0050
62	4.0648	0.1506	0.2601	0.0040
63	3.8775	0.0836	0.2647	0.0033
65	3.9648	0.0985	0.2572	0.0023
68	3.8381	0.0787	0.2620	0.0038
69	3.8555	0.1415	0.2553	0.0065
Mean	**3.89**	**---**	**0.259**	**---**
Std Dev	**0.07**	**---**	**0.006**	**---**
Count	**24**	**24**	**24**	**24**

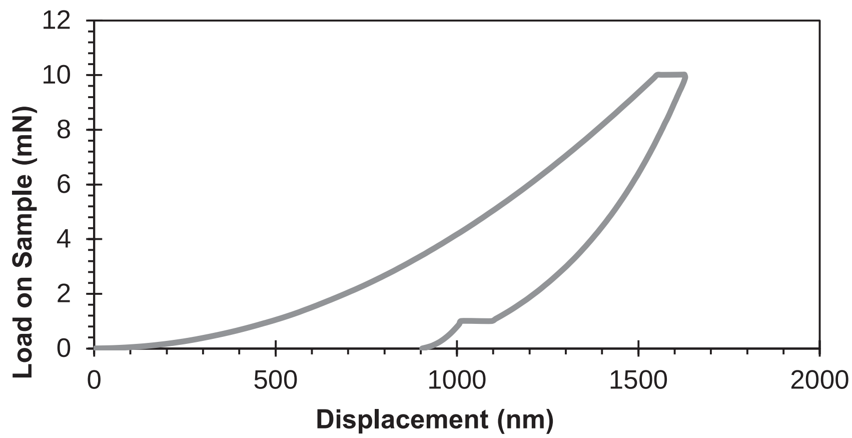


Figure B-25: Load vs. Displacement curve for 2 wt% xGnP®-M-5 in Epoxy Test 26

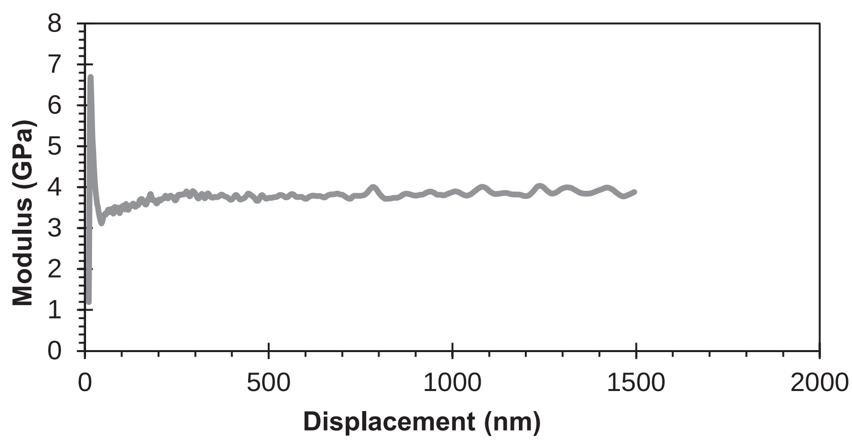


Figure B-26: Modulus vs. Displacement curve for 2 wt% xGnP®-M-5 in Epoxy Test 26

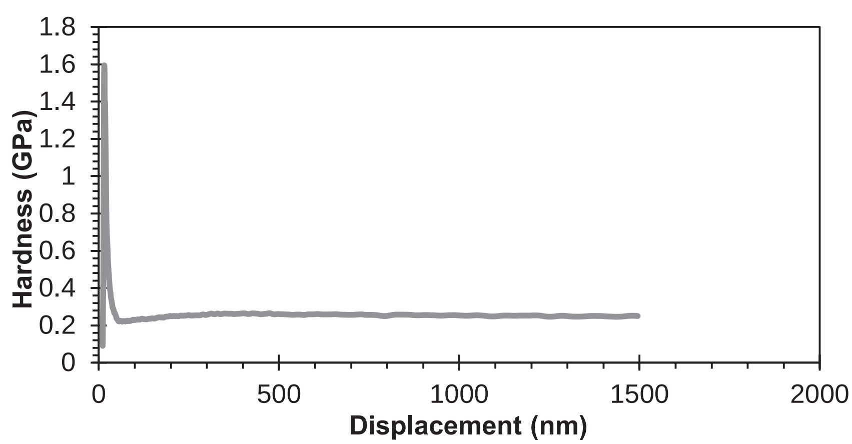


Figure B-27: Hardness vs. Displacement curve for 2 wt% xGnP®-M-5 in Epoxy Test 26

Table B-10: Nanoindentation Averages Between 500 and 1500 nm Depth for 3 wt% xGnP®-M-5 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
2	4.1926	0.1338	0.2391	0.0099
5	3.8800	0.0631	0.2432	0.0051
7	4.1122	0.1230	0.2468	0.0096
14	3.9496	0.1060	0.2543	0.0038
15	3.8495	0.0585	0.2621	0.0069
16	3.8466	0.0683	0.2744	0.0084
20	3.9150	0.1178	0.2493	0.0065
26	3.9559	0.0600	0.2440	0.0113
32	3.8594	0.0752	0.2573	0.0036
33	4.1934	0.0777	0.2553	0.0058
36	3.9692	0.1440	0.2470	0.0081
39	3.8683	0.1047	0.2577	0.0034
40	3.8508	0.0498	0.2646	0.0042
48	4.0644	0.1346	0.2592	0.0065
49	3.8760	0.0718	0.2561	0.0038
51	4.0379	0.1014	0.2381	0.0019
53	3.8615	0.0618	0.2697	0.0051
54	4.0143	0.0508	0.2644	0.0026
58	3.8770	0.0666	0.2667	0.0045
63	3.8833	0.1185	0.2532	0.0030
65	3.8965	0.0770	0.2584	0.0024
67	3.9119	0.0734	0.2574	0.0074
70	3.9828	0.0838	0.2683	0.0083
71	3.8796	0.0614	0.2639	0.0053
Mean	**3.95**	**---**	**0.256**	**---**
Std Dev	**0.11**	**---**	**0.010**	**---**
Count	**24**	**24**	**24**	**24**

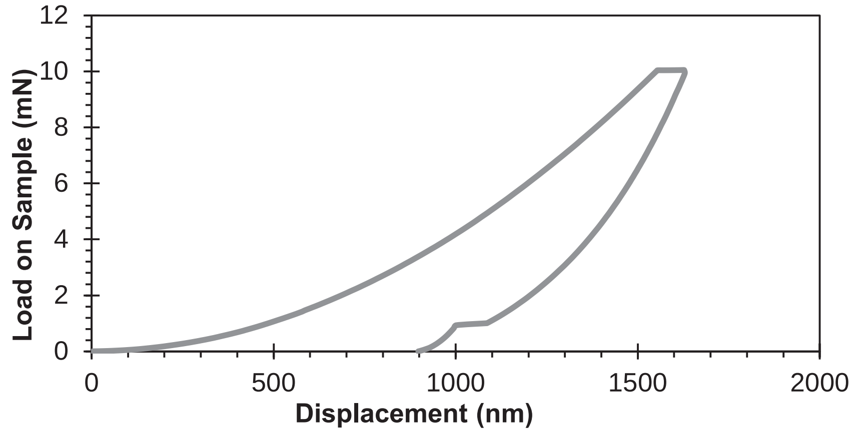


Figure B-28: Load vs. Displacement curve for 3 wt% xGnP®-M-5 in Epoxy Test 32

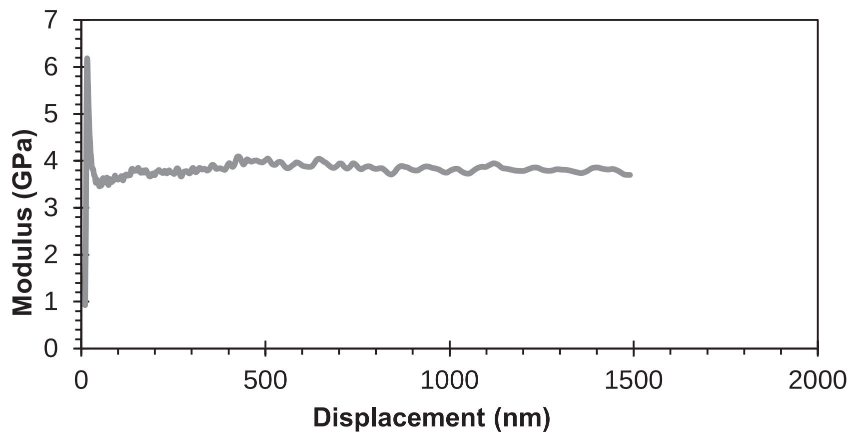


Figure B-29: Modulus vs. Displacement curve for 3 wt% xGnP®-M-5 in Epoxy Test 32

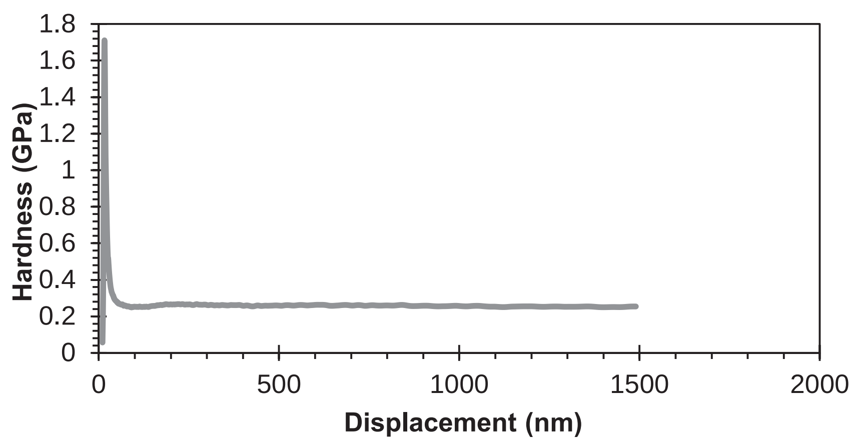


Figure B-30: Hardness vs. Displacement curve for 3 wt% xGnP®-M-5 in Epoxy Test 32

Table B-11: Nanoindentation Averages Between 500 and 1500 nm Depth for 4 wt% xGnP®-M-5 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
3	4.0012	0.0994	0.2517	0.0059
5	3.9648	0.0478	0.2597	0.0049
6	3.9853	0.0580	0.2670	0.0108
10	4.0275	0.0635	0.2582	0.0026
16	3.9398	0.0658	0.2506	0.0069
18	3.9353	0.0782	0.2526	0.0062
20	3.9911	0.0596	0.2527	0.0052
22	3.9760	0.1035	0.2580	0.0094
24	4.1220	0.1554	0.2602	0.0041
25	4.0621	0.0990	0.2469	0.0042
29	4.1062	0.1738	0.2529	0.0097
30	4.3830	0.1028	0.2566	0.0102
35	4.3585	0.2496	0.2588	0.0028
37	3.9667	0.0834	0.2547	0.0031
39	4.0793	0.1253	0.2201	0.0133
42	4.0629	0.0926	0.2678	0.0068
45	4.0658	0.0601	0.2437	0.0042
48	3.9505	0.0854	0.2431	0.0060
50	4.3224	0.0799	0.2437	0.0052
57	3.9428	0.0668	0.2552	0.0043
60	4.3404	0.1160	0.2775	0.0089
61	4.0017	0.1357	0.2400	0.0060
67	4.3149	0.0659	0.2157	0.0029
69	3.9795	0.0765	0.2582	0.0043
Mean	**4.08**	**---**	**0.252**	**---**
Std Dev	**0.15**	**---**	**0.013**	**---**
Count	**24**	**24**	**24**	**24**

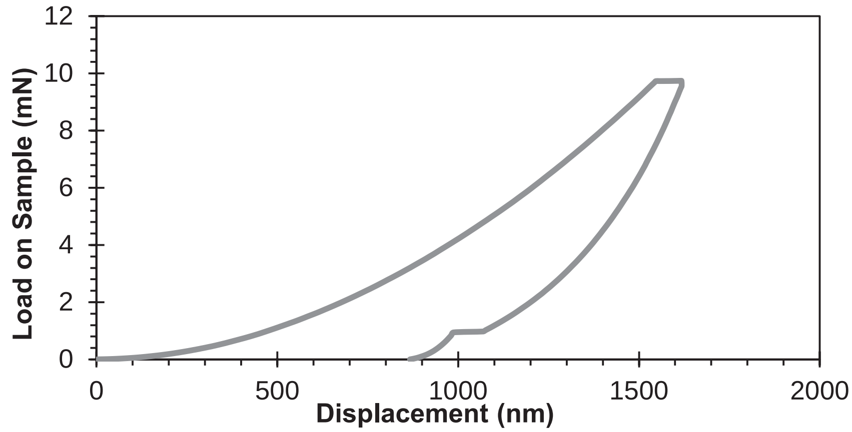


Figure B-31: Load vs. Displacement curve for 4 wt% xGnP®-M-5 in Epoxy Test 22

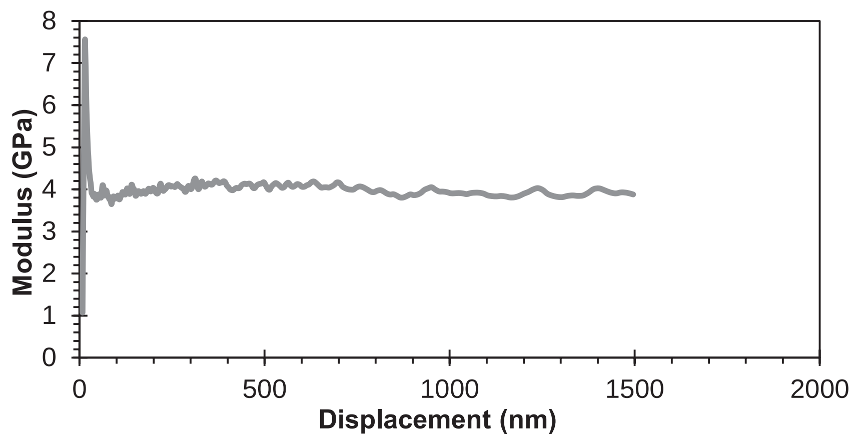


Figure B-32: Modulus vs. Displacement curve for 4 wt% xGnP®-M-5 in Epoxy Test 22

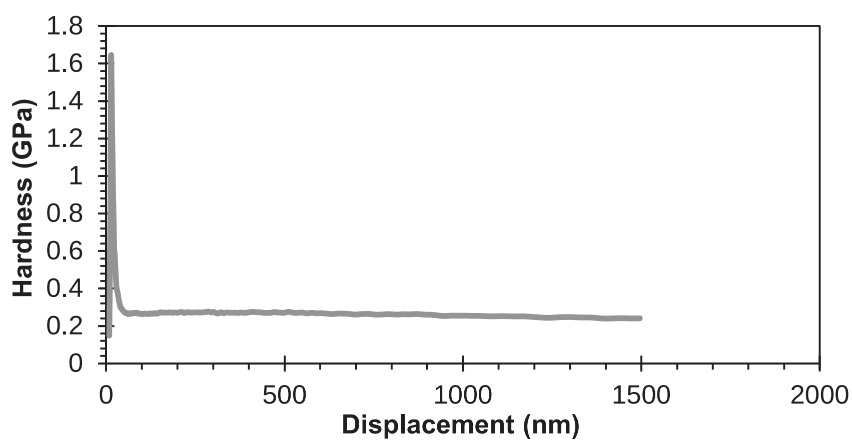


Figure B-33: Hardness vs. Displacement curve for 4 wt% xGnP®-M-5 in Epoxy Test 22

Table B-12: Nanoindentation Averages Between 500 and 1500 nm Depth for 5 wt% xGnP®-M-5 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
2	4.1960	0.0719	0.2821	0.0063
7	4.1514	0.0784	0.2618	0.0031
10	4.2962	0.0729	0.2330	0.0045
16	4.2065	0.0726	0.2605	0.0059
20	4.4672	0.3831	0.2819	0.0110
21	4.5421	0.0756	0.2582	0.0050
28	4.3497	0.0566	0.2600	0.0049
29	4.5050	0.2183	0.2430	0.0018
30	4.1457	0.2597	0.2539	0.0117
31	4.5166	0.3601	0.2603	0.0181
32	4.5070	0.1128	0.2611	0.0169
38	4.2010	0.1273	0.2037	0.0100
41	4.5584	0.1865	0.2574	0.0051
43	4.2835	0.0801	0.2663	0.0103
45	4.1939	0.2538	0.2545	0.0095
51	4.3502	0.0851	0.2402	0.0059
54	4.4981	0.0710	0.2447	0.0103
55	4.1311	0.0776	0.2542	0.0134
56	4.1260	0.0549	0.2538	0.0074
62	4.1066	0.1240	0.2594	0.0023
64	4.1752	0.0788	0.2503	0.0061
68	4.3757	0.0791	0.2611	0.0064
69	4.3977	0.1745	0.2323	0.0083
72	4.2854	0.0839	0.2525	0.0017
Mean	**4.32**	**---**	**0.254**	**---**
Std Dev	**0.15**	**---**	**0.016**	**---**
Count	**24**	**24**	**24**	**24**

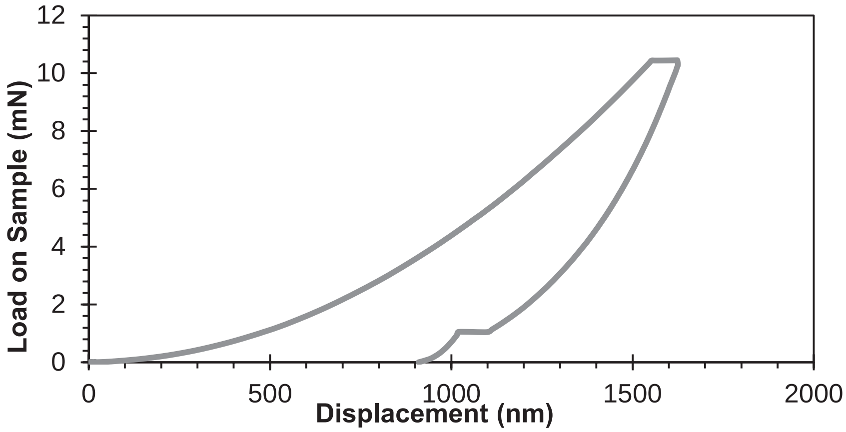


Figure B-34: Load vs. Displacement curve for 5 wt% xGnP®-M-5 in Epoxy Test 28

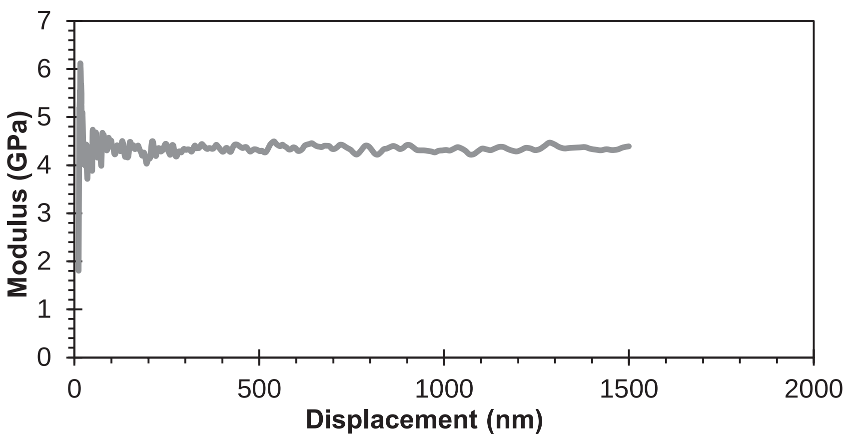


Figure B-35: Modulus vs. Displacement curve for 5 wt% xGnP®-M-5 in Epoxy Test 28

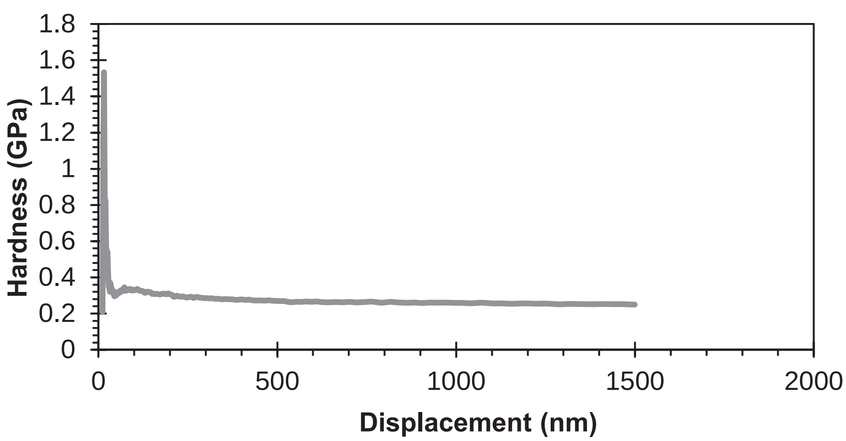


Figure B-36: Hardness vs. Displacement curve for 5 wt% xGnP®-M-5 in Epoxy Test 28

Table B-13: Nanoindentation Averages Between 500 and 1500 nm Depth for 6 wt% xGnP®-M-5 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
4	4.3475	0.0602	0.2930	0.2930
14	4.7146	0.2458	0.2397	0.2397
16	4.4033	0.0641	0.2617	0.2617
17	4.7146	0.2458	0.2397	0.2397
18	4.2756	0.0952	0.2397	0.2397
22	4.2705	0.0808	0.2506	0.2506
26	4.4748	0.0762	0.2459	0.2459
31	4.6819	0.1173	0.2475	0.2475
38	4.3890	0.1356	0.2608	0.0041
40	4.3250	0.1367	0.2475	0.0112
41	4.6418	0.2774	0.2461	0.0058
42	4.4948	0.0872	0.2639	0.0051
44	4.4190	0.0926	0.2552	0.0070
45	4.6027	0.1952	0.2639	0.0131
47	4.4718	0.1258	0.2474	0.0047
48	4.3576	0.1898	0.2651	0.0020
52	4.5228	0.1199	0.2549	0.0054
54	4.3385	0.1346	0.2566	0.0021
62	4.5414	0.1499	0.2347	0.0055
63	4.3321	0.2027	0.2459	0.0037
66	4.3425	0.1581	0.2620	0.0040
67	4.2930	0.0758	0.2632	0.0038
68	4.4437	0.1949	0.2568	0.0030
71	4.2968	0.1065	0.2662	0.0053
Mean	**4.45**	**---**	**0.254**	**---**
Std Dev	**0.14**	**---**	**0.012**	**---**
Count	**24**	**24**	**24**	**24**

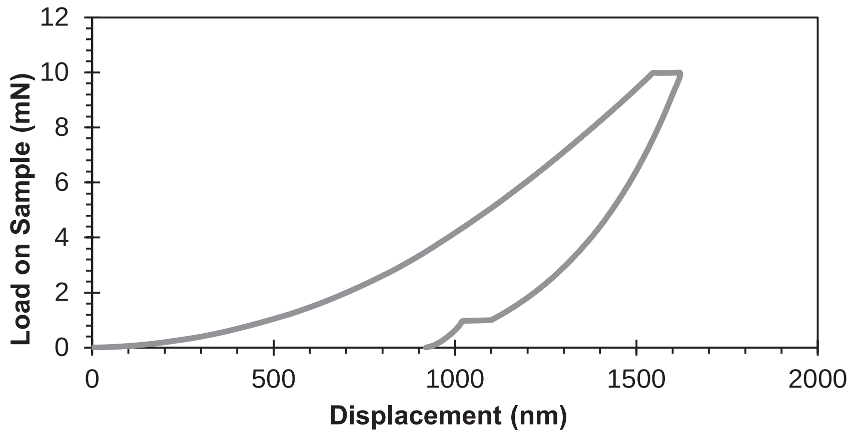


Figure B-37: Load vs. Displacement curve for 6 wt% xGnP®-M-5 in Epoxy Test 18

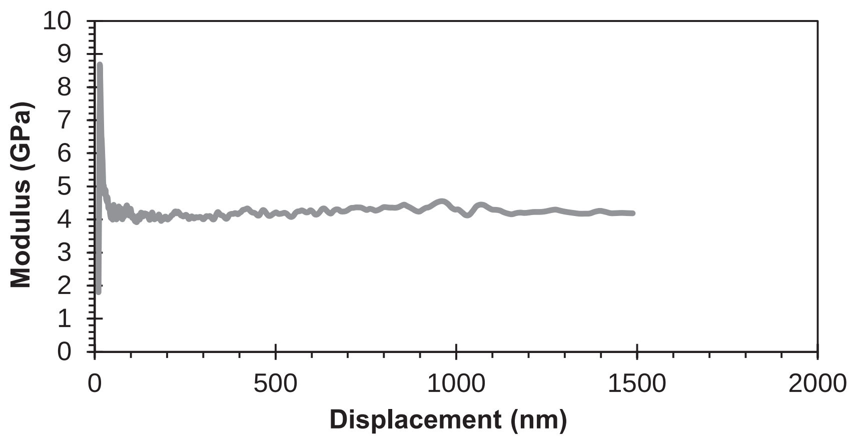


Figure B-38: Modulus vs. Displacement curve for 6 wt% xGnP®-M-5 in Epoxy Test 18

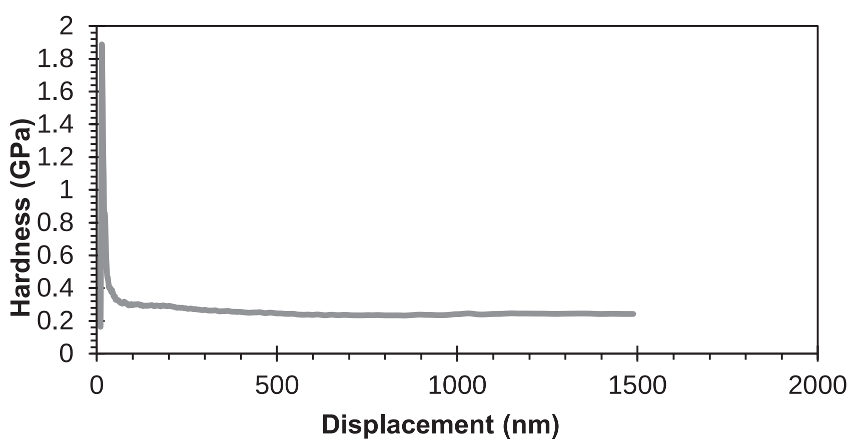


Figure B-39: Hardness vs. Displacement curve for 6 wt% xGnP®-M-5 in Epoxy Test 18

Table B-14: Nanoindentation Averages Between 500 and 1500 nm Depth for 1 wt% xGnP®-C-300 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.5861	0.0441	0.2562	0.0031
2	3.5654	0.0517	0.2557	0.0028
3	3.6534	0.0471	0.2653	0.0053
4	3.6069	0.0556	0.2538	0.0020
5	3.6568	0.0508	0.2648	0.0053
6	3.6506	0.0558	0.2589	0.0027
7	3.6569	0.0536	0.2615	0.0036
8	3.6416	0.0662	0.2637	0.0050
9	3.6312	0.0537	0.2582	0.0022
10	3.6452	0.0626	0.2630	0.0043
11	3.6595	0.0495	0.2617	0.0035
12	3.6393	0.0484	0.2620	0.0050
13	3.6388	0.0520	0.2582	0.0022
14	3.7079	0.0704	0.2650	0.0027
15	3.6834	0.0582	0.2641	0.0046
16	3.6520	0.0565	0.2626	0.0040
17	3.7064	0.1085	0.2667	0.0101
18	3.6237	0.0471	0.2578	0.0023
19	3.5862	0.0435	0.2538	0.0026
20	3.7131	0.0412	0.2636	0.0039
21	3.6062	0.0506	0.2588	0.0029
22	3.6458	0.0634	0.2623	0.0039
23	3.6783	0.0516	0.2625	0.0028
24	3.5750	0.0445	0.2543	0.0017
25	3.6258	0.0427	0.2583	0.0021
26	3.7384	0.0752	0.2693	0.0062
27	3.6040	0.0515	0.2577	0.0025
28	3.6398	0.0457	0.2601	0.0026
29	3.6396	0.0606	0.2608	0.0031
30	3.7010	0.0643	0.2664	0.0042
32	3.6561	0.0580	0.2629	0.0044
33	3.6401	0.0537	0.2615	0.0034
34	3.6620	0.0570	0.2663	0.0050
35	3.6483	0.0567	0.2622	0.0048
36	3.6447	0.0655	0.2587	0.0032
Mean	**3.65**	**---**	**0.261**	**---**
Std Dev	**0.04**	**---**	**0.004**	**---**
Count	**35**	**35**	**35**	**35**

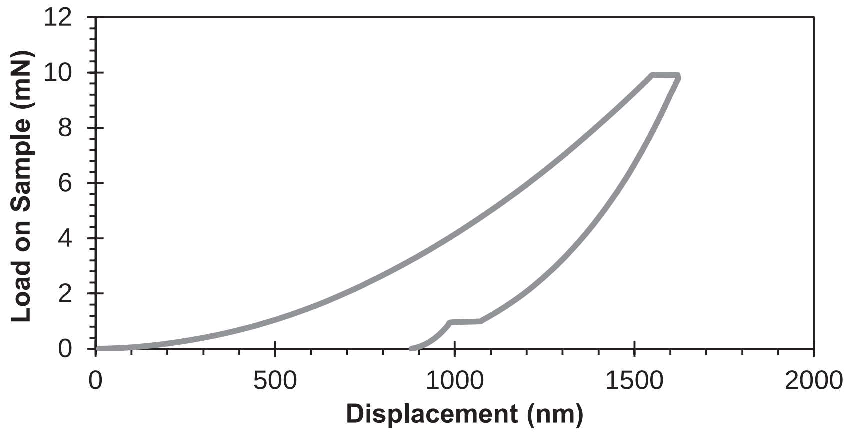


Figure B-40: Load vs. Displacement curve for 1 wt% xGnP®-C-300 in Epoxy Test 36

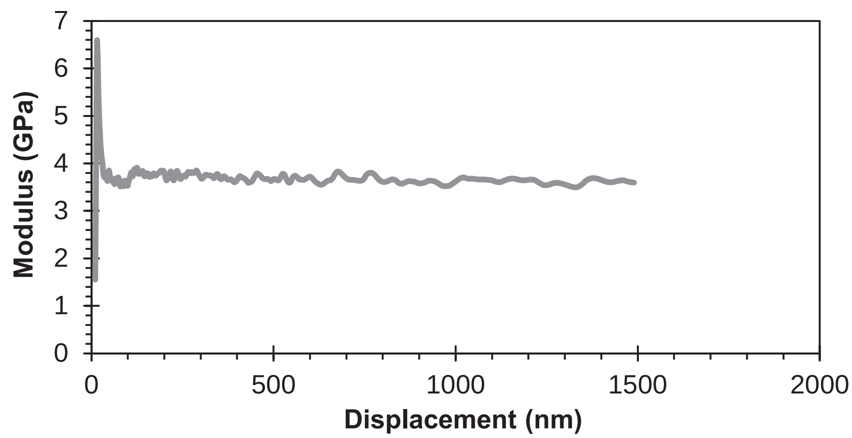


Figure B-41: Modulus vs. Displacement curve for 1 wt% xGnP®-C-300/Epoxy Test 36

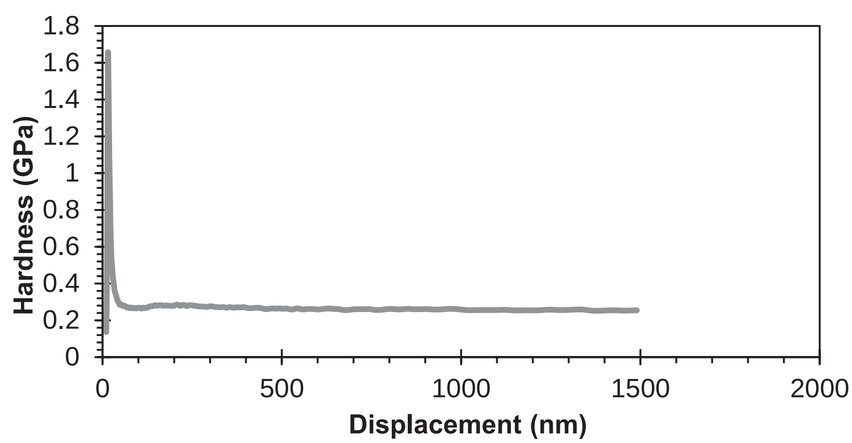


Figure B-42: Hardness vs. Displacement curve for 1 wt% xGnP®-C-300/Epoxy Test 36

Table B-15: Nanoindentation Averages Between 500 and 1500 nm Depth for 2 wt% xGnP®-C-300 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.8225	0.0630	0.2739	0.0044
5	3.8100	0.0564	0.2674	0.0030
16	3.8072	0.0569	0.2721	0.0032
18	3.8049	0.0567	0.2664	0.0024
19	3.8076	0.0491	0.2707	0.0024
23	3.7881	0.0465	0.2680	0.0020
24	3.8156	0.0526	0.2690	0.0020
26	3.8082	0.0619	0.2678	0.0030
29	3.7508	0.0508	0.2645	0.0017
35	3.8038	0.0450	0.2691	0.0026
39	3.8147	0.0711	0.2679	0.0030
42	3.8088	0.0607	0.2700	0.0026
45	3.7681	0.0531	0.2619	0.0022
47	3.8070	0.0548	0.2680	0.0027
49	3.7938	0.0530	0.2653	0.0025
56	3.7697	0.0578	0.2617	0.0024
57	3.7930	0.0698	0.2680	0.0050
62	3.7732	0.0508	0.2650	0.0022
68	3.7050	0.0672	0.2436	0.0070
76	3.7926	0.0580	0.2661	0.0020
80	3.8215	0.0549	0.2650	0.0022
82	3.7832	0.0494	0.2608	0.0038
85	3.7127	0.0661	0.2418	0.0113
88	3.7953	0.0633	0.2636	0.0022
103	3.7524	0.0525	0.2599	0.0024
Mean	**3.79**	**---**	**0.265**	**---**
Std Dev	**0.03**	**---**	**0.007**	**---**
Count	**25**	**25**	**25**	**25**

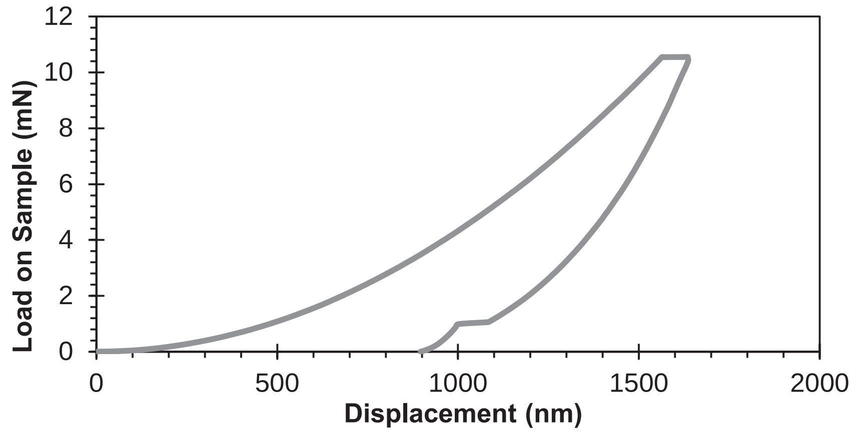


Figure B-43: Load vs. Displacement curve for 2 wt% xGnP®-C-300 in Epoxy Test 35

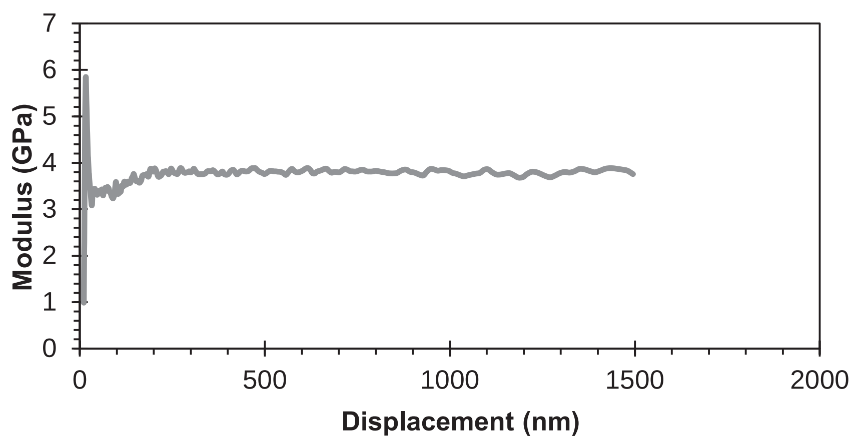


Figure B-44: Modulus vs. Displacement curve for 2 wt% xGnP®-C-300/Epoxy Test 35

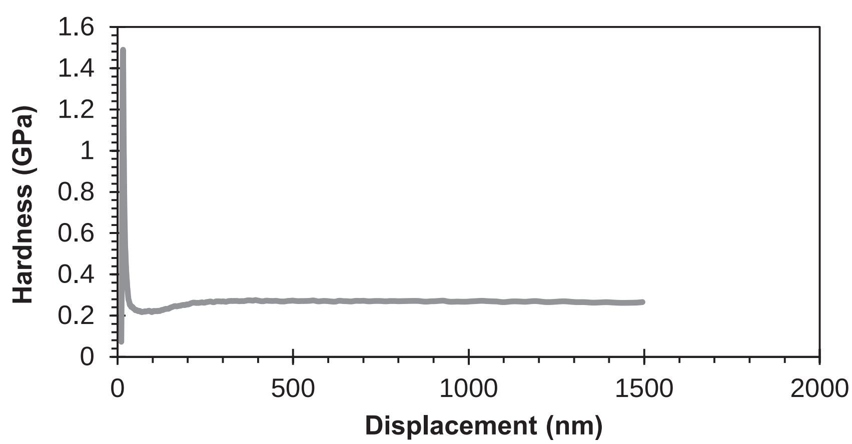


Figure B-45: Hardness vs. Displacement curve for 2 wt% xGnP®-C-300/Epoxy Test 35

Table B-16: Nanoindentation Averages Between 500 and 1500 nm Depth for 3 wt% xGnP®-C-300 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
5	3.8713	0.0786	0.2552	0.0026
7	3.7644	0.0468	0.2643	0.0059
14	3.7674	0.0869	0.2601	0.0029
16	3.8406	0.0727	0.2718	0.0078
17	3.7809	0.0525	0.2458	0.0040
18	3.8004	0.0617	0.2613	0.0018
22	3.8576	0.0725	0.2504	0.0015
23	3.7707	0.0626	0.2662	0.0626
25	3.7572	0.0661	0.2659	0.0060
30	3.8014	0.0530	0.2530	0.0034
35	3.7615	0.0590	0.2711	0.0070
37	3.7676	0.0534	0.2637	0.0033
41	3.7827	0.0669	0.2631	0.0040
42	3.7811	0.0678	0.2680	0.0034
43	3.8374	0.0528	0.2672	0.0028
49	3.8568	0.0635	0.2695	0.0034
50	3.7737	0.0428	0.2553	0.0029
53	3.8114	0.0702	0.2610	0.0021
57	3.8190	0.0687	0.2746	0.0068
58	3.7800	0.0579	0.2614	0.0021
60	3.8282	0.0630	0.2715	0.0050
61	3.7618	0.0657	0.2577	0.0022
62	3.7885	0.0674	0.2664	0.0032
63	3.7574	0.0937	0.2632	0.0042
65	3.7778	0.0651	0.2631	0.0023
68	3.7796	0.0650	0.2653	0.0032
69	3.7655	0.0555	0.2599	0.0020
70	3.7912	0.0619	0.2671	0.0050
72	3.7706	0.0723	0.2629	0.0046
Mean	**3.79**	**---**	**0.263**	**---**
Std Dev	**0.03**	**---**	**0.007**	**---**
count	**29**	**29**	**29**	**29**

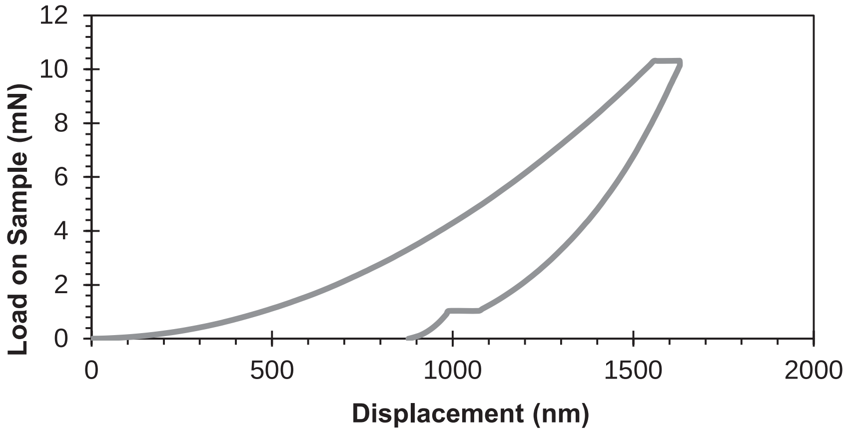


Figure B-46: Load vs. Displacement curve for 3 wt% xGnP®-C-300 in Epoxy Test 35

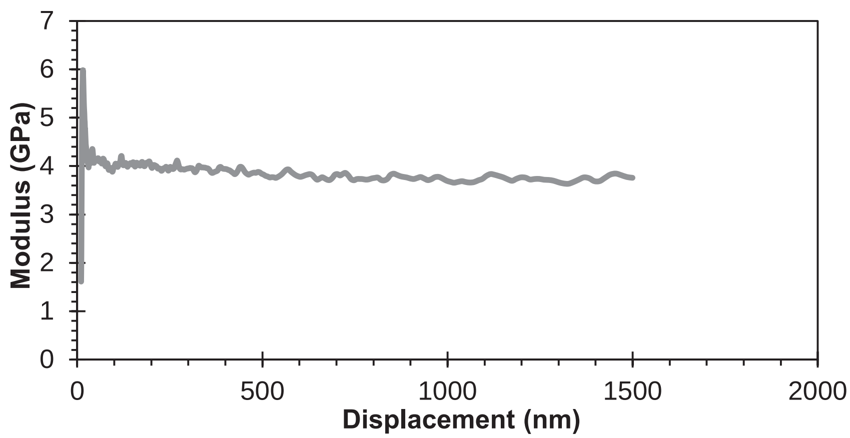


Figure B-47: Modulus vs. Displacement curve for 3 wt% xGnP®-C-300/Epoxy Test 35

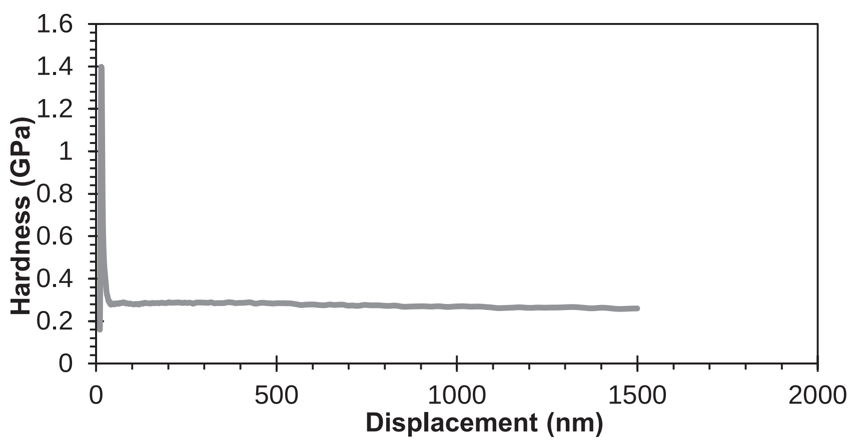


Figure B-48: Hardness vs. Displacement curve for 3 wt% xGnP®-C-300/Epoxy Test 35

Table B-17: Nanoindentation Averages Between 500 and 1500 nm Depth for 4 wt% xGnP®-C-300 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
3	3.7926	0.0660	0.2675	0.0037
6	3.9248	0.0627	0.2720	0.0041
7	3.7870	0.0609	0.2675	0.0044
9	3.7997	0.0903	0.2675	0.0083
10	3.7904	0.0578	0.2630	0.0028
13	3.8177	0.0760	0.2649	0.0027
14	3.8080	0.0623	0.2724	0.0053
16	3.9288	0.0791	0.2607	0.0053
18	3.8590	0.0632	0.2665	0.0026
19	3.8147	0.0684	0.2610	0.0029
23	3.7848	0.0638	0.2612	0.0039
27	3.7896	0.0588	0.2601	0.0020
30	3.7863	0.0556	0.2647	0.0027
31	3.7853	0.0552	0.2594	0.0020
32	3.8371	0.0817	0.2669	0.0045
34	3.7967	0.0836	0.2620	0.0027
35	3.8554	0.0642	0.2737	0.0065
38	3.7915	0.0595	0.2581	0.0021
42	3.8625	0.0744	0.2598	0.0021
45	3.9063	0.0745	0.2706	0.0031
47	3.8001	0.0573	0.2604	0.0029
51	3.8017	0.0558	0.2705	0.0043
52	3.8240	0.0485	0.2608	0.0031
56	3.7935	0.0543	0.2647	0.0022
62	3.8408	0.0609	0.2619	0.0024
63	3.8581	0.0605	0.2652	0.0029
66	3.8498	0.0558	0.2725	0.0045
67	3.8442	0.0594	0.2678	0.0043
68	3.8903	0.1072	0.2698	0.0059
70	3.8004	0.0586	0.2624	0.0024
Mean	**3.83**	**---**	**0.265**	**---**
Std Dev	**0.04**	**---**	**0.005**	**---**
Count	**30**	**30**	**30**	**30**

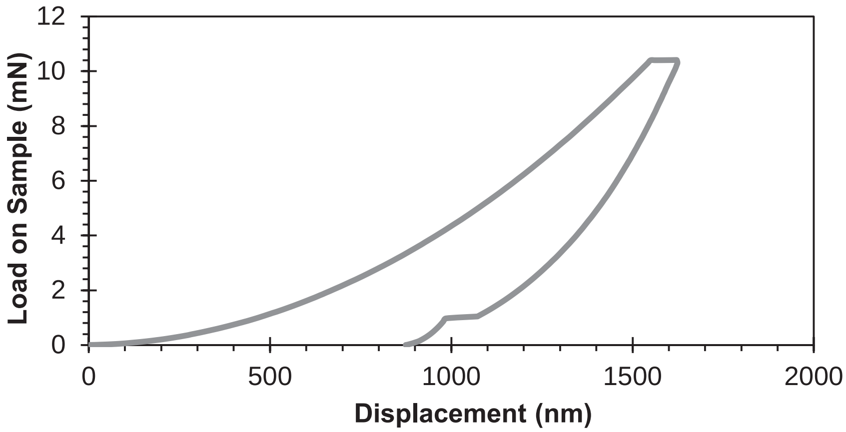


Figure B-49: Load vs. Displacement curve for 4 wt% xGnP®-C-300 in Epoxy Test 35

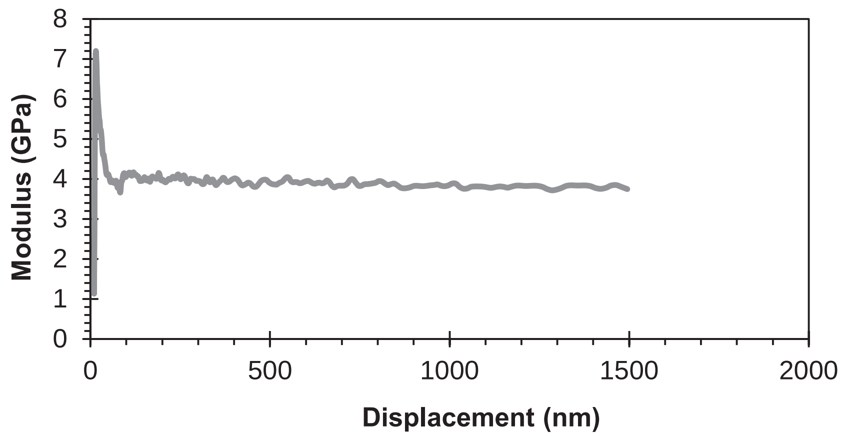


Figure B-50: Modulus vs. Displacement curve for 4 wt% xGnP®-C-300/Epoxy Test 35

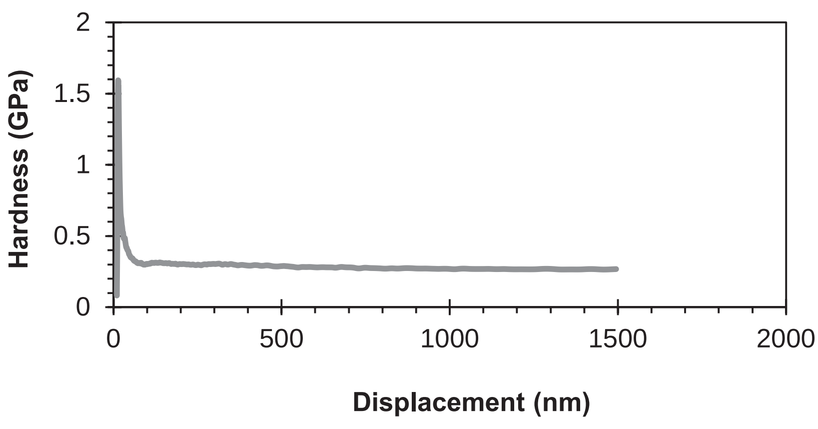


Figure B-51: Hardness vs. Displacement curve for 4 wt% xGnP®-C-300/Epoxy Test 35

Table B-18: Nanoindentation Averages Between 500 and 1500 nm Depth for 5 wt% xGnP®-C-300 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.8176	0.0475	0.2654	0.0050
2	3.8510	0.0574	0.2646	0.0039
3	3.9563	0.0773	0.2605	0.0032
5	3.8600	0.0611	0.2637	0.0043
6	3.8601	0.0815	0.2694	0.0049
9	3.9793	0.0833	0.2742	0.0069
12	4.0334	0.0909	0.2713	0.0060
15	3.8645	0.0717	0.2708	0.0068
17	3.9625	0.0682	0.2620	0.0019
18	3.8960	0.0778	0.2778	0.0072
19	3.8528	0.0559	0.2587	0.0024
21	3.8379	0.0679	0.2657	0.0057
22	3.8028	0.0589	0.2577	0.0026
23	3.9037	0.0516	0.2716	0.0050
24	3.9037	0.0516	0.2716	0.0050
25	3.9149	0.0645	0.2710	0.0059
26	4.1250	0.0506	0.2665	0.0029
28	3.8974	0.0711	0.2684	0.0030
29	3.8089	0.0640	0.2624	0.0039
30	3.8733	0.0745	0.2633	0.0032
31	4.0875	0.0648	0.2644	0.0018
32	3.8056	0.0628	0.2582	0.0021
35	3.8340	0.0658	0.2650	0.0047
36	3.8735	0.0577	0.2604	0.0024
Mean	**3.90**	**---**	**0.266**	**---**
Std Dev	**0.09**	**---**	**0.005**	**---**
Count	**24**	**24**	**24**	**24**

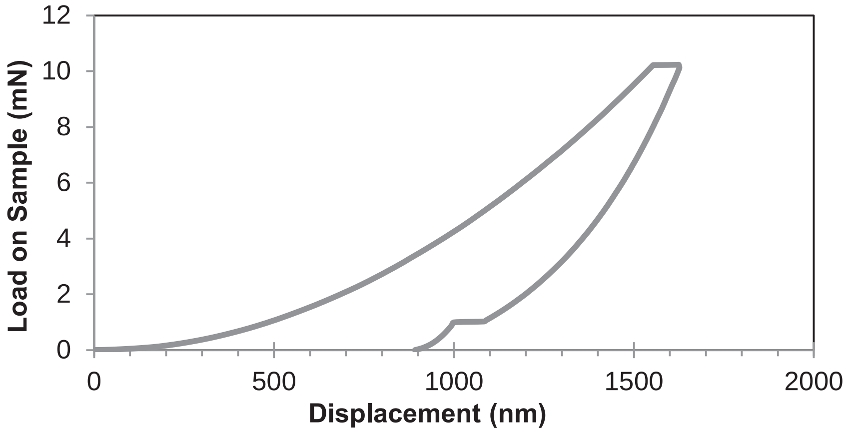


Figure B-52: Load vs. Displacement curve for 5 wt% xGnP®-C-300 in Epoxy Test 36

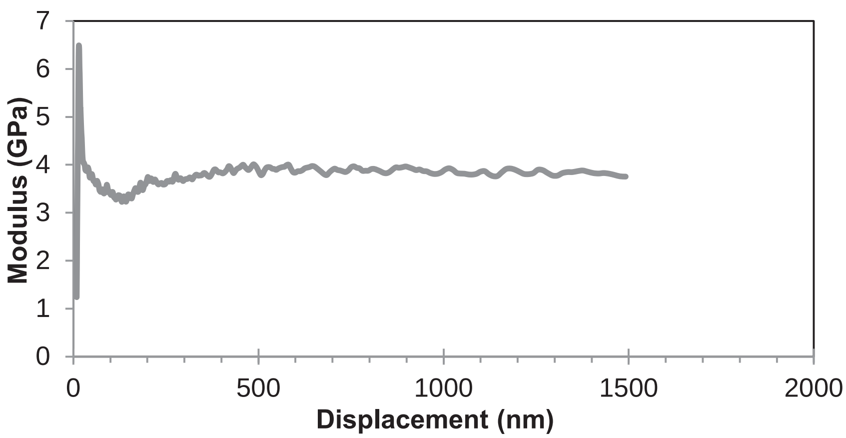


Figure B-53: Modulus vs. Displacement curve for 5 wt% xGnP®-C-300/Epoxy Test 36

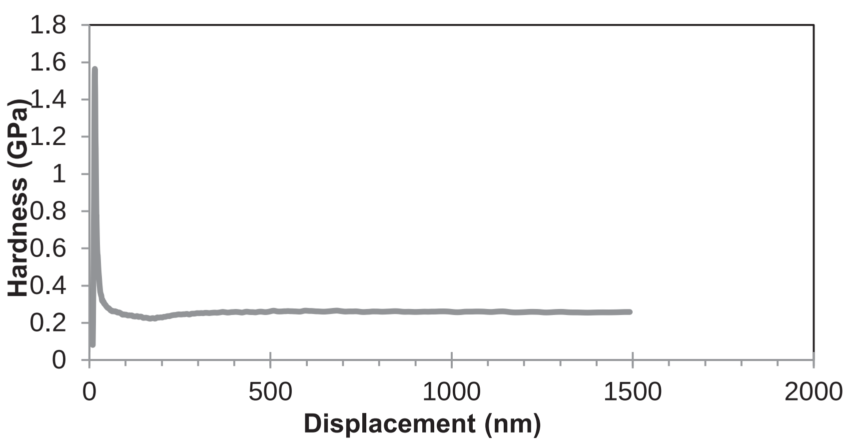


Figure B-54: Hardness vs. Displacement curve for 5 wt% xGnP®-C-300/Epoxy Test 36

Table B-19: Nanoindentation Averages Between 500 and 1500 nm Depth for 6 wt% xGnP®-C-300 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.8713	0.0579	0.2571	0.0030
2	3.8579	0.0752	0.2694	0.0035
3	3.8559	0.0542	0.2642	0.0017
5	3.8568	0.0623	0.2661	0.0023
7	3.9230	0.0642	0.2652	0.0025
9	3.8772	0.0638	0.2639	0.0026
10	3.9582	0.0774	0.2685	0.0028
12	3.9858	0.0564	0.2695	0.0029
13	3.8908	0.0528	0.2684	0.0028
17	3.8630	0.0508	0.2681	0.0036
20	3.8524	0.0700	0.2624	0.0026
21	3.9717	0.0934	0.2677	0.0030
22	3.8303	0.0544	0.2687	0.0038
23	3.8531	0.0593	0.2618	0.0019
24	3.9409	0.0696	0.2713	0.0026
25	3.8511	0.0703	0.2656	0.0032
26	4.0552	0.0761	0.2729	0.0025
27	3.8864	0.0574	0.2659	0.0032
29	3.8919	0.0711	0.2730	0.0047
31	4.1289	0.0856	0.2736	0.0021
32	3.8782	0.0553	0.2614	0.0029
33	3.9110	0.0550	0.2739	0.0053
34	3.8487	0.0669	0.2660	0.0025
35	4.0282	0.0696	0.2674	0.0023
Mean	**3.91**	**---**	**0.267**	**---**
Std Dev	**0.08**	**---**	**0.004**	**---**
Count	**24**	**24**	**24**	**24**

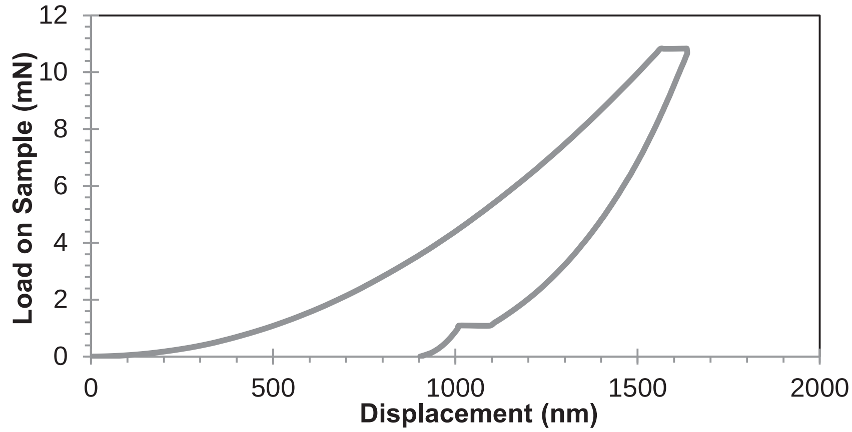


Figure B-55: Load vs. Displacement curve for 6 wt% xGnP®-C-300 in Epoxy Test 35

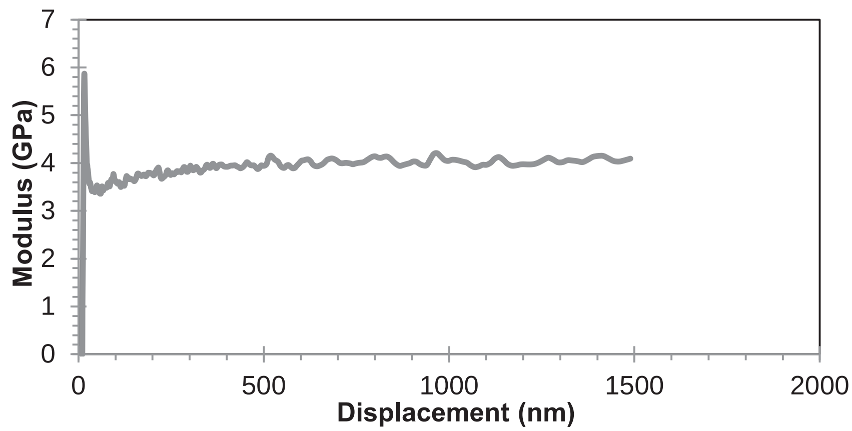


Figure B-56: Modulus vs. Displacement curve for 6 wt% xGnP®-C-300/Epoxy Test 35

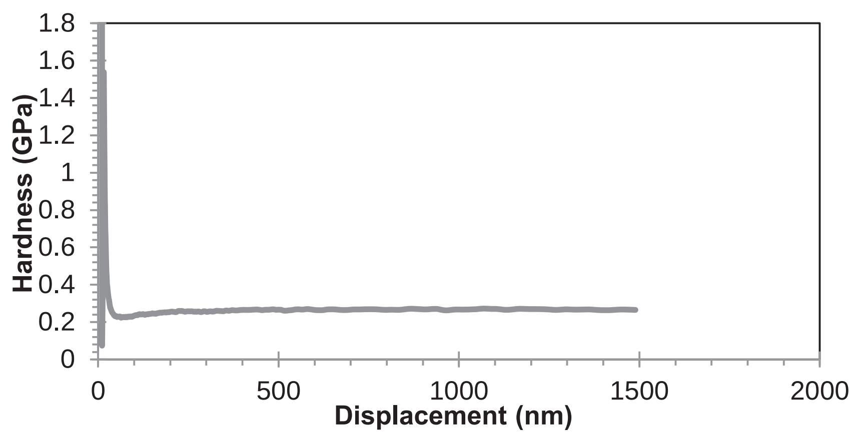


Figure B-57: Hardness vs. Displacement curve for 6 wt% xGnP®-C-300/Epoxy Test 35

Table B-20: Nanoindentation Averages Between 500 and 1500 nm Depth for 1 wt% TC307 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.7239	0.0509	0.2735	0.0033
2	3.6832	0.0568	0.2716	0.0034
3	3.7131	0.0675	0.2736	0.0037
4	3.6419	0.0525	0.2630	0.0018
5	3.6929	0.0690	0.2724	0.0042
6	3.6864	0.0532	0.2708	0.0028
7	3.6632	0.0549	0.2721	0.0035
8	3.7103	0.0671	0.2723	0.0039
9	3.6564	0.0427	0.2668	0.0017
10	3.7974	0.0775	0.2899	0.0109
12	3.7056	0.0529	0.2744	0.0041
13	3.7452	0.0615	0.2786	0.0059
14	3.6778	0.0572	0.2731	0.0041
15	3.7003	0.0524	0.2724	0.0039
16	3.6993	0.0624	0.2745	0.0044
17	3.6797	0.0617	0.2690	0.0030
18	3.7769	0.0741	0.2827	0.0061
19	3.6834	0.0561	0.2682	0.0021
20	3.6972	0.0636	0.2759	0.0049
21	3.7184	0.0569	0.2713	0.0025
22	3.6674	0.0532	0.2683	0.0034
23	3.7086	0.0493	0.2706	0.0034
24	3.6875	0.0639	0.2712	0.0038
25	3.7035	0.0519	0.2716	0.0028
27	3.7174	0.0580	0.2749	0.0053
28	3.7198	0.0691	0.2796	0.0068
29	3.7267	0.0588	0.2756	0.0048
30	3.6903	0.0577	0.2699	0.0051
31	3.6979	0.0561	0.2707	0.0043
33	3.6917	0.0595	0.2712	0.0044
34	3.6816	0.0467	0.2704	0.0040
35	3.7307	0.0840	0.2783	0.0057
36	3.7006	0.0617	0.2687	0.0019
Mean	**3.70**	**---**	**0.27**	**---**
Std Dev	**0.03**	**---**	**0.00**	**---**
Count	**33**	**33**	**33**	**33**

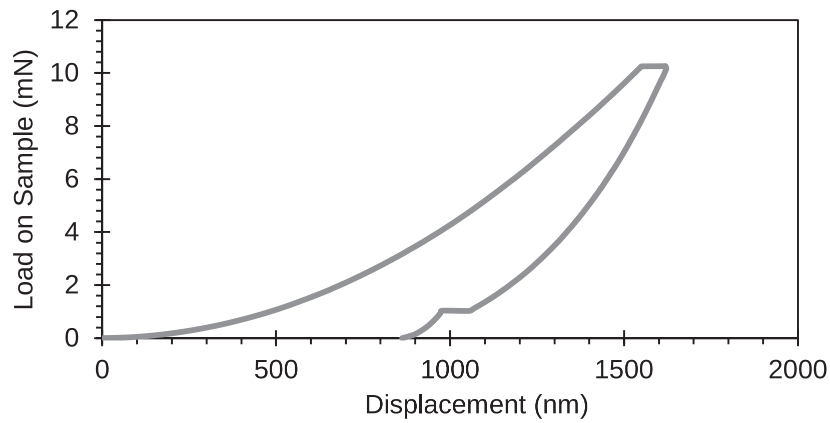


Figure B-58: Load vs. Displacement curve for 1 wt% TC307 in Epoxy Test 36

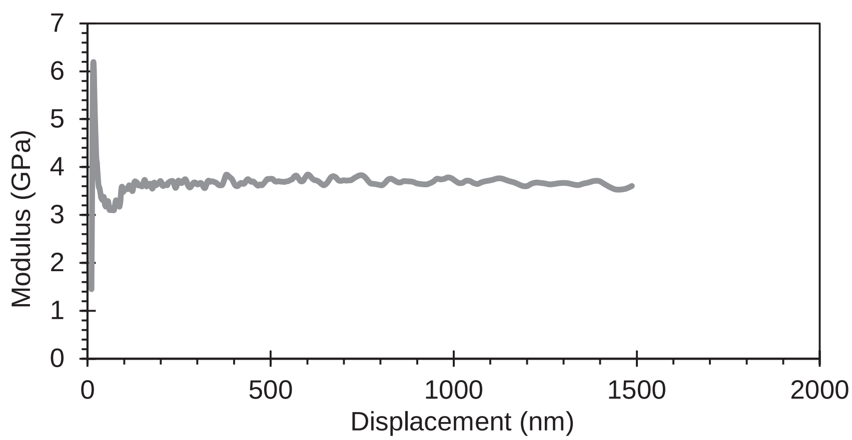


Figure B-59: Modulus vs. Displacement curve for 1 wt% TC307 in Epoxy Test 36

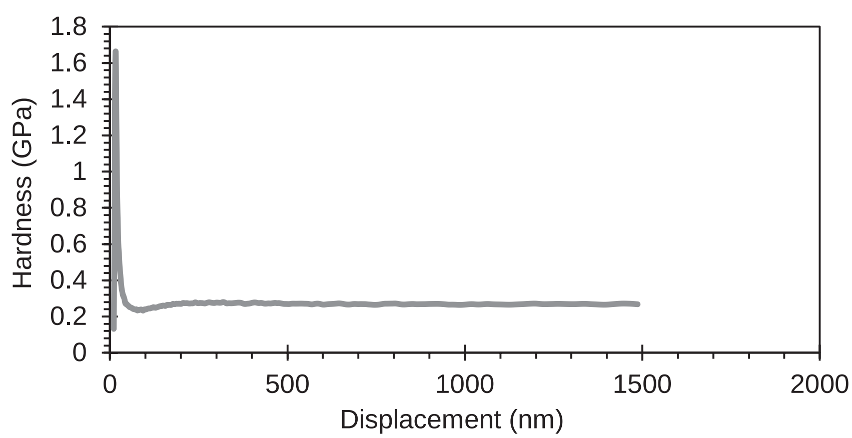


Figure B-60: Hardness vs. Displacement curve for 1 wt% TC307 in Epoxy Test 36

Table B-21: Nanoindentation Averages Between 500 and 1500 nm Depth for 2 wt% TC307 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
2	3.7162	0.0616	0.2711	0.0038
3	3.6920	0.0648	0.2671	0.0031
4	3.7231	0.0630	0.2692	0.0034
5	3.7911	0.0748	0.2745	0.0052
6	3.7813	0.0755	0.2811	0.0066
7	3.5016	0.0676	0.2434	0.0051
8	3.7366	0.0535	0.2753	0.0046
9	3.7568	0.0496	0.2757	0.0047
10	3.8141	0.0602	0.2755	0.0036
11	3.7964	0.0635	0.2741	0.0036
12	3.7364	0.0474	0.2739	0.0053
13	3.7318	0.0549	0.2740	0.0035
14	3.8237	0.0640	0.2827	0.0071
15	3.7009	0.0575	0.2682	0.0022
16	3.7977	0.0996	0.2767	0.0052
18	3.7573	0.0594	0.2751	0.0039
19	3.8009	0.0725	0.2817	0.0047
20	3.7071	0.0497	0.2690	0.0033
21	3.7839	0.0511	0.2748	0.0023
22	3.7642	0.0603	0.2815	0.0062
23	3.7547	0.0714	0.2759	0.0035
24	3.7434	0.0562	0.2742	0.0027
25	3.7724	0.0752	0.2750	0.0028
26	3.6854	0.0576	0.2666	0.0028
27	3.9585	0.0858	0.2977	0.0083
28	3.7634	0.0579	0.2778	0.0032
30	3.7080	0.0576	0.2697	0.0026
31	3.7465	0.0562	0.2786	0.0027
32	3.7323	0.0610	0.2754	0.0039
33	3.5856	0.0787	0.2493	0.0043
34	3.7392	0.0578	0.2748	0.0046
35	3.7298	0.0545	0.2743	0.0046
36	3.7157	0.0453	0.2697	0.0022
Mean	**3.74**	**---**	**0.27**	**---**
Std Dev	**0.07**	**---**	**0.01**	**---**
Count	**33**	**33**	**33**	**33**

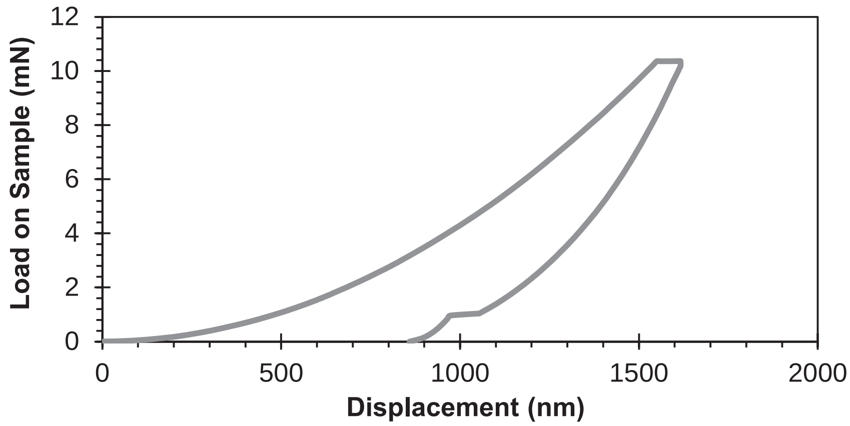


Figure B-61: Load vs. Displacement curve for 2 wt% TC307 in Epoxy Test 36

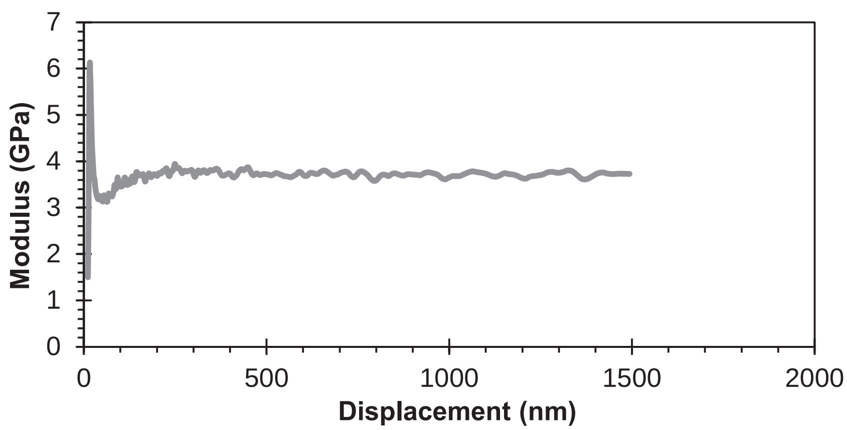


Figure B-62: Modulus vs. Displacement curve for 2 wt% TC307 in Epoxy Test 36

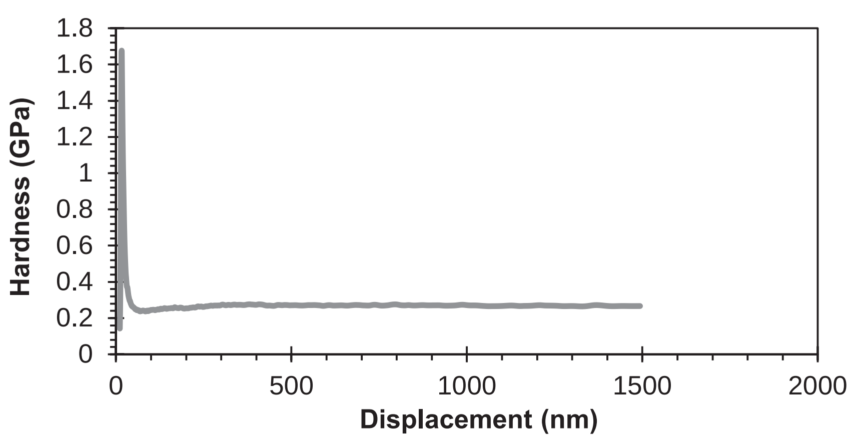


Figure B-63: Hardness vs. Displacement curve for 2 wt% TC307 in Epoxy Test 36

Table B-22: Nanoindentation Averages Between 500 and 1500 nm Depth for 3 wt% TC307 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
1	3.7613	0.0529	0.2699	0.0020
2	3.8119	0.0463	0.2788	0.0036
3	3.8351	0.0680	0.2805	0.0058
4	3.7452	0.0533	0.2738	0.0023
5	3.7956	0.0579	0.2759	0.0039
6	3.7571	0.0594	0.2685	0.0027
7	3.7592	0.0485	0.2717	0.0021
8	3.8285	0.0572	0.2793	0.0060
9	4.0016	0.1155	0.2968	0.0067
10	3.8040	0.0511	0.2760	0.0051
11	3.7877	0.0624	0.2743	0.0027
12	3.9456	0.0711	0.2941	0.0074
13	3.9006	0.0754	0.2819	0.0037
14	3.9505	0.0653	0.2847	0.0030
15	3.8163	0.0654	0.2801	0.0066
16	3.7603	0.0594	0.2704	0.0028
17	3.7614	0.0514	0.2734	0.0043
18	3.7921	0.0623	0.2787	0.0041
20	3.7293	0.0501	0.2717	0.0021
21	3.7947	0.0550	0.2786	0.0051
22	3.7835	0.0589	0.2745	0.0030
23	3.8165	0.0608	0.2785	0.0043
24	3.7838	0.0465	0.2755	0.0040
26	3.7326	0.0606	0.2710	0.0031
27	3.7919	0.0572	0.2745	0.0035
28	3.8279	0.0580	0.2781	0.0024
29	3.8835	0.0943	0.2851	0.0057
30	3.7986	0.0681	0.2772	0.0072
31	3.8531	0.0508	0.2801	0.0048
32	3.8687	0.0615	0.2873	0.0068
33	3.8019	0.0623	0.2769	0.0036
34	3.7906	0.0633	0.2754	0.0047
35	3.7765	0.0602	0.2732	0.0036
36	3.7933	0.0647	0.2763	0.0027
Mean	**3.81**	**---**	**0.28**	**---**
Std Dev	**0.06**	**---**	**0.01**	**---**
Count	**34**	**34**	**34**	**34**

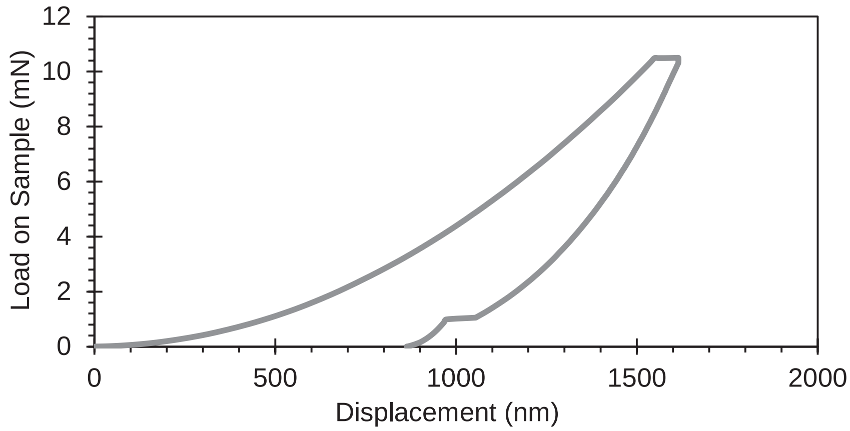


Figure B-64: Load vs. Displacement curve for 3 wt% TC307 in Epoxy Test 36

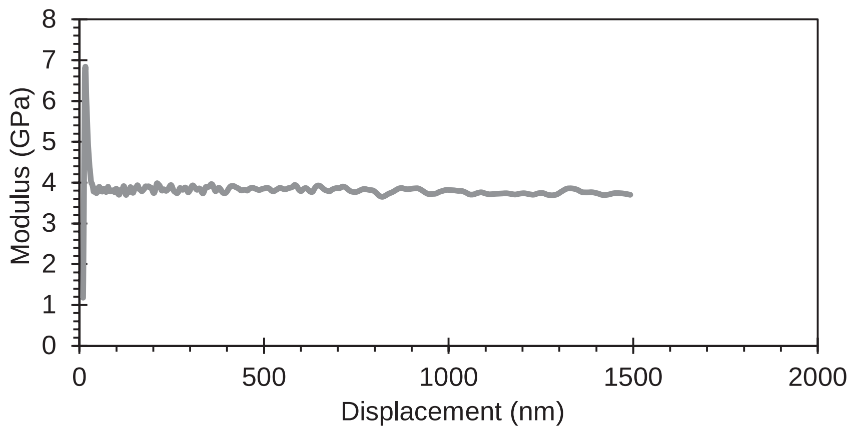


Figure B-65: Modulus vs. Displacement curve for 3 wt% TC307 in Epoxy Test 36

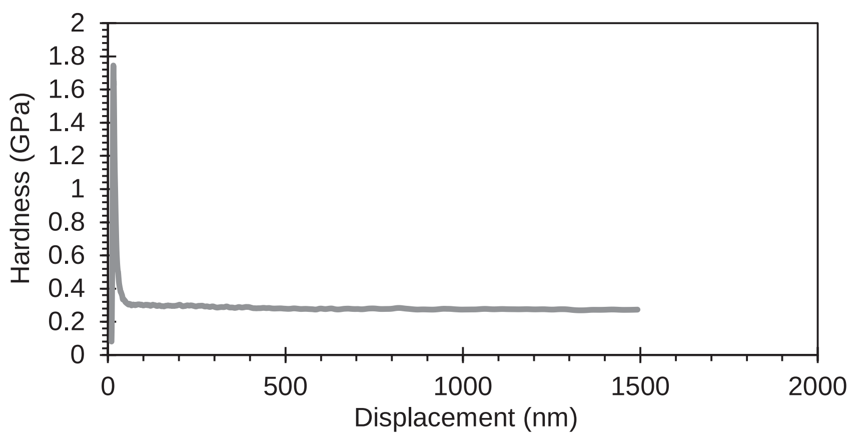


Figure B-66: Hardness vs. Displacement curve for 3 wt% TC307 in Epoxy Test 36

Table B-23: Nanoindentation Averages Between 500 and 1500 nm Depth for 4 wt% TC307 in Epoxy

Test	Average Modulus (GPa)	Modulus Std Dev	Average Hardness (GPa)	Hardness Std Dev
2	3.7709	0.0583	0.2725	0.0030
3	3.8621	0.0742	0.2825	0.0052
4	3.8513	0.0633	0.2805	0.0049
5	3.7873	0.0666	0.2691	0.0057
6	3.7898	0.0617	0.2795	0.0040
7	3.7733	0.0541	0.2749	0.0025
8	3.8160	0.0631	0.2736	0.0024
9	3.8258	0.0656	0.2841	0.0047
10	3.8387	0.0674	0.2833	0.0059
11	3.7992	0.0604	0.2764	0.0024
12	3.8163	0.0569	0.2732	0.0029
13	3.8191	0.0744	0.2803	0.0069
14	3.8472	0.0634	0.2836	0.0043
16	3.7791	0.0537	0.2737	0.0036
17	4.1407	0.2393	0.3171	0.0197
18	3.8238	0.0582	0.2813	0.0031
19	3.8016	0.0678	0.2687	0.0029
20	4.0229	0.1110	0.3023	0.0078
21	3.9048	0.0600	0.2773	0.0021
22	3.8231	0.0544	0.2796	0.0044
23	3.9114	0.0753	0.2901	0.0064
24	3.7981	0.0595	0.2771	0.0048
25	3.8550	0.0586	0.2825	0.0049
26	3.8950	0.0648	0.2858	0.0054
27	3.8688	0.0721	0.2846	0.0078
28	3.8579	0.0642	0.2852	0.0082
29	3.8456	0.0834	0.2865	0.0070
30	3.8859	0.1090	0.2789	0.0041
31	3.7992	0.0525	0.2788	0.0056
32	3.8481	0.0611	0.2836	0.0050
33	3.8129	0.0600	0.2767	0.0029
34	3.8080	0.0562	0.2769	0.0033
35	3.8226	0.0492	0.2829	0.0041
Mean	**3.85**	**---**	**0.28**	**---**
Std Dev	**0.07**	**---**	**0.01**	**---**
Count	**33**	**33**	**33**	**33**

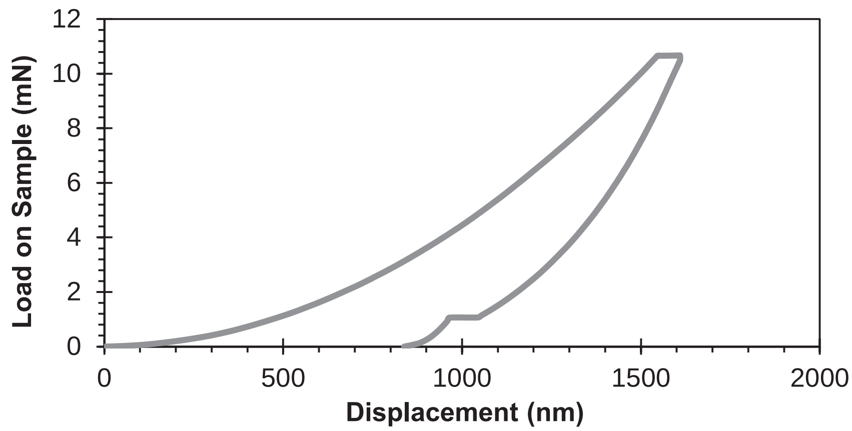


Figure B-67: Load vs. Displacement curve for 4 wt% TC307 in Epoxy Test 36

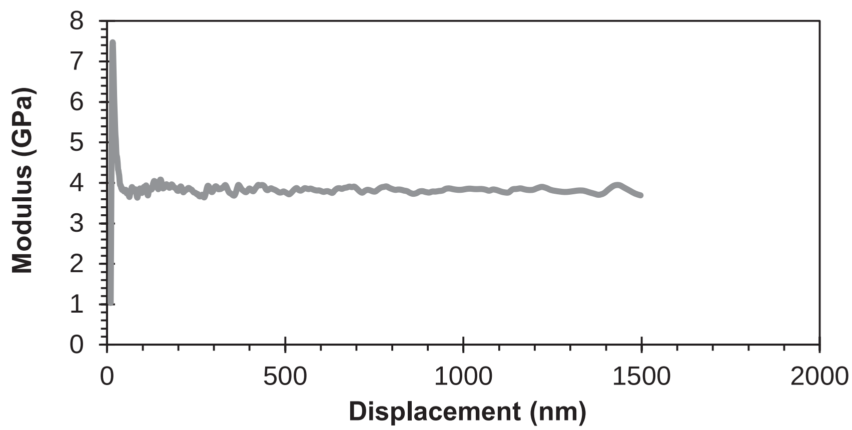


Figure B-68: Modulus vs. Displacement curve for 4 wt% TC307 in Epoxy Test 36

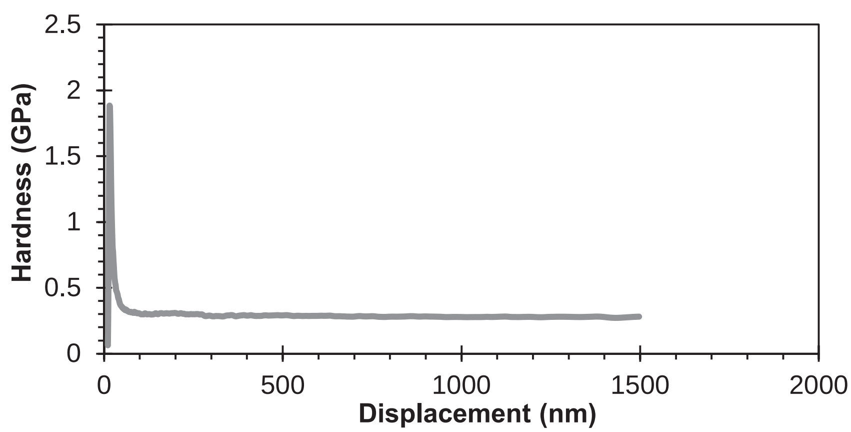


Figure B-69: Hardness vs. Displacement curve for 4 wt% TC307 in Epoxy Test 36

Appdenix C: Electrical Resistivity Results

Table C-1: ASTM D257 Thru-Plane Electrical Resistivity Results for Neat Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-4-17-12	1	100	2.8174E+16
A862-4-17-12	2	100	3.1263E+16
A862-4-17-12	3	100	2.8694E+16
A862-4-17-12	4	100	3.3570E+16
A862-4-17-12	5	100	2.5305E+16
A862-4-17-12	6	100	2.5670E+16
Average			**2.87793E+16**
Standard Deviation			**3.20099E+15**
Count			**6**

Table C-2: ASTM D257 Thru-Plane Electrical Resistivity Results for 1 wt% xGnP®-M-15 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M15-1-5-10-12	1	100	6.9052E+15
A862-M15-1-5-10-12	2	100	3.0085E+15
A862-M15-1-5-10-12	3	100	4.754E+15
A862-M15-1-5-10-12	4	100	7.5537E+15
A862-M15-1-5-10-12	5	100	1.1287E+16
A862-M15-1-5-10-12	6	100	7.1802E+15
Average			**6.78E+15**
Standard Deviation			**2.81E+15**
Count			**6**

Table C-3: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 2 wt% xGnP®-M-15 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M15-2-5-2-12	1	20	1.3453E+09
A862-M15-2-5-2-12	3	20	2.9236E+09
A862-M15-2-5-2-12	4	20	4.1336E+09
A862-M15-2-5-2-12	6	20	2.4117E+09
A862-M15-2-5-2-12	7	20	2.2122E+09
Average			**2.61E+09**
Standard Deviation			**1.03E+09**
Count			**5**

Table C-4: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 3 wt% xGnP®-M-15 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M15-3-5-1-12	2	20	1.0894E+07
A862-M15-3-5-1-12	3	20	1.5487E+08
A862-M15-3-5-1-12	5	20	3.0470E+08
A862-M15-3-5-1-12	6	20	1.7174E+07
A862-M15-3-5-1-12	7	20	2.5402E+07
Average			**1.03E+08**
Standard Deviation			**1.28E+08**
Count			**5**

Table C-5: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 4 wt% xGnP®-M-15 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M15-4-4-27-12	1	20	1.9267E+07
A862-M15-4-4-27-12	2	20	1.3121E+06
A862-M15-4-4-27-12	5	20	4.3010E+07
A862-M15-4-4-27-12	6	20	1.5718E+07
A862-M15-4-4-27-12	10	20	4.6043E+06
Average			**1.68E+07**
Standard Deviation			**1.65E+07**
Count			**5**

Table C-6: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 5 wt% xGnP®-M-15 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M15-5-4-26-12	4	10	1.0092E+07
A862-M15-5-4-26-12	7	10	1.5715E+07
A862-M15-5-4-26-12	8	10	1.5547E+07
A862-M15-5-4-26-12	9	10	1.8111E+05
A862-M15-5-4-26-12	10	10	4.9666E+06
Average			**9.30E+06**
Standard Deviation			**6.76E+06**
Count			**5**

Table C-7: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 6 wt% xGnP®-M-15 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M15-6-4-19-12	1	20	4.0667E+06
A862-M15-6-4-19-12	2	20	8.2678E+06
A862-M15-6-4-19-12	3	20	3.3021E+06
A862-M15-6-4-19-12	4	20	8.2510E+05
A862-M15-6-4-19-12	7	20	7.5786E+06
A862-M15-6-4-19-12	8	20	6.5565E+06
Average			**5.10E+06**
Standard Deviation			**2.86E+06**
Count			**6**

Table C-8: ASTM D257 Thru-Plane Electrical Resistivity Results for 1 wt% xGnP®-M-5 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M5-1-9-28-12	1	100	2.9730E+15
A862-M5-1-9-28-12	2	100	1.6002E+15
A862-M5-1-9-28-12	3	100	1.7335E+15
A862-M5-1-9-28-12	4	100	3.9281E+15
A862-M5-1-9-28-12	5	100	3.3609E+15
Average			**2.72E+15**
Standard Deviation			**1.02E+15**
Count			**5**

Table C-9: ASTM D257 Thru-Plane Electrical Resistivity Results for 2 wt% xGnP®-M-5 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M5-2-8-28-12	1	100	6.7435E+15
A862-M5-2-8-28-12	2	100	5.7857E+15
A862-M5-2-8-28-12	3	100	2.8083E+15
A862-M5-2-8-28-12	4	100	3.4980E+15
Average			**4.71E+15**
Standard Deviation			**1.86E+15**
Count			**4**

Table C-10: ASTM D257 Thru-Plane Electrical Resistivity Results for 3 wt% xGnP®-M-5 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M5-3-10-3-12	1	100	1.0830E+15
A862-M5-3-10-3-12	2	100	7.8721E+13
A862-M5-3-10-3-12	3	100	1.6381E+15
A862-M5-3-10-3-12	4	100	1.0940E+15
Average			**9.73E+14**
Standard Deviation			**6.50E+14**
Count			**4**

Table C-11: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 4 wt% xGnP®-M-5 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M5-4-10-9-12	1	20	3.1778E+09
A862-M5-4-10-9-12	2	20	6.0006E+09
A862-M5-4-10-9-12	3	20	6.4053E+09
A862-M5-4-10-9-12	4	20	1.2004E+08
A862-M5-4-10-9-12	5	20	3.1864E+09
Average			**3.78E+09**
Standard Deviation			**2.55E+09**
Count			**5**

Table C-12: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 5 wt% xGnP®-M-5 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M5-5-10-10-12	1	20	3.3884E+06
A862-M5-5-10-10-12	2	20	5.0785E+06
A862-M5-5-10-10-12	3	20	8.5093E+07
A862-M5-5-10-10-12	4	20	2.0494E+09
A862-M5-5-10-10-12	5	20	8.8652E+08
Average			**6.06E+08**
Standard Deviation			**8.88E+08**
Count			**5**

Table C-13: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 6 wt% xGnP®-M-5 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-M5-6-10-15-12	1	20	1.9957E+08
A862-M5-6-10-15-12	2	20	1.6696E+06
A862-M5-6-10-15-12	3	20	4.8455E+07
A862-M5-6-10-15-12	4	20	7.9778E+08
A862-M5-6-10-15-12	5	20	2.1487E+07
Average			**2.14E+08**
Standard Deviation			**3.36E+08**
Count			**5**

Table C-14: ASTM D257 Thru-Plane Electrical Resistivity Results for 1 wt% xGnP®-C-300 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-C300-1-3-22-13	1	100	3.7129E+16
A862-C300-1-3-22-13	2	100	4.4553E+16
A862-C300-1-3-22-13	3	100	2.6489E+16
A862-C300-1-3-22-13	4	100	2.2575E+15
A862-C300-1-3-22-13	5	100	1.331E+16
A862-C300-1-3-22-13	6	100	3.3842E+16
Average			**2.63E+16**
Standard Deviation			**1.58E+16**
Count			**6**

Table C-15: ASTM D257 Thru-Plane Electrical Resistivity Results for 2 wt% xGnP®-C-300 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-C300-2-3-7-13	1	100	6.9635E+15
A862-C300-2-3-7-13	2	100	2.6773E+15
A862-C300-2-3-7-13	3	100	4.8582E+15
A862-C300-2-3-7-13	4	100	5.8175E+15
A862-C300-2-3-7-13	5	100	7.8765E+15
A862-C300-2-3-7-13	6	100	2.2335E+15
Average			**5.07E+15**
Standard Deviation			**2.27E+15**
Count			**6**

Table C-16: ASTM D257 Thru-Plane Electrical Resistivity Results for 3 wt% xGnP®-C-300 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-C300-3-3-5-13	2	100	5.6470E+15
A862-C300-3-3-5-13	3	100	4.6057E+15
A862-C300-3-3-5-13	4	100	4.1309E+15
A862-C300-3-3-5-13	5	100	5.6662E+15
A862-C300-3-3-5-13	6	100	9.2082E+15
Average			**5.85E+15**
Standard Deviation			**1.99E+15**
Count			**5**

Table C-17: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 4 wt% xGnP®- C-300 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-C300-4-7-15-13	1	20	4.1362E+08
A862-C300-4-7-15-13	2	20	2.9932E+08
A862-C300-4-7-15-13	3	20	2.7520E+08
A862-C300-4-7-15-13	4	20	4.1075E+08
A862-C300-4-7-15-13	5	20	3.4078E+08
Average			**3.48E+08**
Standard Deviation			**6.32E+07**
Count			**5**

Table C-18: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 5 wt% xGnP®- C-300 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-C300-5-4-1-13	1	20	3.8954E+08
A862-C300-5-4-1-13	2	20	1.7501E+08
A862-C300-5-4-1-13	3	20	2.8087E+08
A862-C300-5-4-1-13	4	20	3.0243E+07
A862-C300-5-4-1-13	5	20	1.1860E+08
Average			**1.99E+08**
Standard Deviation			**1.40E+08**
Count			**5**

Table C-19: ASTM D4496 Two Point In-Plane Electrical Resistivity Results for 6 wt% xGnP®- C-300 in Epoxy

Sample	No.	Applied Voltage (V)	Volume Electrical Resistivity (Ω-cm)
A862-C300-6-4-4-13	1	20	1.5909E+08
A862-C300-6-4-4-13	2	20	2.3288E+07
A862-C300-6-4-4-13	3	20	4.6670E+06
A862-C300-6-4-4-13	4	20	1.5175E+07
A862-C300-6-4-4-13	5	20	1.5353E+07
Average			**4.35E+07**
Standard Deviation			**6.49E+07**
Count			**5**

Appdenix D: DMA Results

Table D-1: DMA Results for Neat Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-3-6-13	1	139.15	149.40	155.29
A862-3-6-13	2	139.54	149.84	155.57
A862-3-6-13	3	139.76	149.76	155.48
A862-3-6-13	4	139.12	150.11	156.29
A862-3-6-13	5	138.49	149.61	155.79
A862-3-6-13	6	139.74	150.12	156.13
Average		**139.3**	**149.8**	**155.8**
Standard Deviation		**0.5**	**0.3**	**0.4**
Count		**6**	**6**	**6**

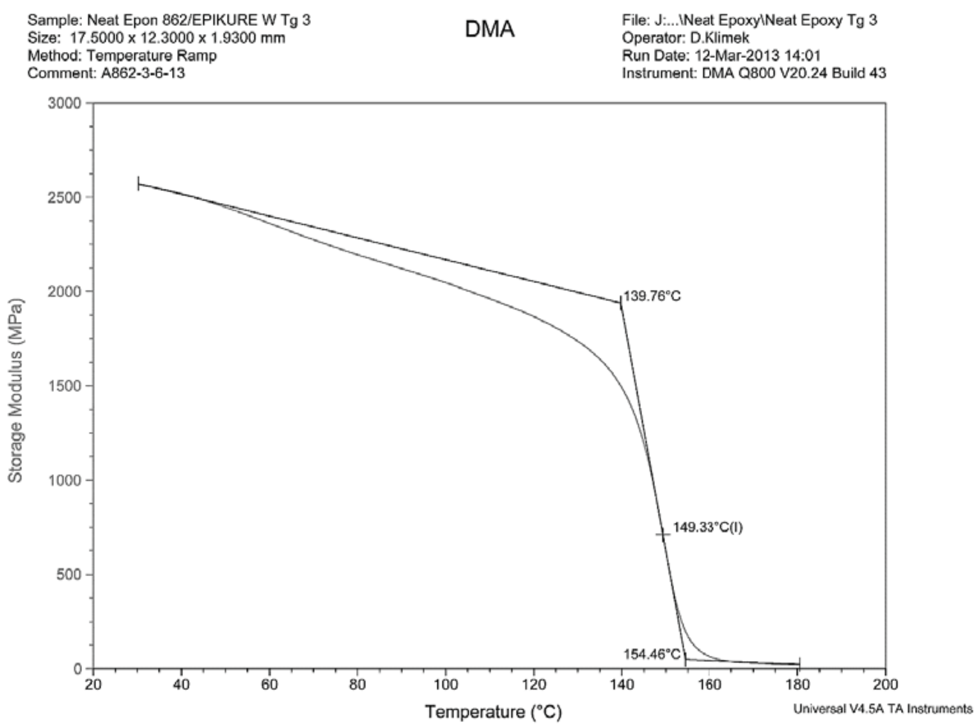


Figure D-1: Storage Modulus for Neat Epoxy Test 3

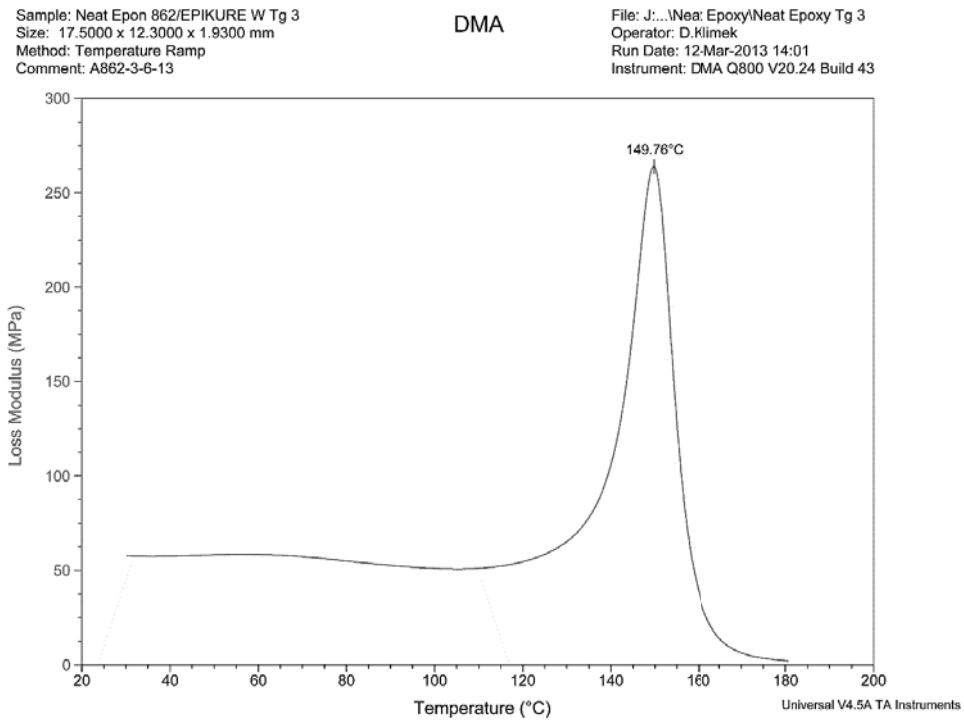


Figure D-2: Loss Modulus for Neat Epoxy Test 3

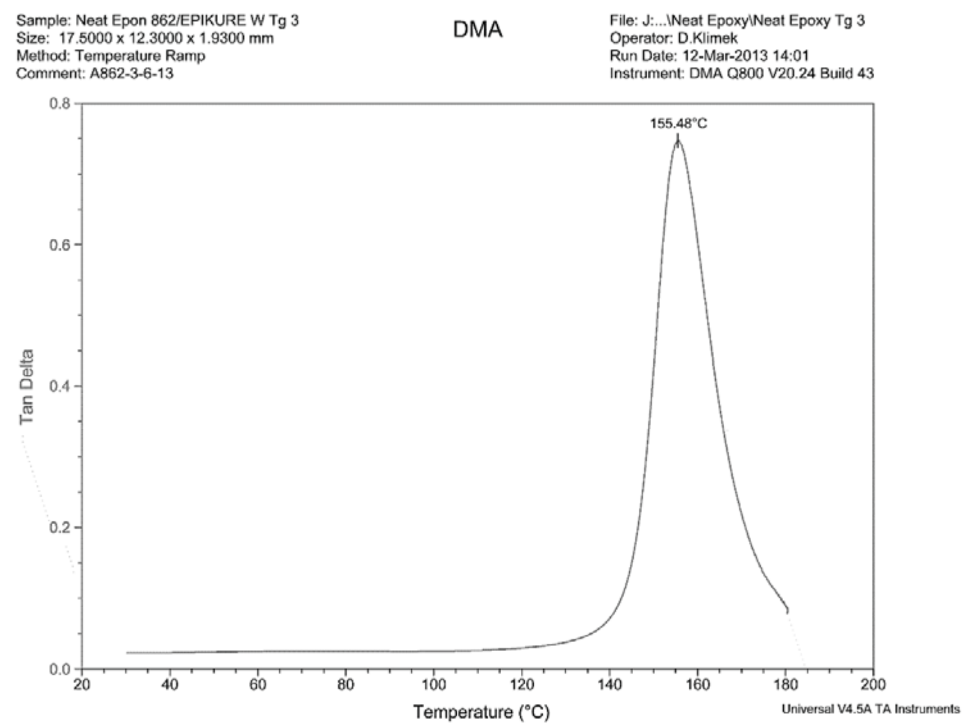


Figure D-3: Tan Delta for Neat Epoxy Test 3

Table D-2: DMA Results for 1 wt% xGnP®-M-15 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M-151-3-7-12	1	130.96	144.97	152.89
A862-M-151-3-7-12	2	132.24	145.62	153.70
A862-M-151-3-7-12	3	132.55	145.42	153.83
Average		**131.9**	**145.3**	**153.5**
Standard Deviation		**0.8**	**0.3**	**0.5**
Count		**3**	**3**	**3**

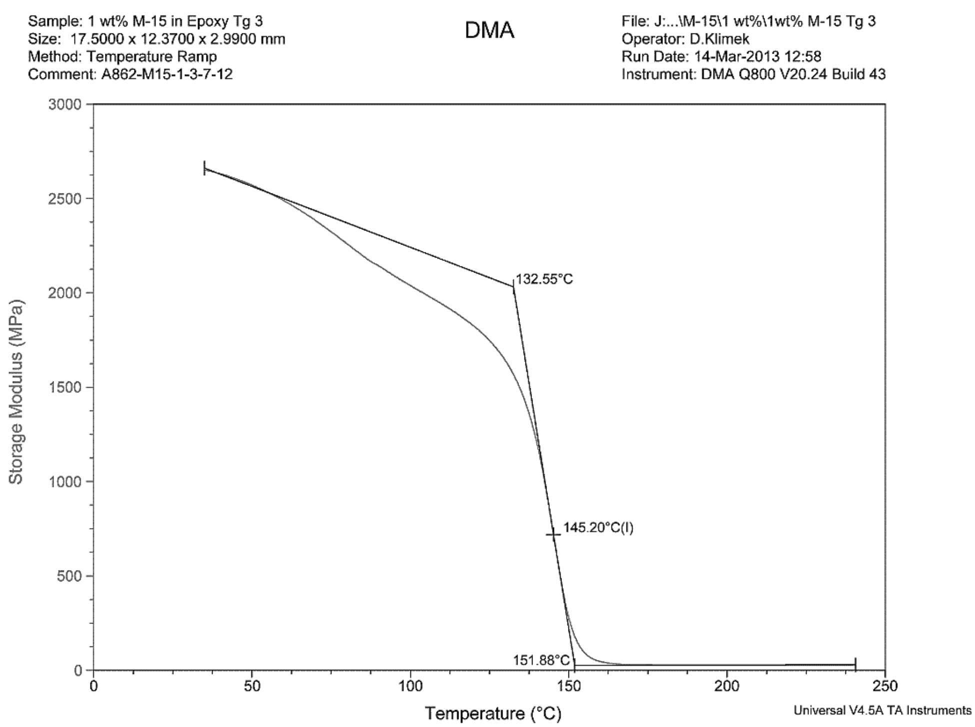
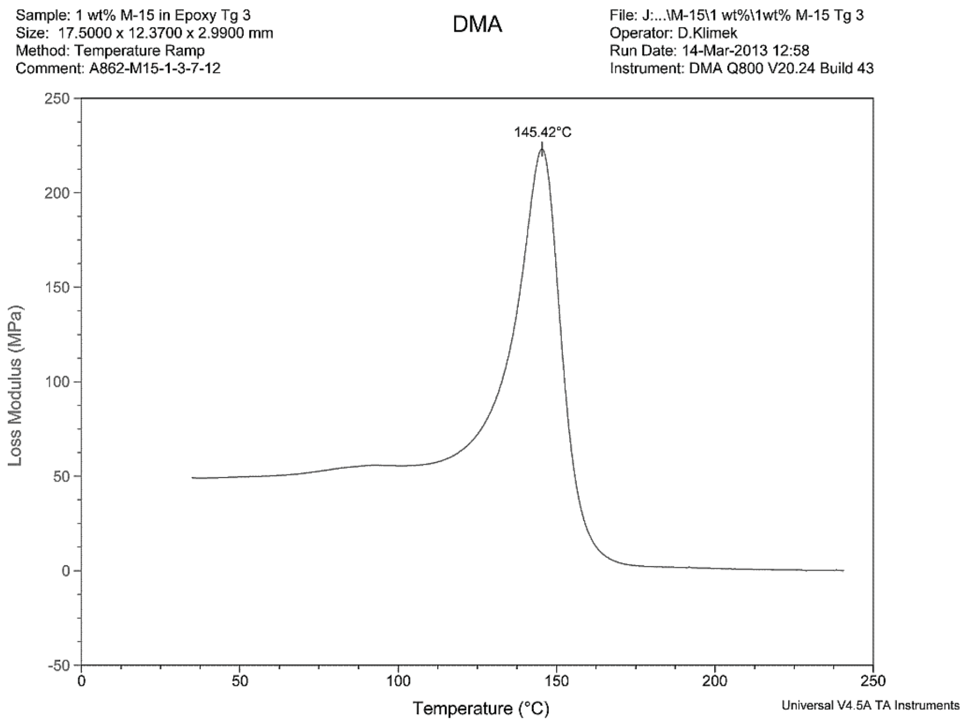


Figure D-4: Storage Modulus for 1 wt% xGnP®-M-15 in Epoxy Test 3



3

Figure D-5: Loss Modulus for 1 wt% xGnP®-M-15 in Epoxy Test 3

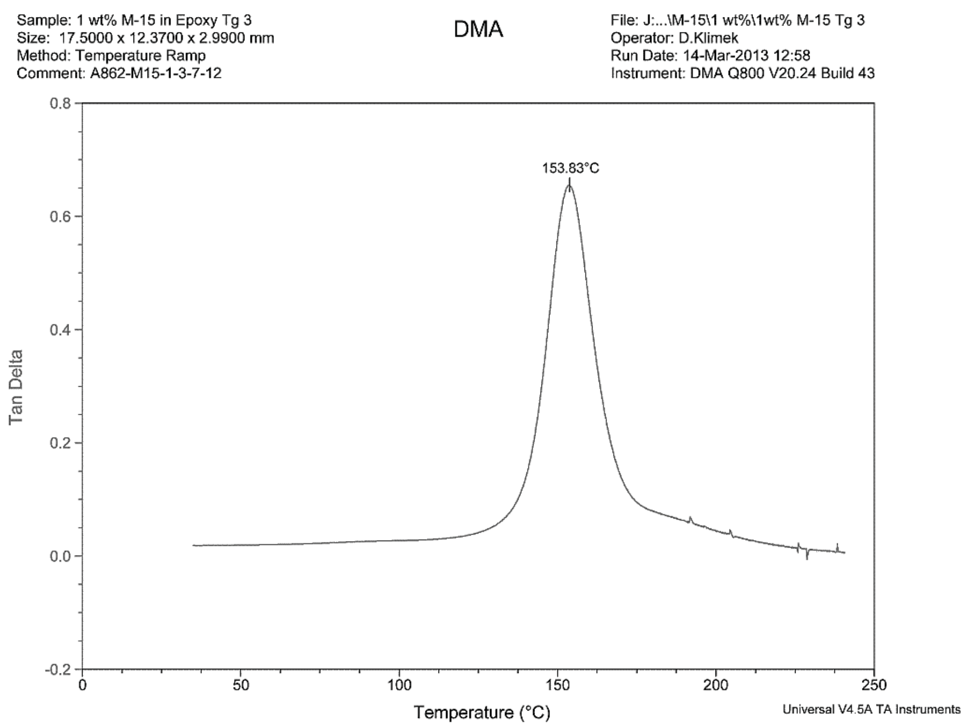


Figure D-6: Tan Delta for 1 wt% xGnP®-M-15 in Epoxy Test 3

Table D-3: DMA Results for 2 wt% xGnP®-M-15 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M15-2-2-22-12	1	130.50	144.20	152.81
A862-M15-2-2-22-12	2	129.23	142.46	151.55
A862-M15-2-2-22-12	3	129.92	142.92	152.02
A862-M15-2-5-2-12	4	131.25	143.96	153.01
A862-M15-2-5-2-12	5	130.33	144.30	153.65
Average		**130.2**	**143.6**	**152.6**
Standard Deviation		**0.7**	**0.8**	**0.8**
Count		**5**	**5**	**5**

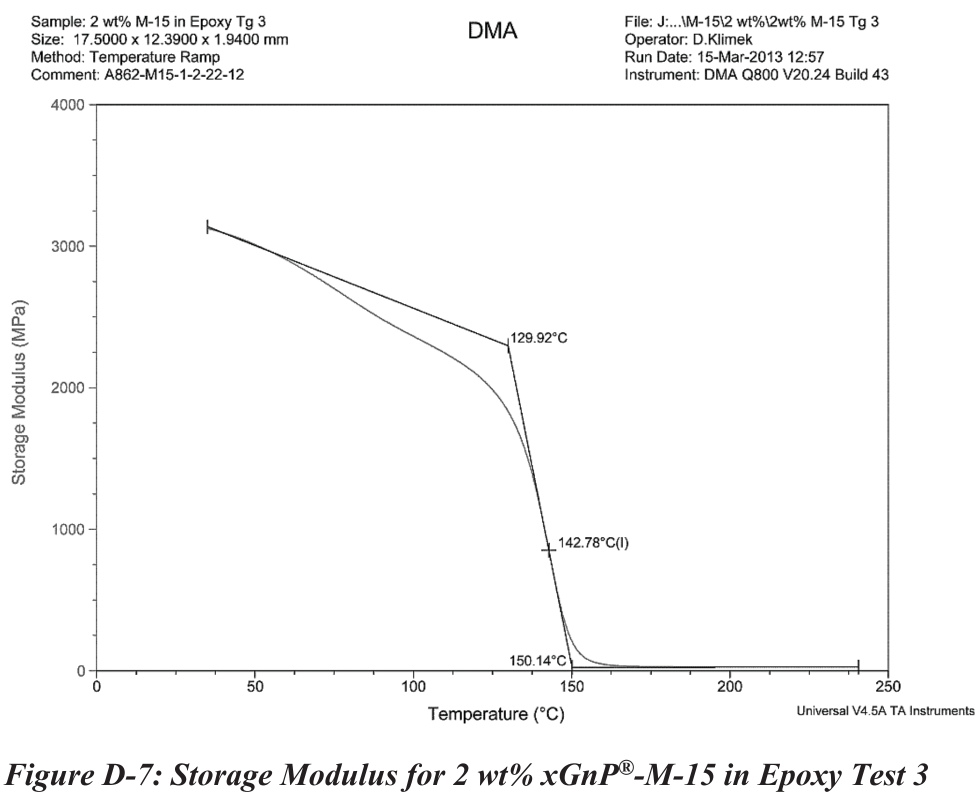


Figure D-7: Storage Modulus for 2 wt% xGnP®-M-15 in Epoxy Test 3

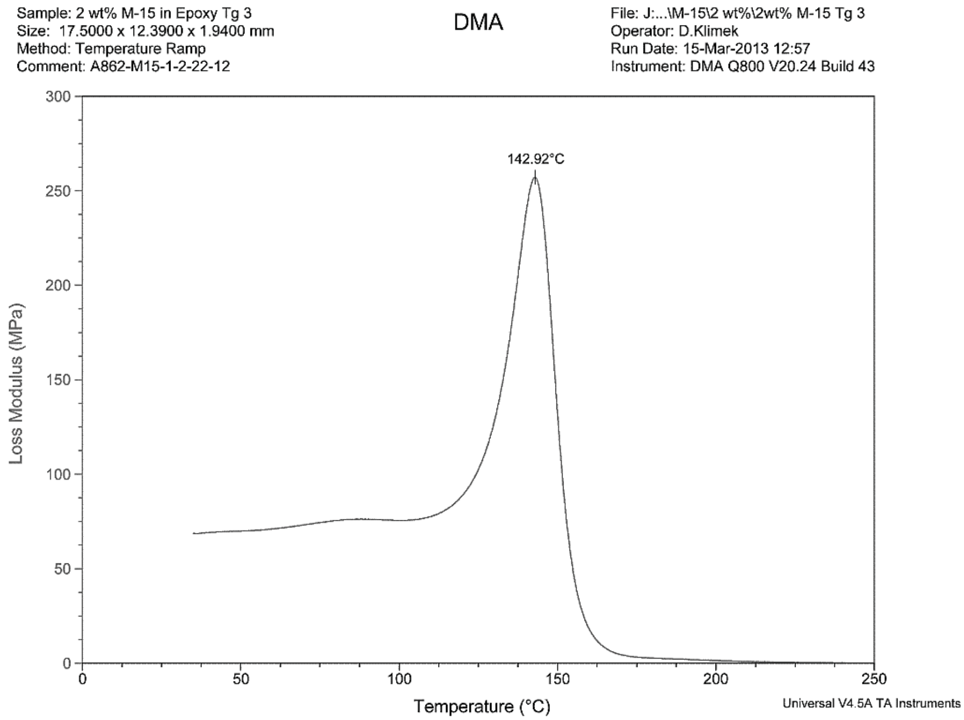


Figure D-8: Loss Modulus for 2 wt% xGnP®-M-15 in Epoxy Test 3

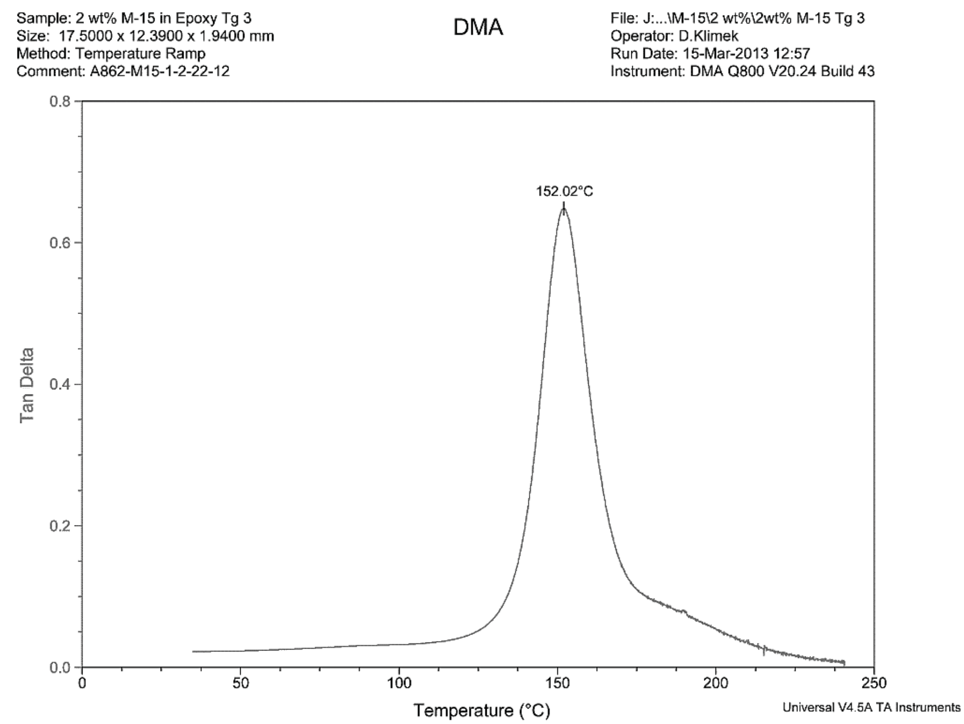


Figure D-9: Tan Delta for 2 wt% xGnP®-M-15 in Epoxy Test 3

Table D-4: DMA Results for 3 wt% xGnP®-M-15 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M15-3-3-14-12	1	130.59	143.70	150.83
A862-M15-3-3-14-12	2	129.92	142.74	150.22
A862-M15-3-3-14-12	3	130.82	142.77	150.26
Average		**130.4**	**143.1**	**150.4**
Standard Deviation		**0.5**	**0.5**	**0.3**
Count		**3**	**3**	**3**

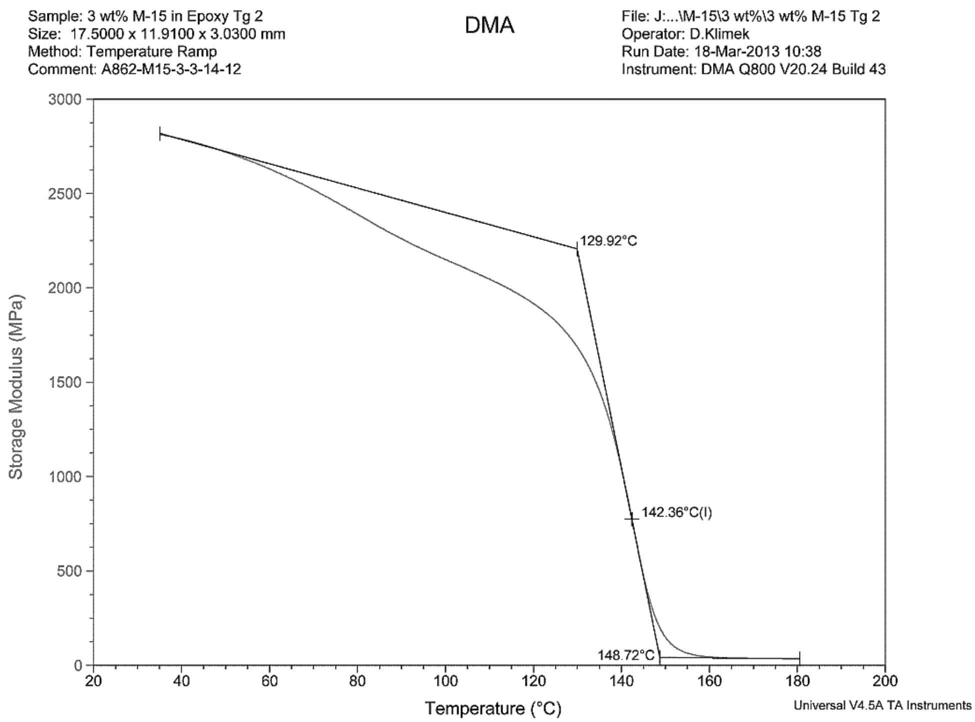


Figure D-10: Storage Modulus for 3 wt% xGnP®-M-15 in Epoxy Test 2

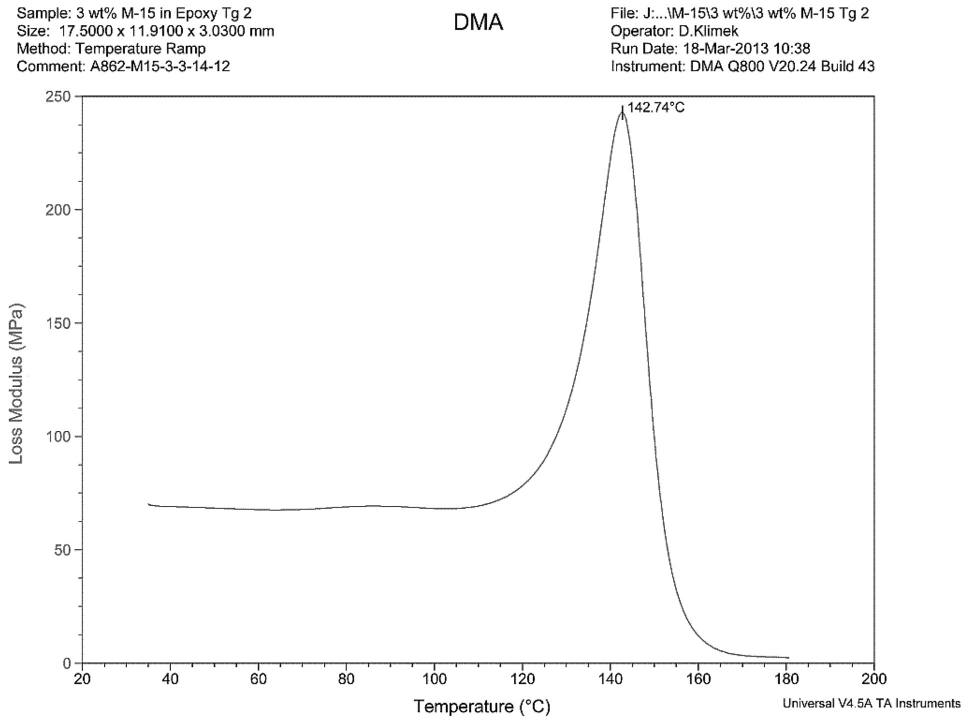


Figure D-11: Loss Modulus for 3 wt% xGnP®-M-15 in Epoxy Test 2

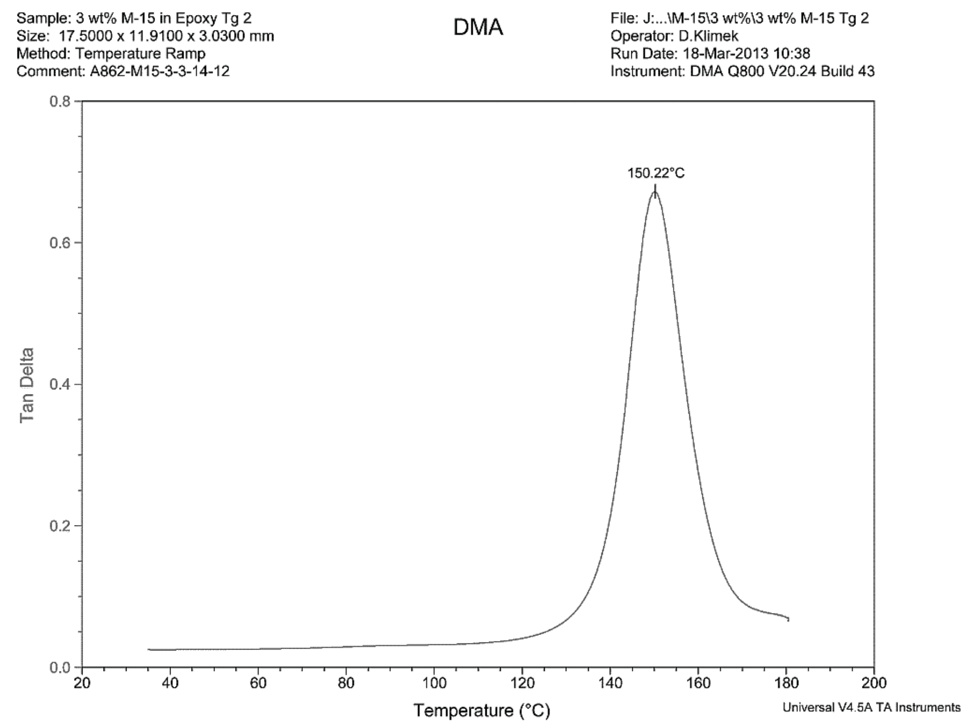


Figure D-12: Tan Delta for 3 wt% xGnP®-M-15 in Epoxy Test 2

Table D-5: DMA Results for 4 wt% xGnP®-M-15 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M15-4-3-15-12	1	131.82	144.63	152.71
A862-M15-4-3-15-12	2	130.79	143.68	152.26
A862-M15-4-3-15-12	3	131.90	143.98	152.26
Average		**131.5**	**144.1**	**152.4**
Standard Deviation		**0.6**	**0.5**	**0.3**
Count		**3**	**3**	**3**

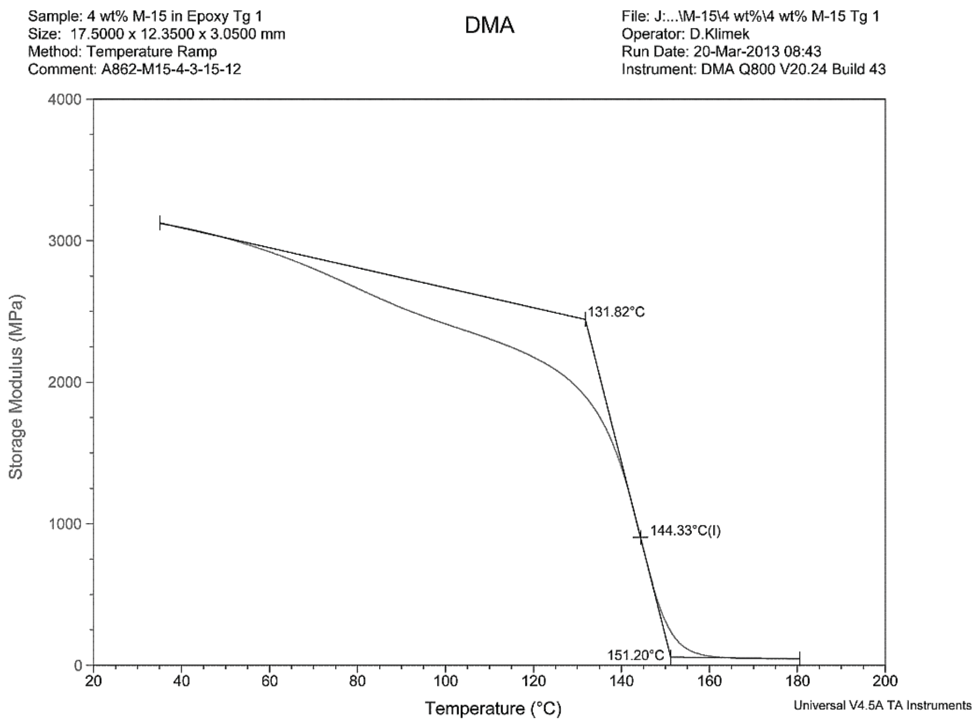


Figure D-13: Storage Modulus for 4 wt% xGnP®-M-15 in Epoxy Test 1

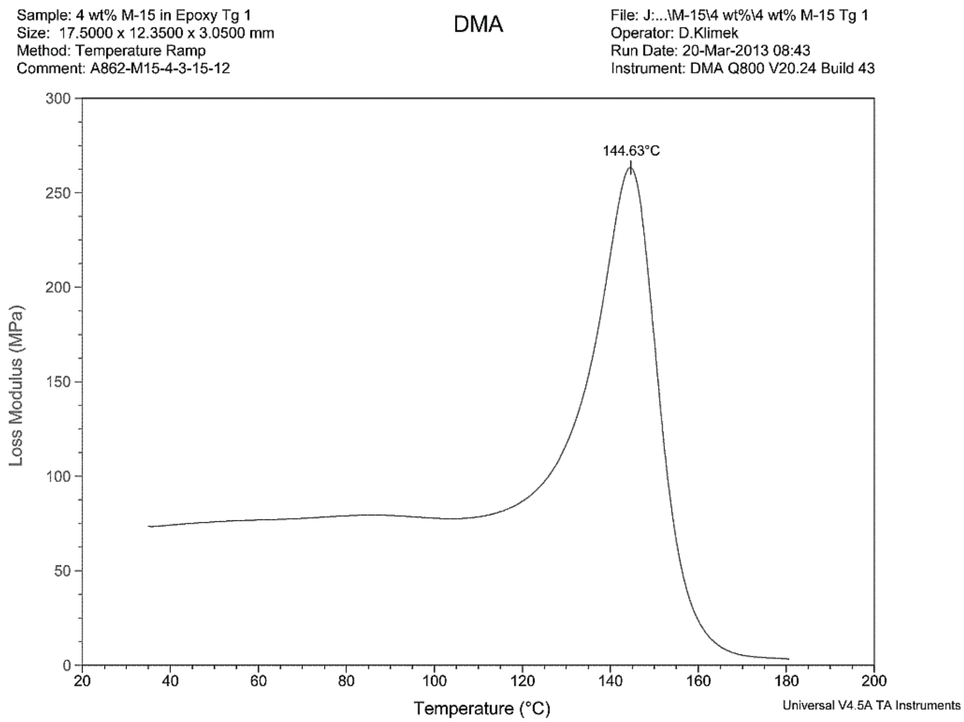


Figure D-14: Loss Modulus for 4 wt% xGnP®-M-15 in Epoxy Test 1

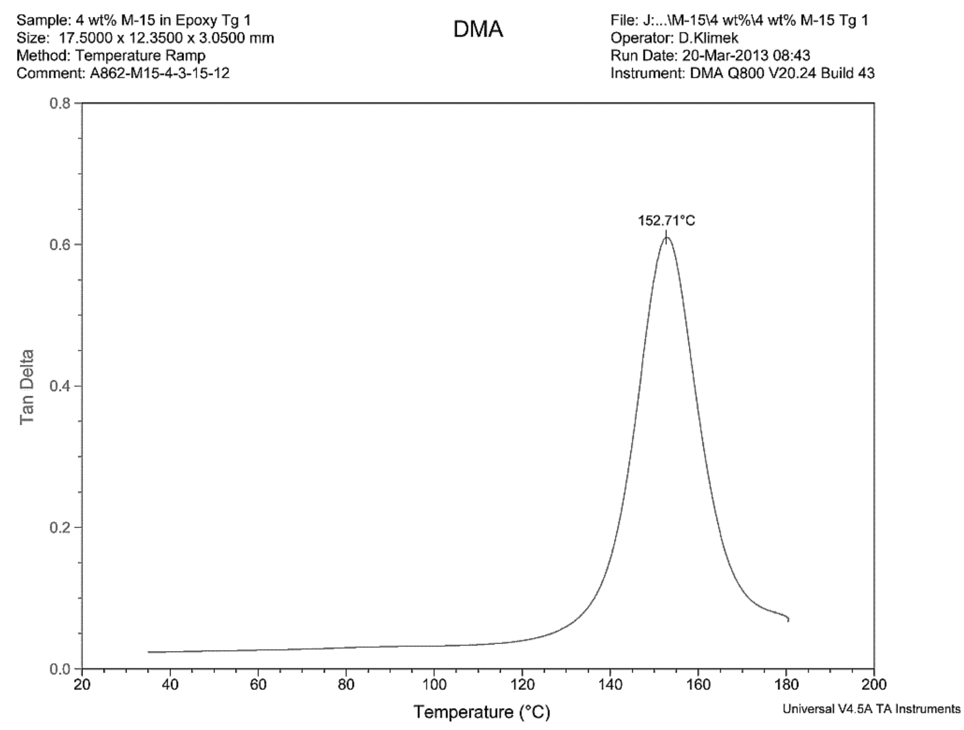


Figure D-15: Tan Delta for 4 wt% xGnP®-M-15 in Epoxy Test 1

Table D-6: DMA Results for 5 wt% xGnP®-M-15 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M15-5-3-19-12	1	137.15	149.59	156.27
A862-M15-5-3-19-12	2	136.60	149.05	156.04
A862-M15-5-3-19-12	3	137.13	148.75	155.57
Average		**137.0**	**149.1**	**156.0**
Standard Deviation		**0.3**	**0.4**	**0.4**
Count		**3**	**3**	**3**

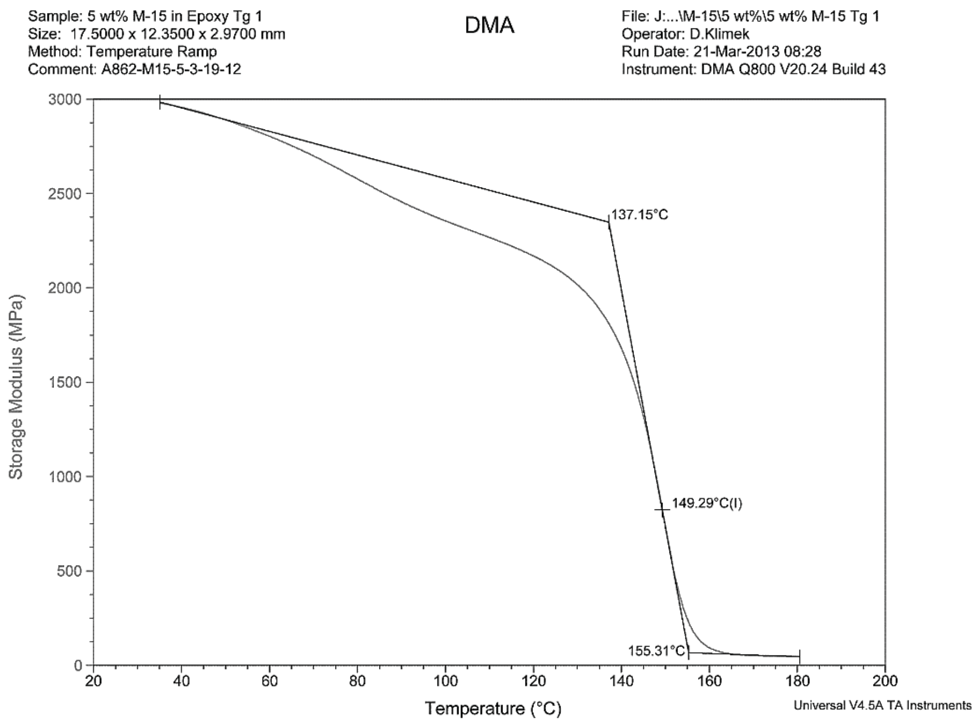


Figure D-16: Storage Modulus for 5 wt% xGnP®-M-15 in Epoxy Test 1

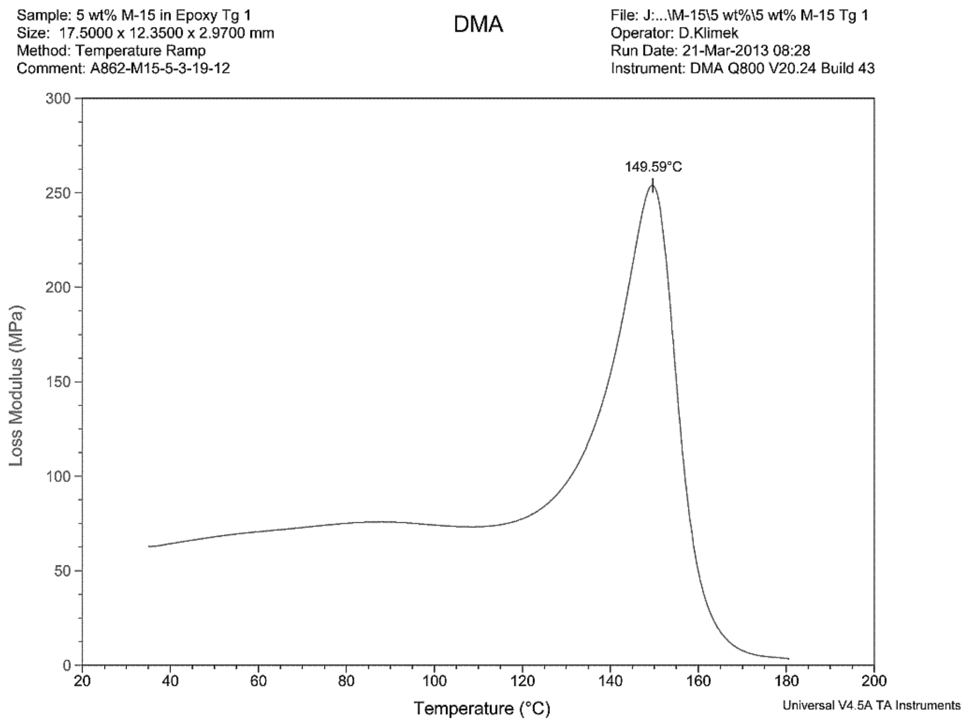


Figure D-17: Loss Modulus for 5 wt% xGnP®-M-15 in Epoxy Test 1

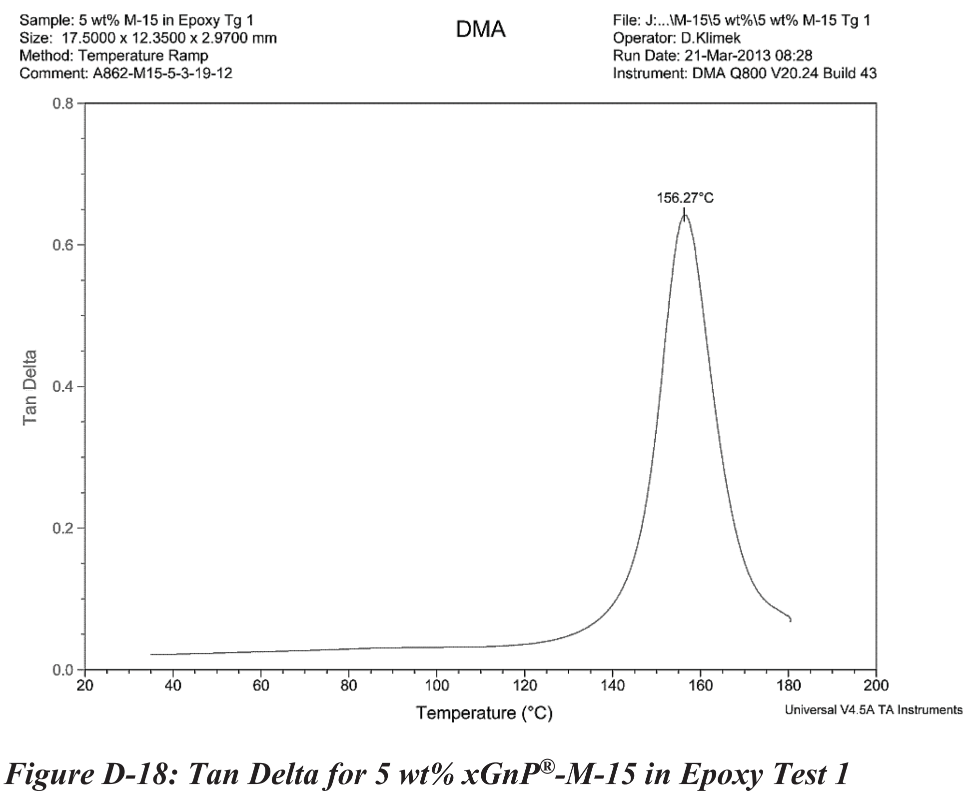


Figure D-18: Tan Delta for 5 wt% xGnP®-M-15 in Epoxy Test 1

Table D-7: DMA Results for 6 wt% xGnP®-M-15 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M15-6-3-28-12	1	136.34	148.60	155.92
A862-M15-6-3-28-12	2	134.71	147.38	155.20
A862-M15-6-3-28-12	3	135.61	149.02	156.54
A862-M15-6-4-19-12	4	137.37	148.75	156.11
A862-M15-6-4-19-12	5	136.19	148.31	155.83
Average		**136.0**	**148.4**	**155.9**
Standard Deviation		**1.0**	**0.6**	**0.5**
Count		**5**	**5**	**5**

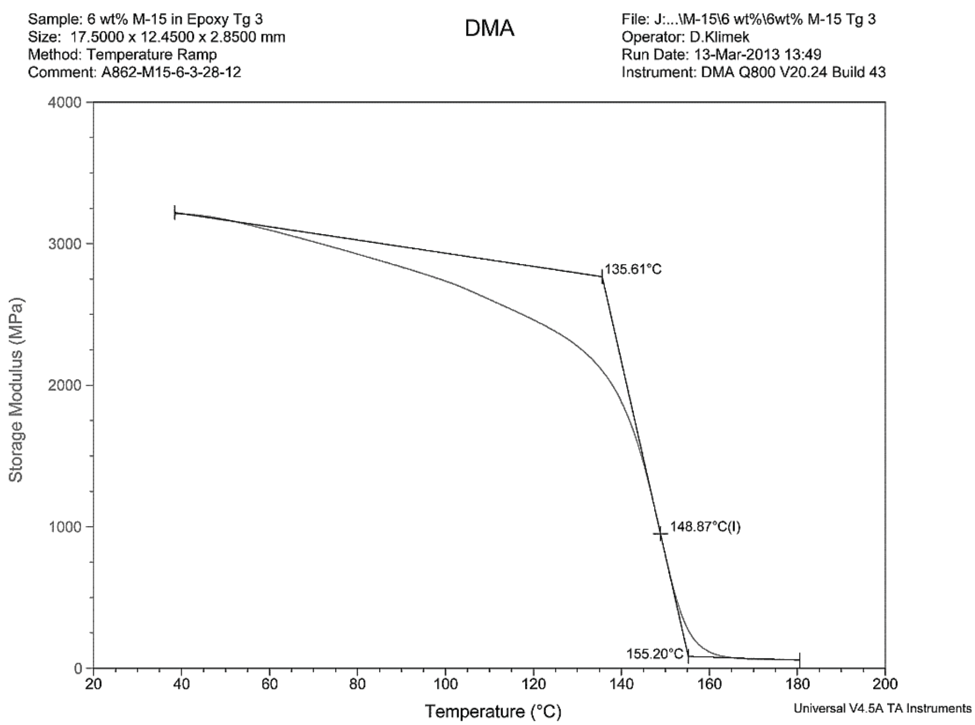


Figure D-19: Storage Modulus for 6 wt% xGnP®-M-15 in Epoxy Test 3

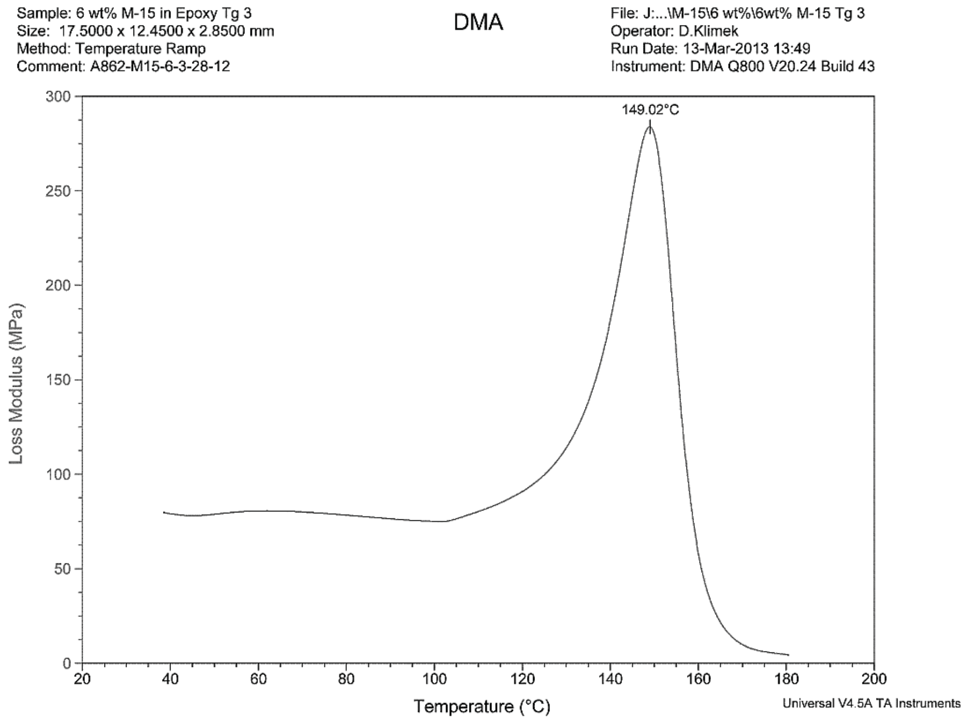


Figure D-20: Loss Modulus for 6 wt% xGnP®-M-15 in Epoxy Test 3

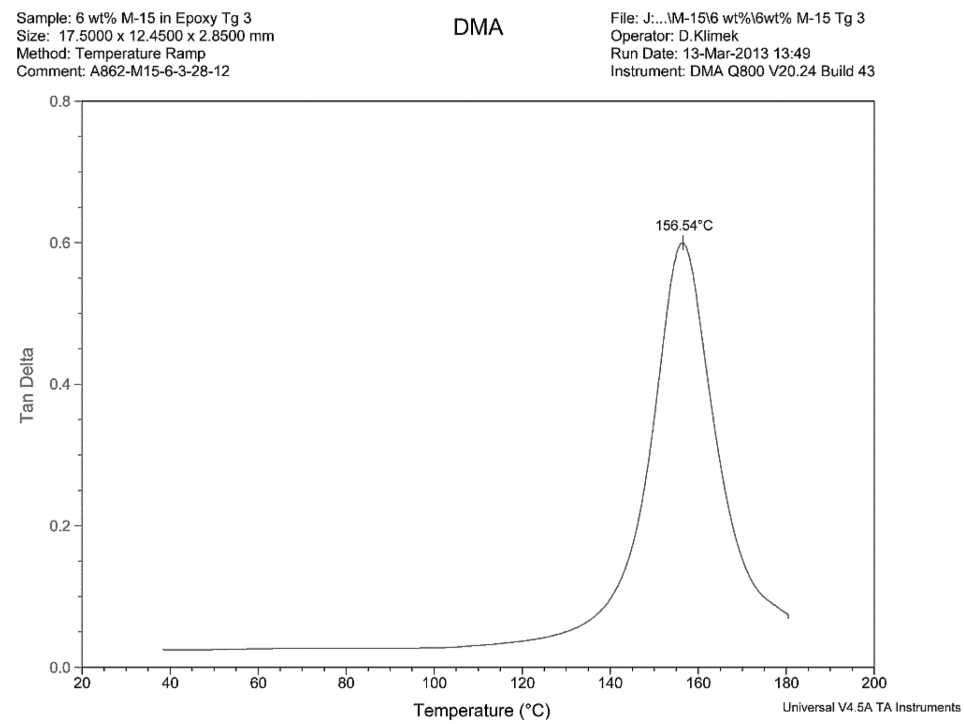


Figure D-21: Tan Delta for 6 wt% xGnP®-M-15 in Epoxy Test 3

Table D-8: DMA Results for 1 wt% xGnP®-M-5 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M5-1-9-28-12	1	136.19	147.80	154.59
A862-M5-1-9-28-12	2	135.77	147.58	154.17
A862-M5-1-9-28-12	3	136.61	147.29	154.05
Average		**136.2**	**147.6**	**154.3**
Standard Deviation		**0.4**	**0.3**	**0.3**
Count		**3**	**3**	**3**

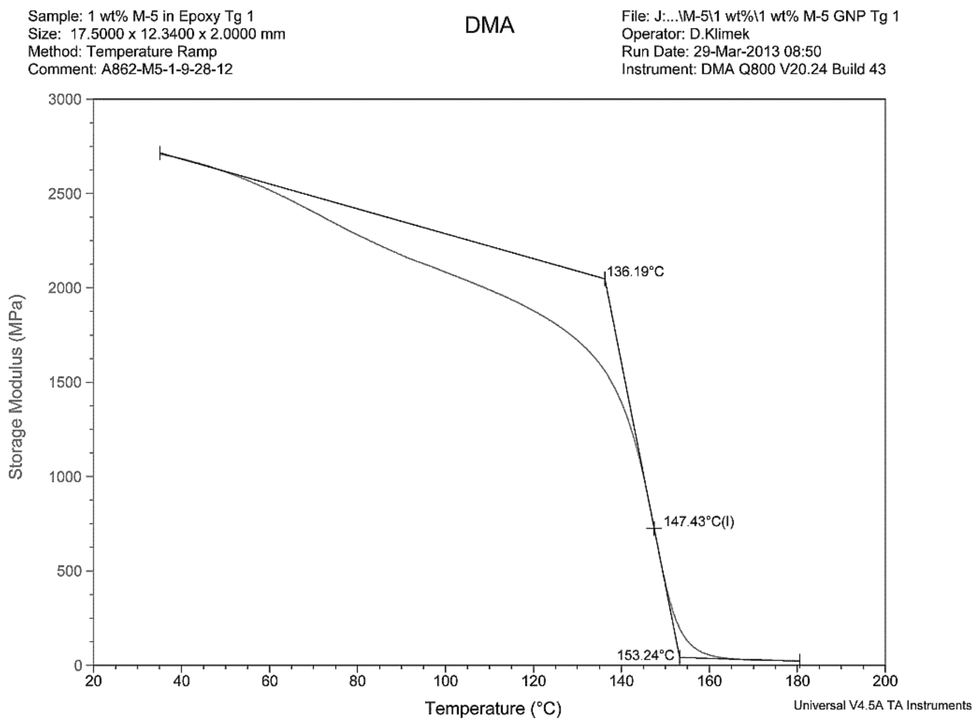


Figure D-22: Storage Modulus for 1 wt% xGnP®-M-5 in Epoxy Test 1

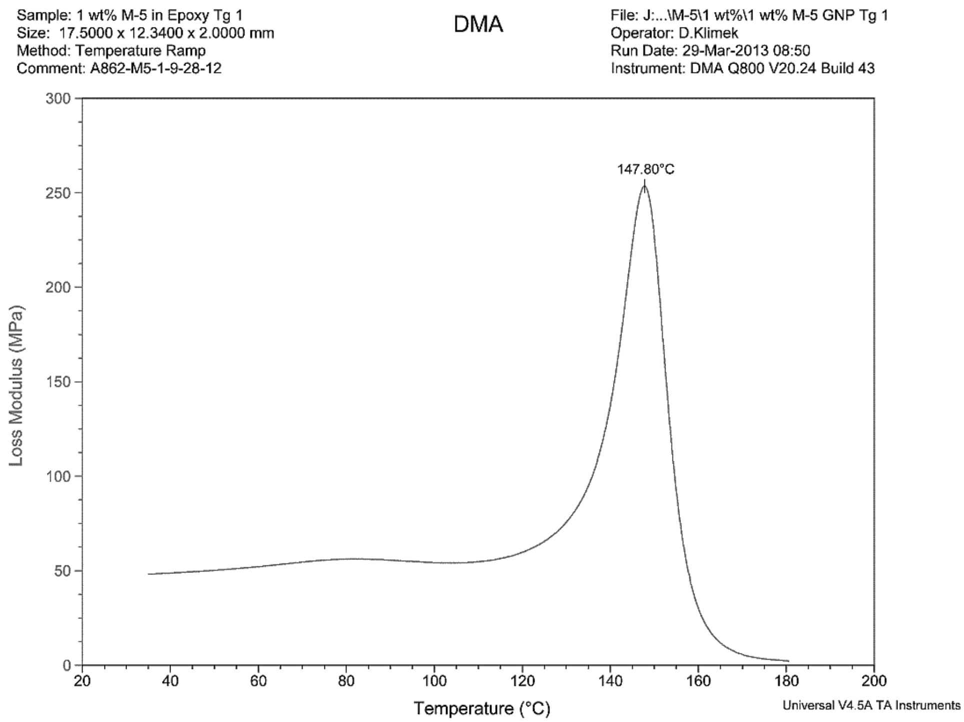


Figure D-23: Loss Modulus for 1 wt% xGnP®-M-5 in Epoxy Test 1

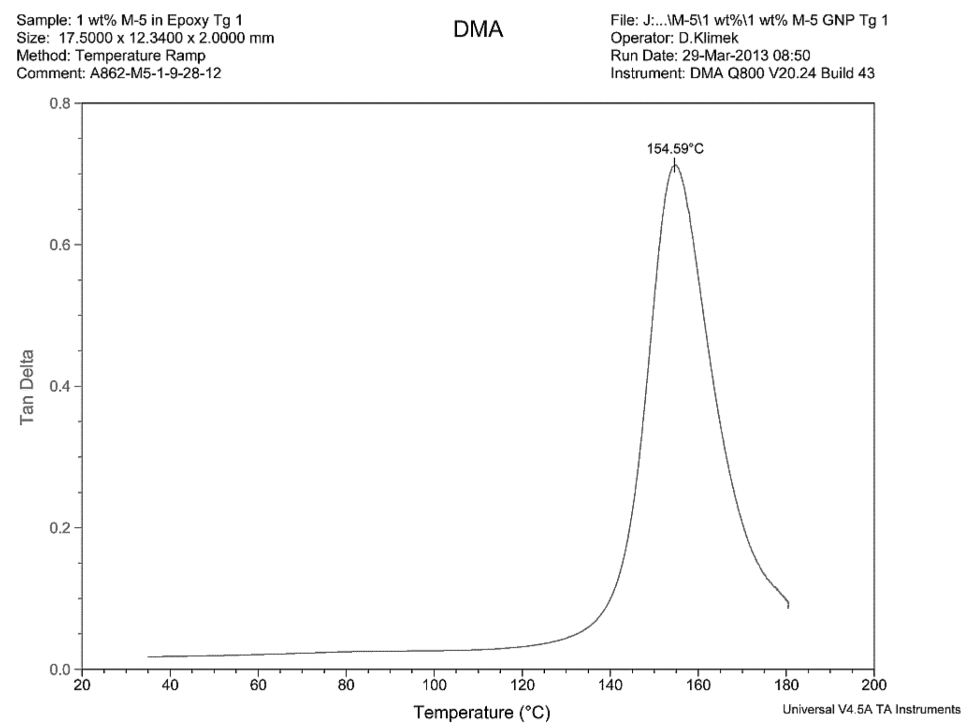


Figure D-24: Tan Delta for 1 wt% xGnP®-M-5 in Epoxy Test 1

Table D-9: DMA Results for 2 wt% xGnP®-M-5 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M5-2-8-28-12	1	134.99	146.04	153.59
A862-M5-2-8-28-12	2	134.15	145.82	153.01
A862-M5-2-8-28-12	3	134.90	145.58	152.82
Average		**134.7**	**145.8**	**153.1**
Standard Deviation		**0.5**	**0.2**	**0.4**
Count		**3**	**3**	**3**

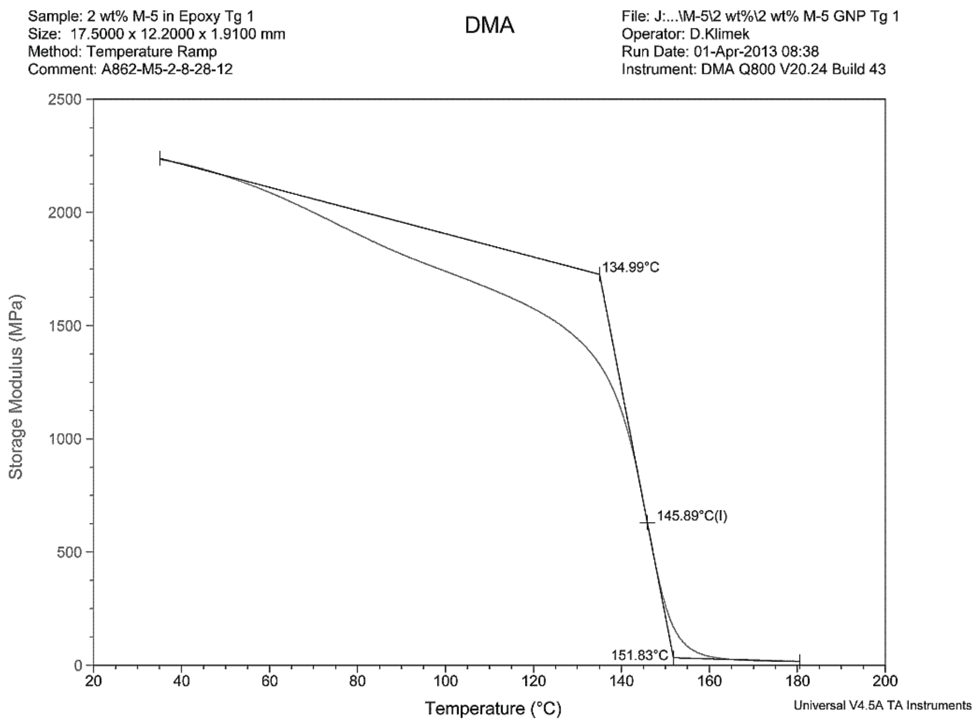


Figure D-25: Storage Modulus for 2 wt% xGnP®-M-5 in Epoxy Test 1

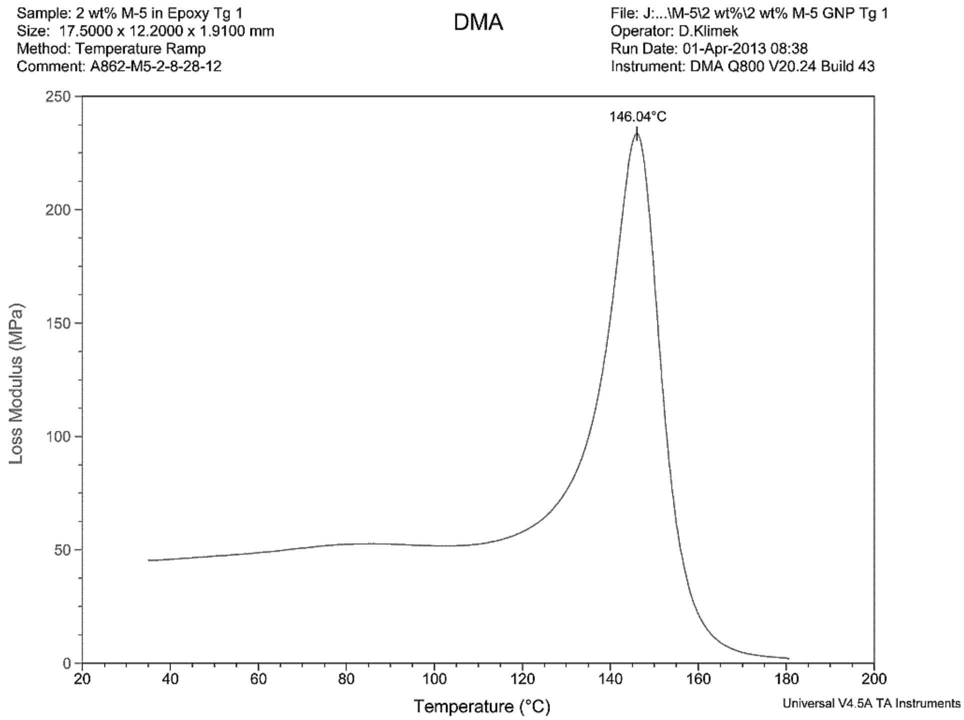


Figure D-26: Loss Modulus for 2 wt% xGnP®-M-5 in Epoxy Test 1

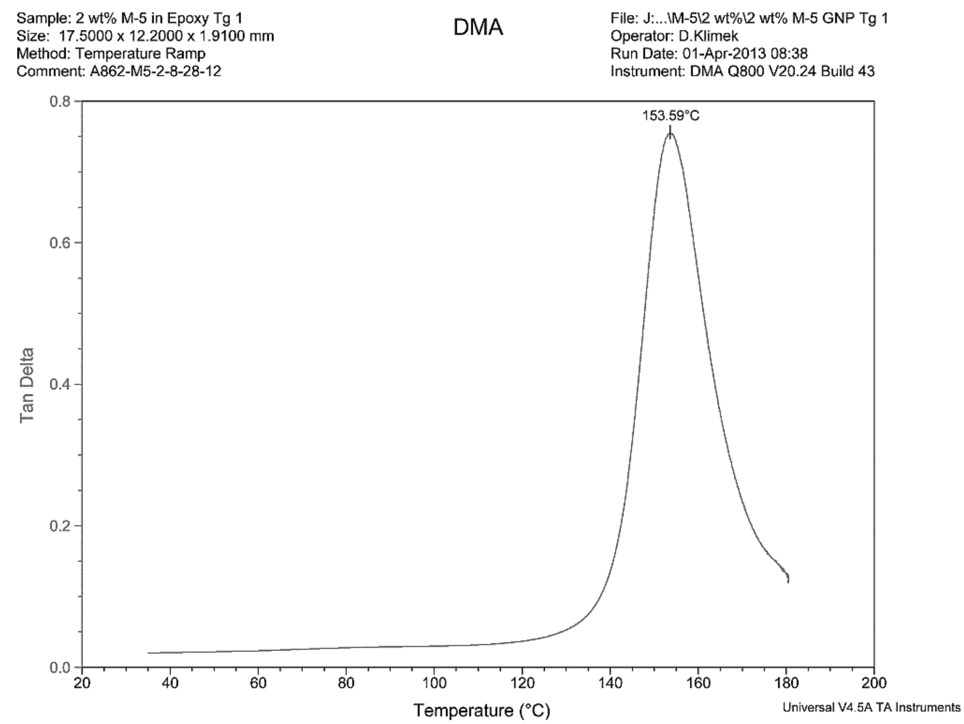


Figure D-27: Tan Delta for 2 wt% xGnP®-M-5 in Epoxy Test 1

Table D-10: DMA Results for 3 wt% xGnP®-M-5 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M5-3-10-3-12	1	137.17	147.87	154.76
A862-M5-3-10-3-12	2	137.53	147.78	154.34
A862-M5-3-10-3-12	3	137.57	148.51	154.93
Average		**137.4**	**148.1**	**154.7**
Standard Deviation		**0.2**	**0.4**	**0.3**
Count		**3**	**3**	**3**

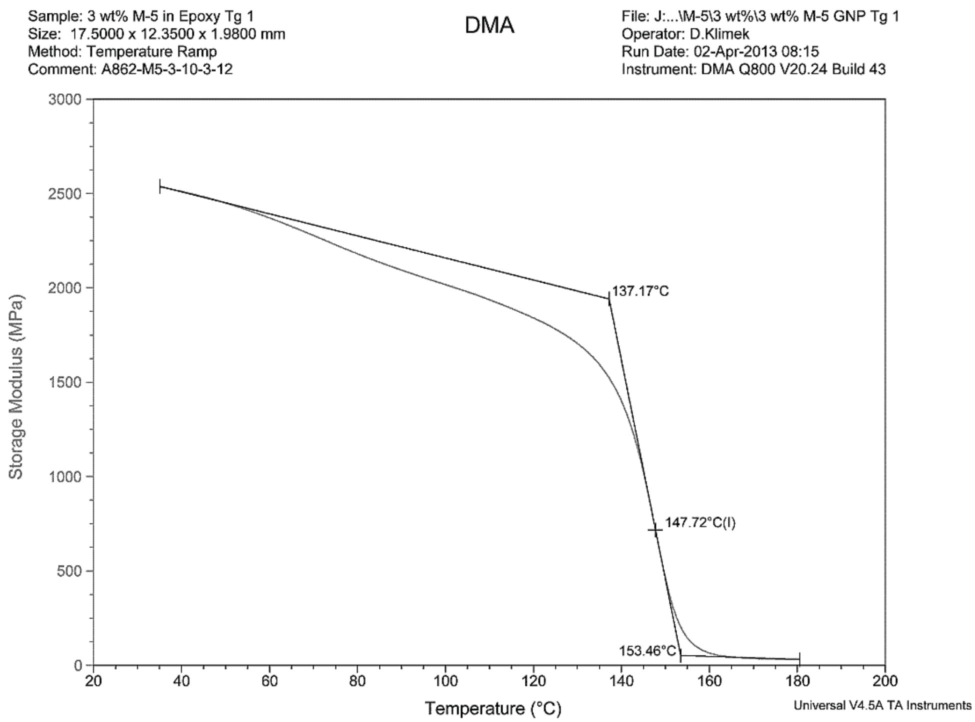


Figure D-28: Storage Modulus for 3 wt% xGnP®-M-5 in Epoxy Test 1

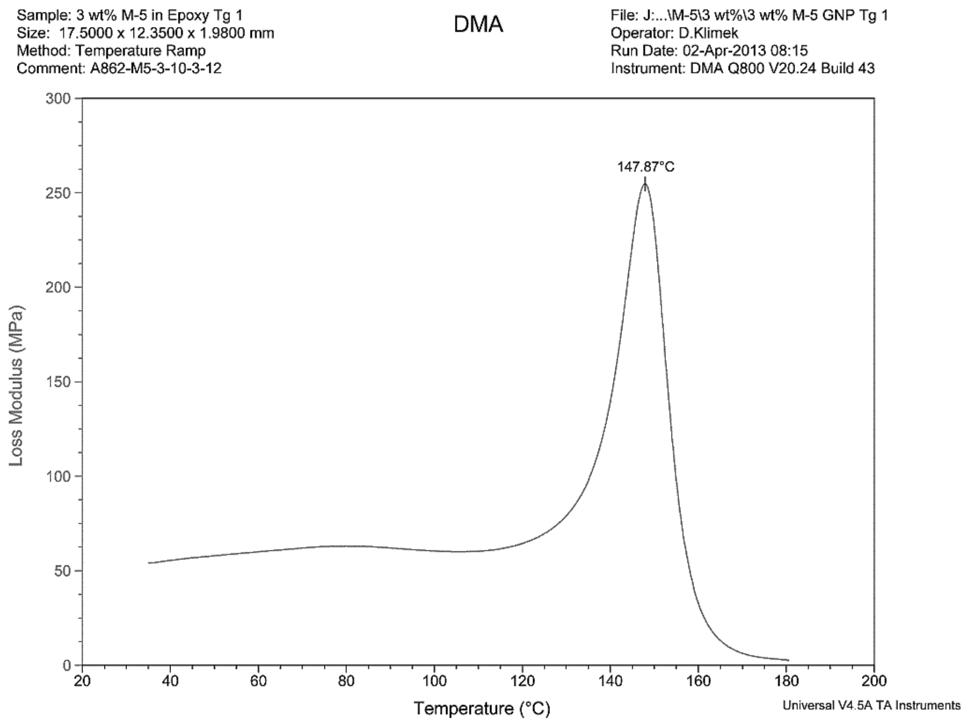


Figure D-29: Loss Modulus for 3 wt% xGnP®-M-5 in Epoxy Test 1.

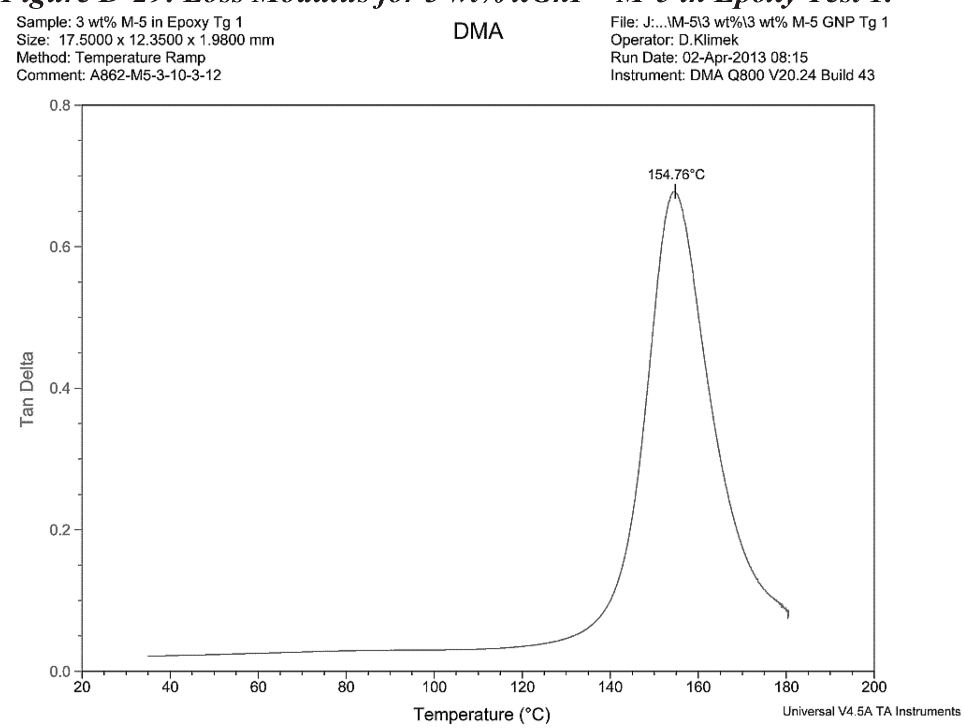


Figure D-30: Tan Delta for 3 wt% xGnP®-M-5 in Epoxy Test 1

Table D-11: DMA Results for 4 wt% xGnP®-M-5 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M5-4-10-9-12	1	139.66	150.42	156.82
A862-M5-4-10-9-12	2	139.34	150.00	156.58
A862-M5-4-10-9-12	3	139.92	149.50	156.09
Average		**139.6**	**150.0**	**156.5**
Standard Deviation		**0.3**	**0.5**	**0.4**
Count		**3**	**3**	**3**

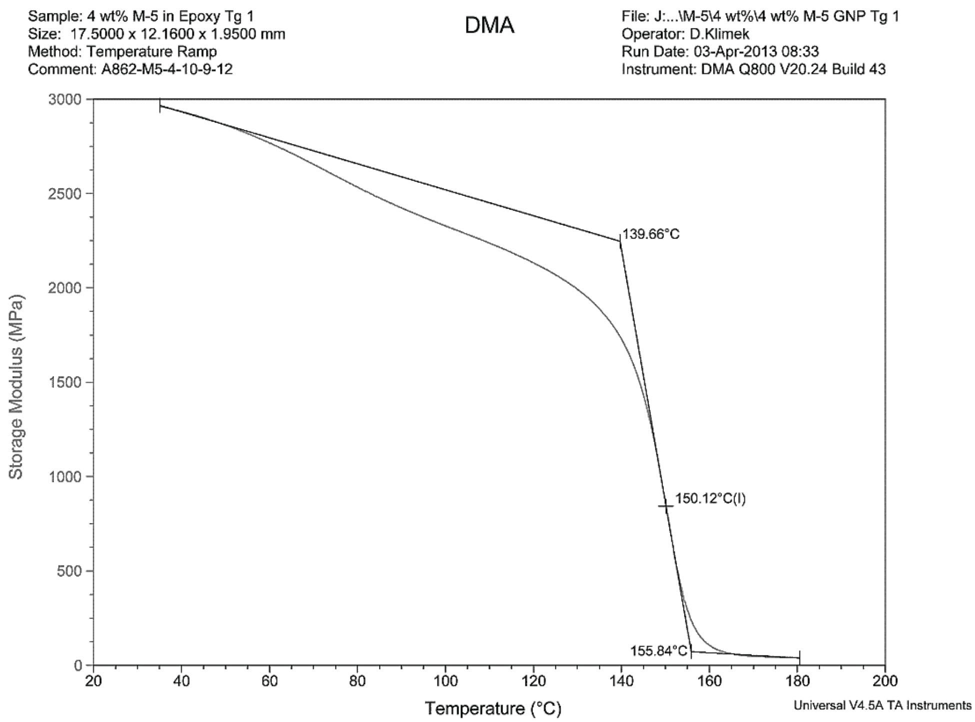


Figure D-31: Storage Modulus for 4 wt% xGnP®-M-5 in Epoxy Test 1

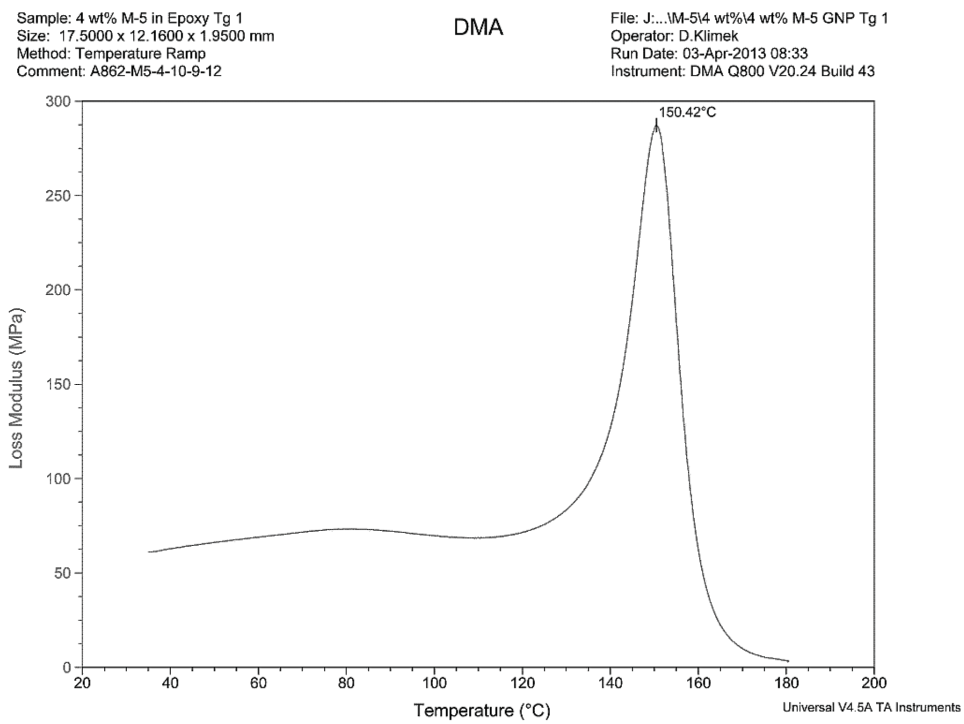


Figure D-32: Loss Modulus for 4 wt% xGnP®-M-5 in Epoxy Test 1

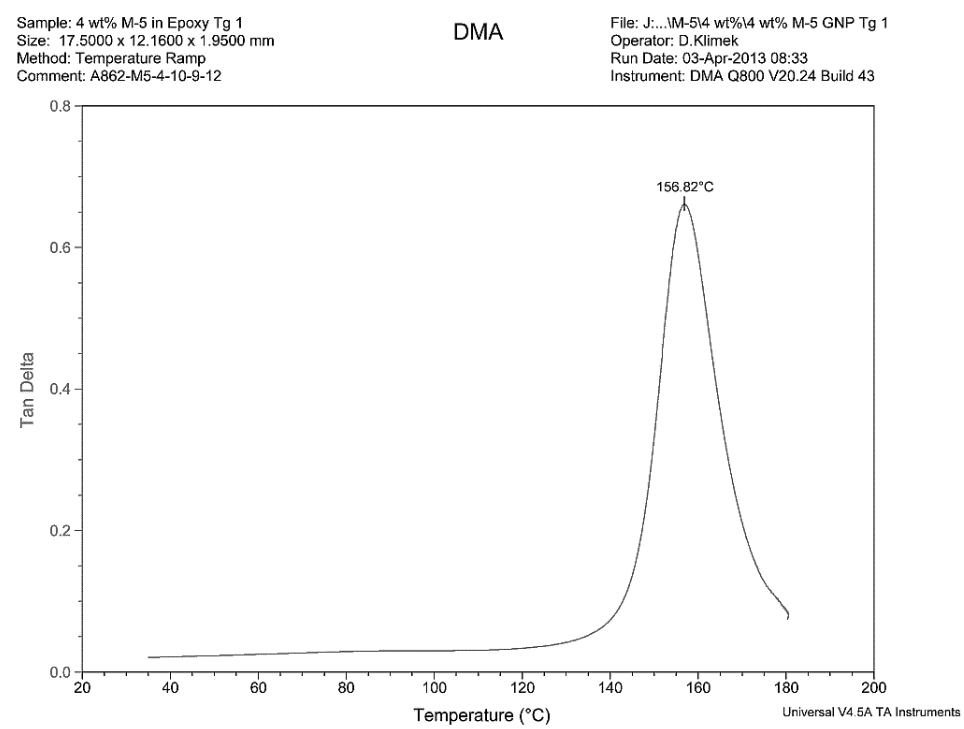


Figure D-33: Tan Delta for 4 wt% xGnP®-M-5 in Epoxy Test 1

Table D-12: DMA Results for 5 wt% xGnP®-M-5 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M5-5-10-10-12	1	140.85	150.68	157.09
A862-M5-5-10-10-12	2	140.93	151.06	157.62
A862-M5-5-10-10-12	3	140.78	150.48	156.88
Average		**140.9**	**150.7**	**157.2**
Standard Deviation		**0.1**	**0.3**	**0.4**
Count		**3**	**3**	**3**

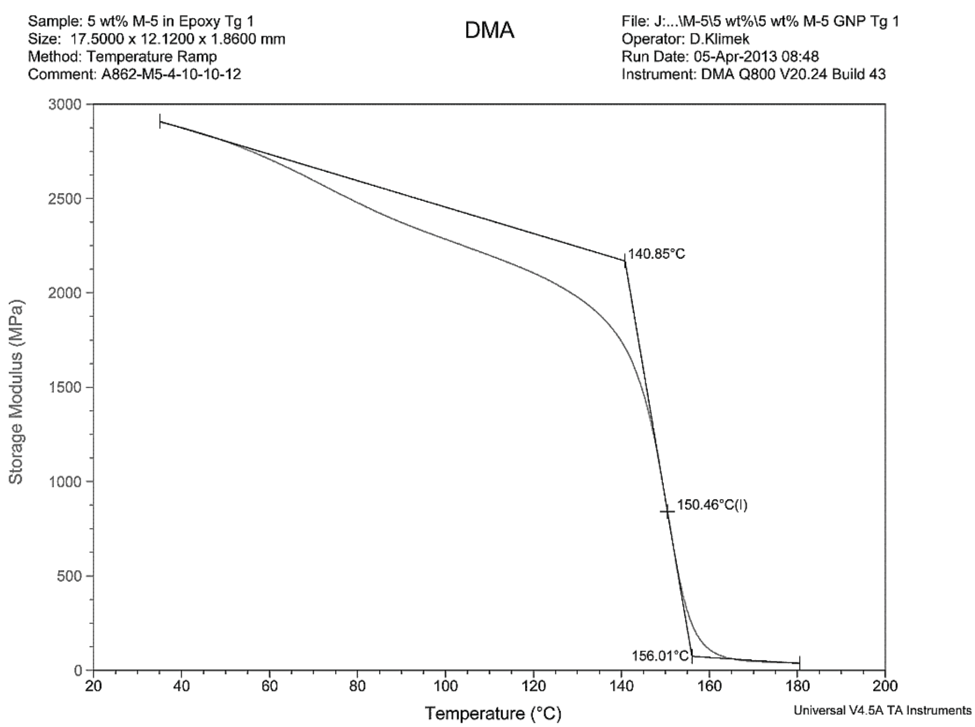


Figure D-34: Storage Modulus for 5 wt% xGnP®-M-5 in Epoxy Test 1

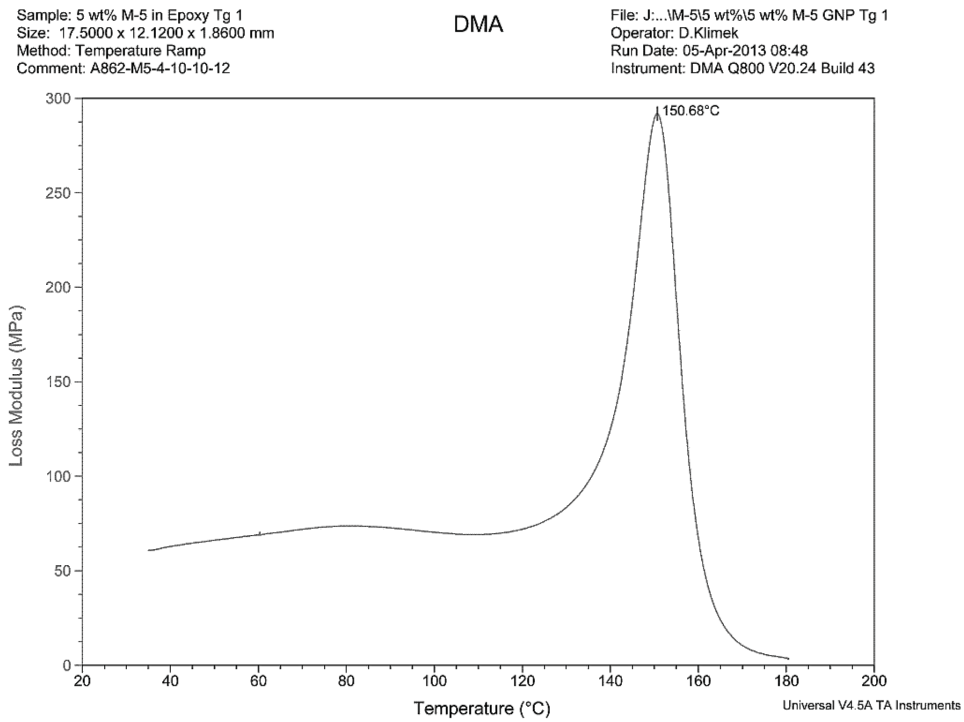


Figure D-35: Loss Modulus for 5 wt% xGnP®-M-5 in Epoxy Test 1

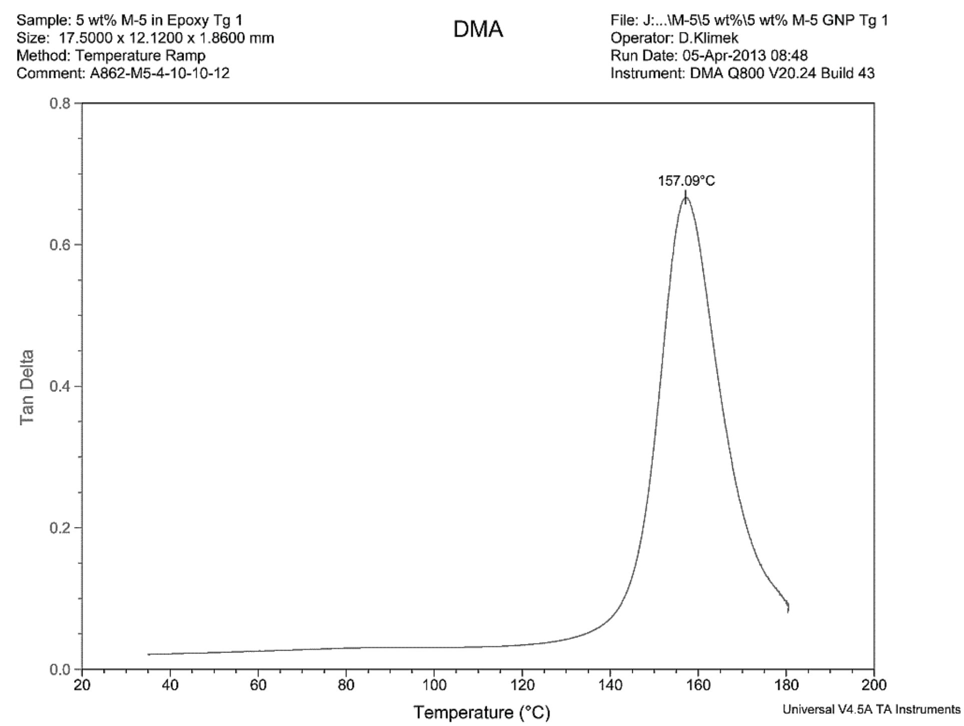


Figure D-36: Tan Delta for 5 wt% xGnP®-M-5 in Epoxy Test 1

Table D-13: DMA Results for 6 wt% xGnP®-M-5 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-M5-6-10-15-12	1	139.08	149.76	156.42
A862-M5-6-10-15-12	2	139.20	149.88	156.38
A862-M5-6-10-15-12	3	139.68	149.85	156.50
Average		**139.3**	**149.8**	**156.4**
Standard Deviation		**0.3**	**0.1**	**0.1**
Count		**3**	**3**	**3**

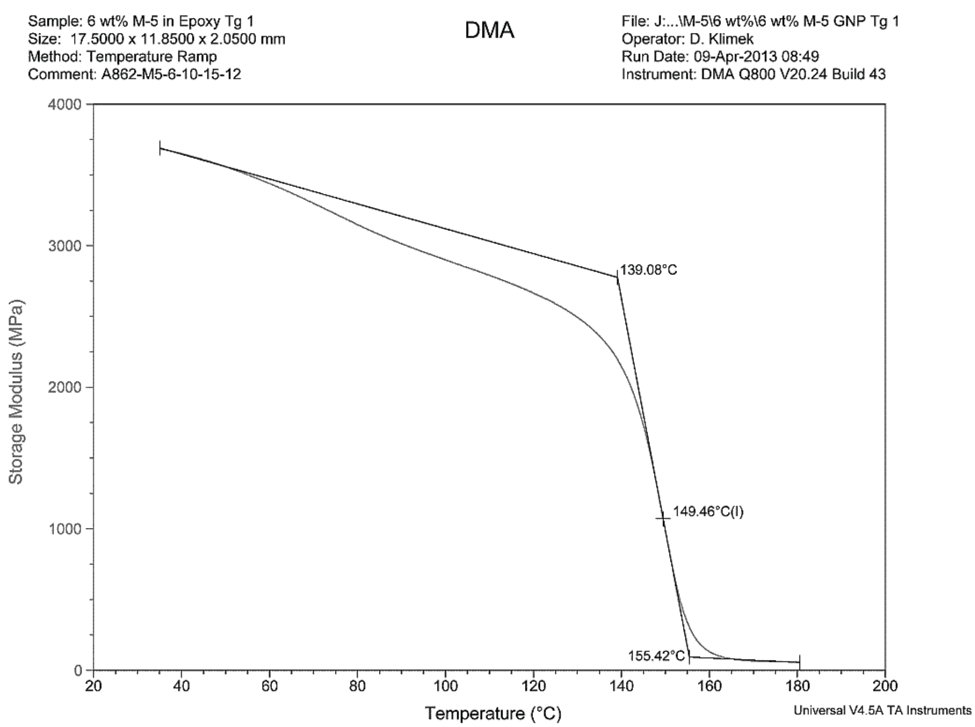


Figure D-37: Storage Modulus for 6 wt% xGnP®-M-5 in Epoxy Test 1

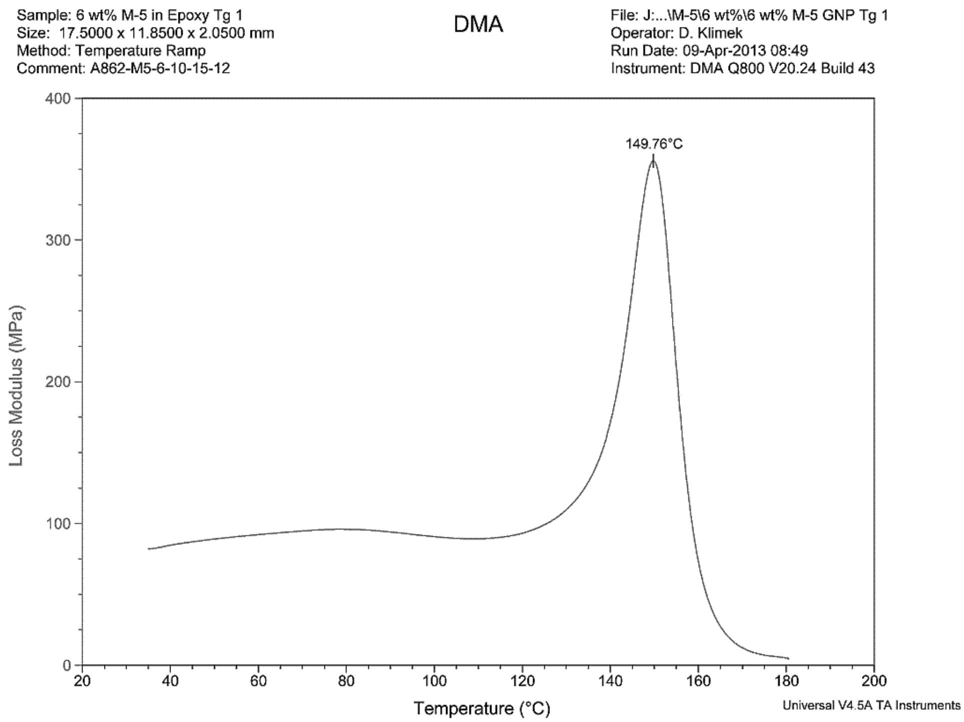


Figure D-38: Loss Modulus for 6 wt% xGnP®-M-5 in Epoxy Test 1

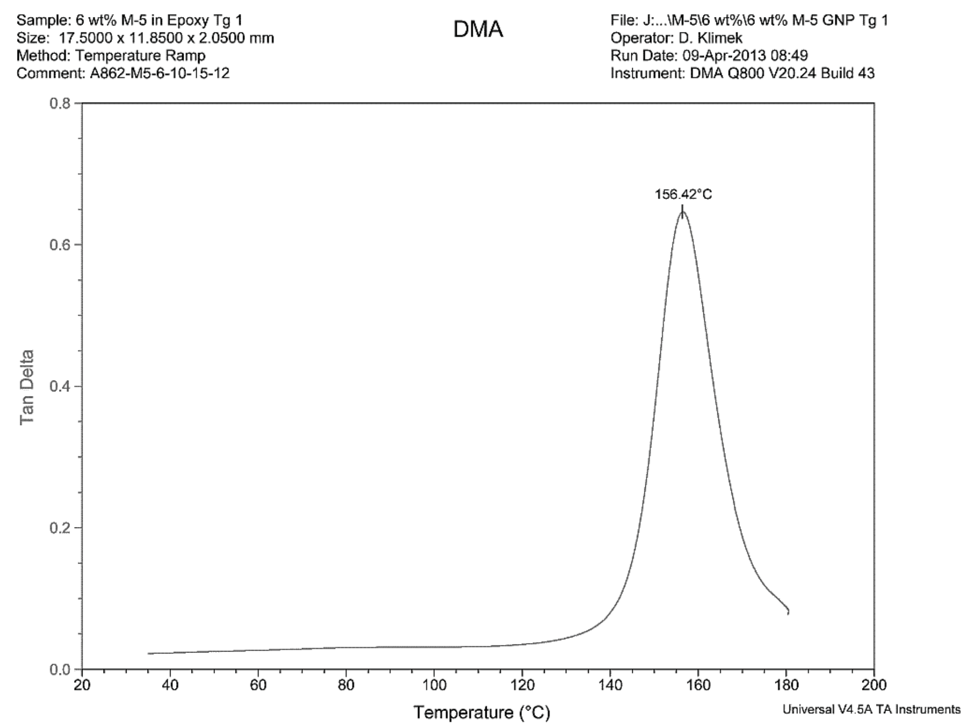


Figure D-39: Tan Delta for 6 wt% xGnP®-M-5 in Epoxy Test 1

Table D-14: DMA Results for 1 wt% xGnP®- C-300 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-C300-1-3-22-13	1	132.52	145.48	152.92
A862-C300-1-3-22-13	2	132.43	145.27	152.71
A862-C300-1-3-22-13	3	131.73	145.18	152.32
Average		**132.2**	**145.3**	**152.7**
Standard Deviation		**0.4**	**0.2**	**0.3**
Count		**3**	**3**	**3**

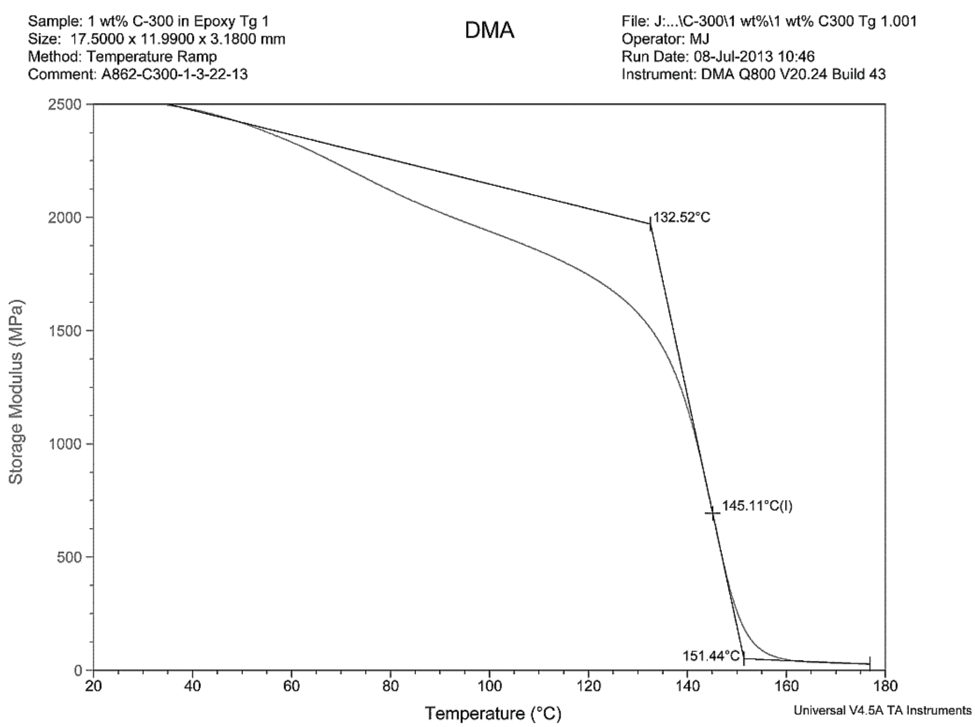


Figure D-40: Storage Modulus for 1 wt% xGnP®-C-300 in Epoxy Test 1

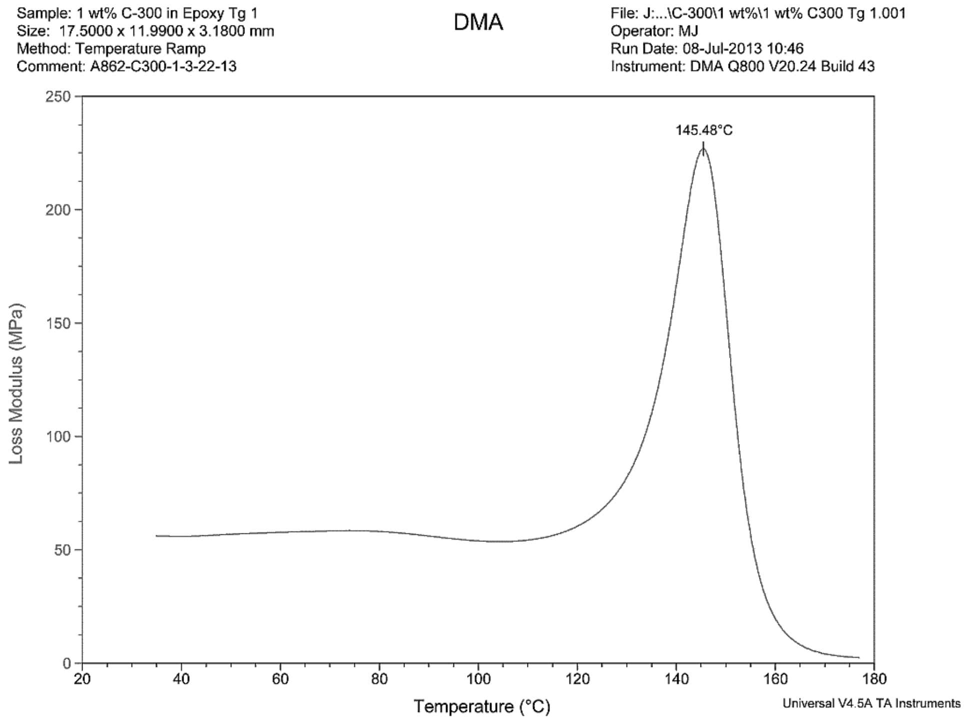


Figure D-41: Loss Modulus for 1 wt% xGnP®- C-300 in Epoxy Test 1

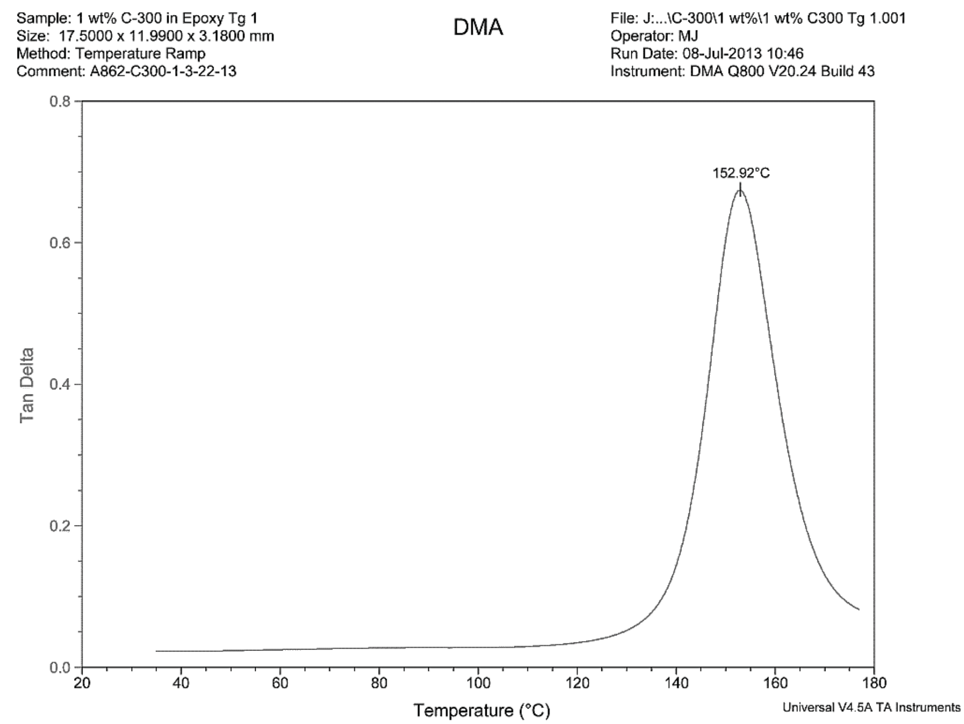


Figure D-42: Tan Delta for 1 wt% xGnP®- C-300 in Epoxy Test 1

Table D-15: DMA Results for 2 wt% xGnP®- C-300 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-C300-2-3-7-13	1	120.45	134.87	143.52
A862-C300-2-3-7-13	2	120.21	134.86	143.50
A862-C300-2-3-7-13	3	121.11	134.67	143.13
Average		**120.6**	**134.8**	**143.4**
Standard Deviation		**0.5**	**0.1**	**0.2**
Count		**3**	**3**	**3**

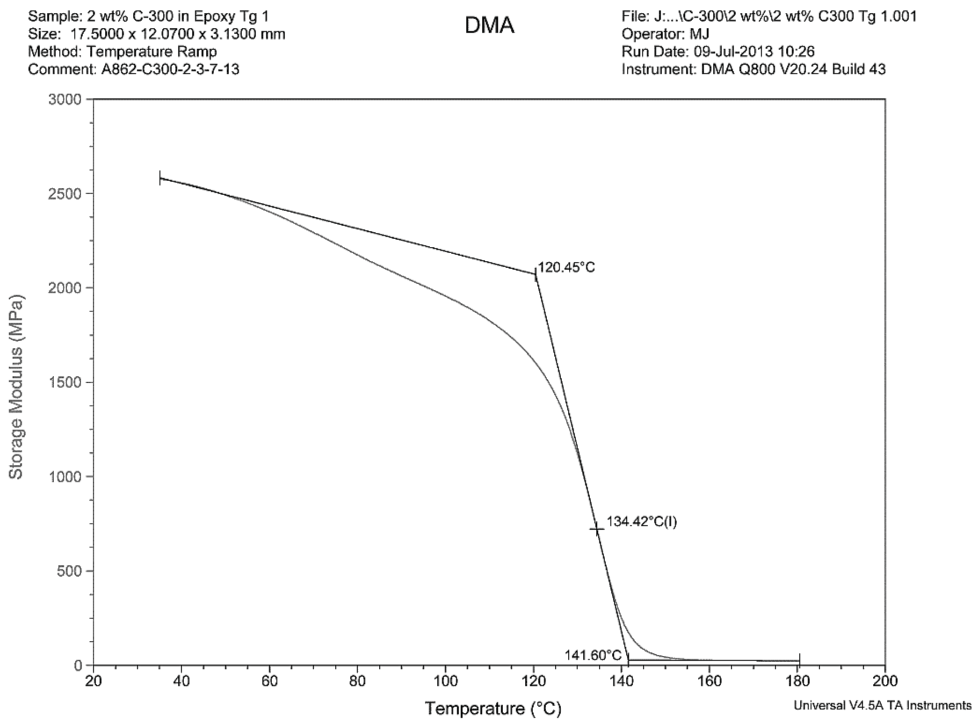


Figure D-43: Storage Modulus for 2 wt% xGnP®-C-300 in Epoxy Test 1

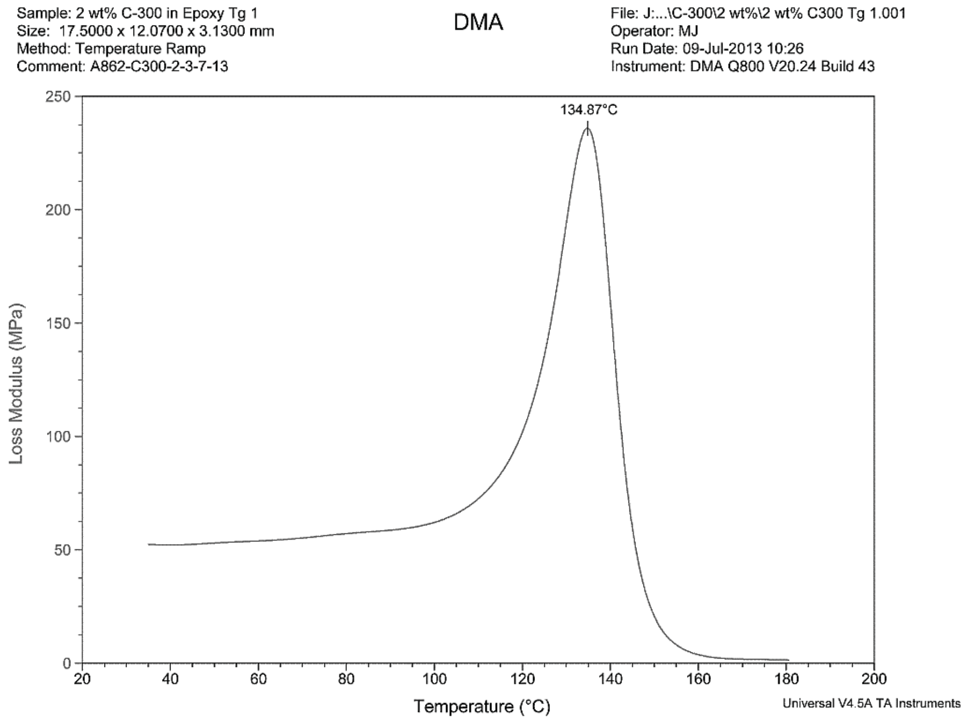


Figure D-44: Loss Modulus for 2 wt% xGnP®- C-300 in Epoxy Test 1

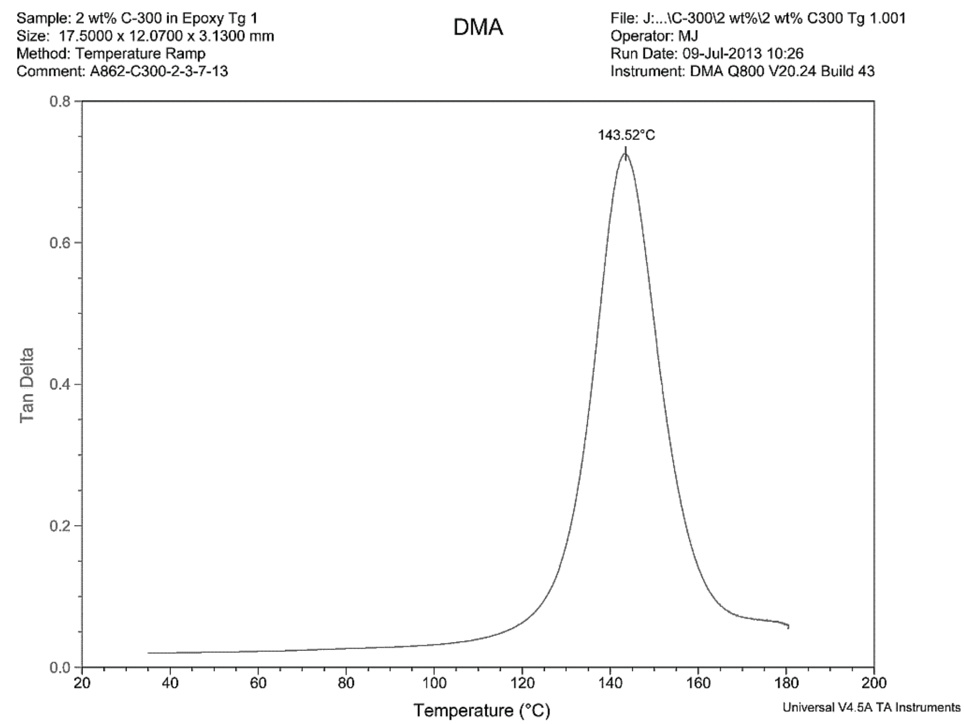


Figure D-45: Tan Delta for 2 wt% xGnP®- C-300 in Epoxy Test 1

Table D-16: DMA Results for 3 wt% xGnP®- C-300 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-C300-3-3-5-13	1	132.03	145.15	152.54
A862-C300-3-3-5-13	2	132.33	144.88	152.47
A862-C300-3-3-5-13	3	133.17	145.12	153.16
Average		**132.5**	**145.1**	**152.7**
Standard Deviation		**0.6**	**0.1**	**0.4**
Count		**3**	**3**	**3**

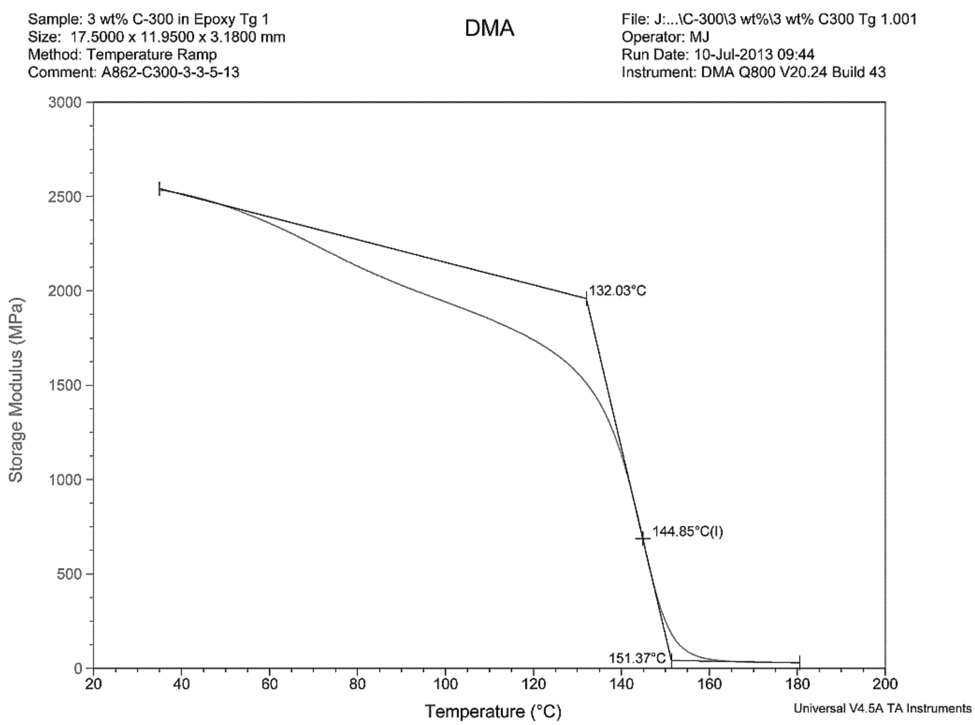


Figure D-46: Storage Modulus for 3 wt% xGnP®-C-300 in Epoxy Test 1

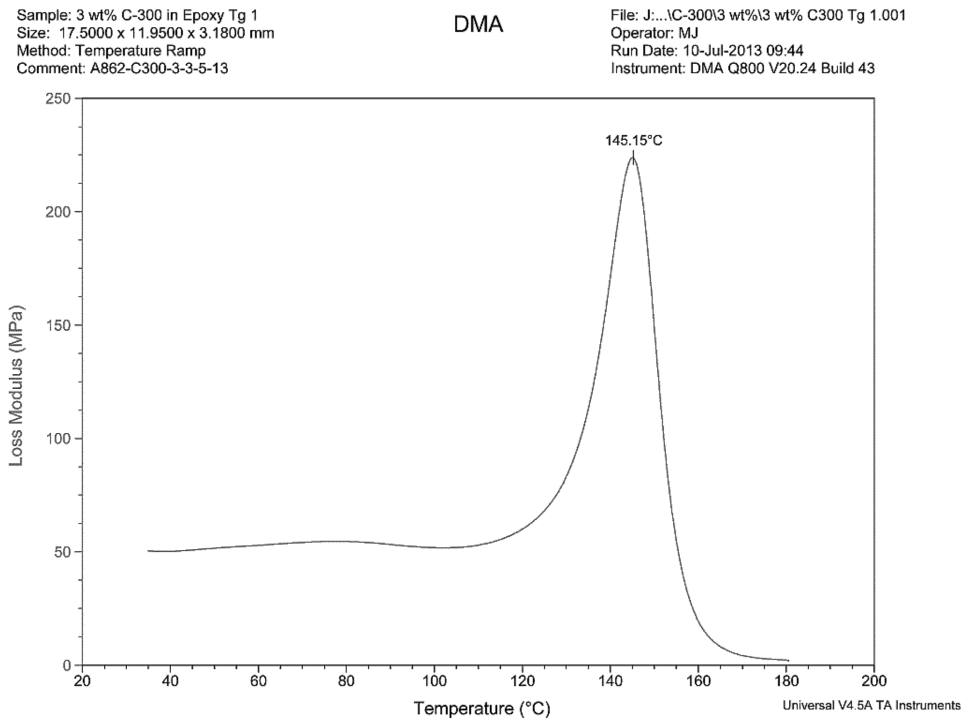


Figure D-47: Loss Modulus for 3 wt% xGnP®- C-300 in Epoxy Test 1

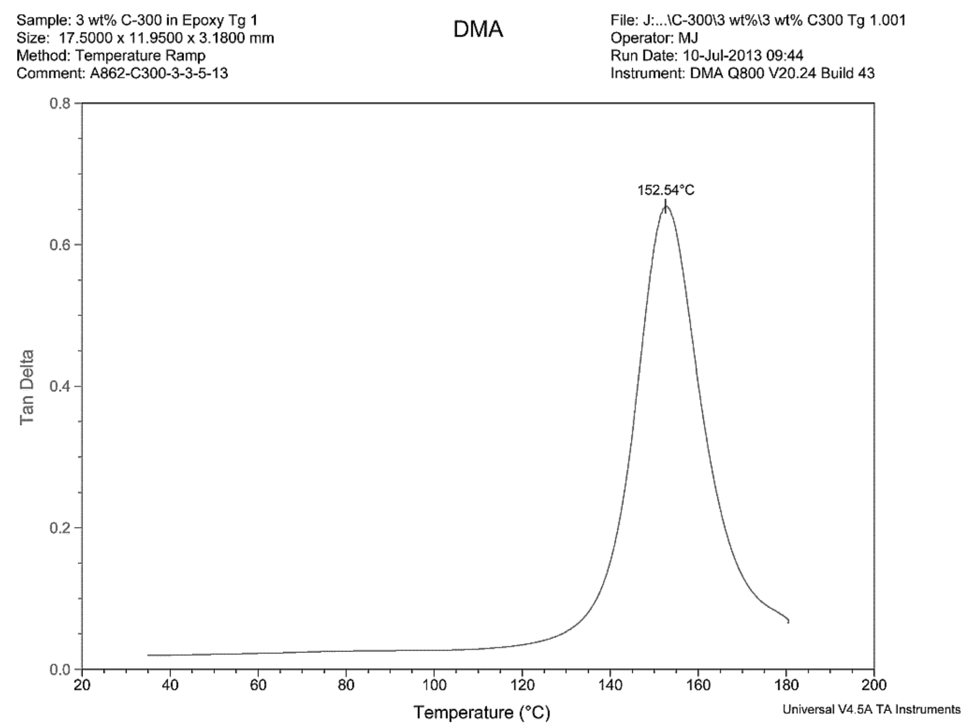


Figure D-48: Tan Delta for 3 wt% xGnP®- C-300 in Epoxy Test 1

Table D-17: DMA Results for 4 wt% xGnP®- C-300 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-C300-4-3-28-13	1	134.39	147.20	154.60
A862-C300-4-3-28-13	2	134.56	147.22	154.76
A862-C300-4-3-28-13	3	135.97	147.69	155.23
Average		**135.0**	**147.4**	**154.9**
Standard Deviation		**0.9**	**0.3**	**0.3**
Count		**3**	**3**	**3**

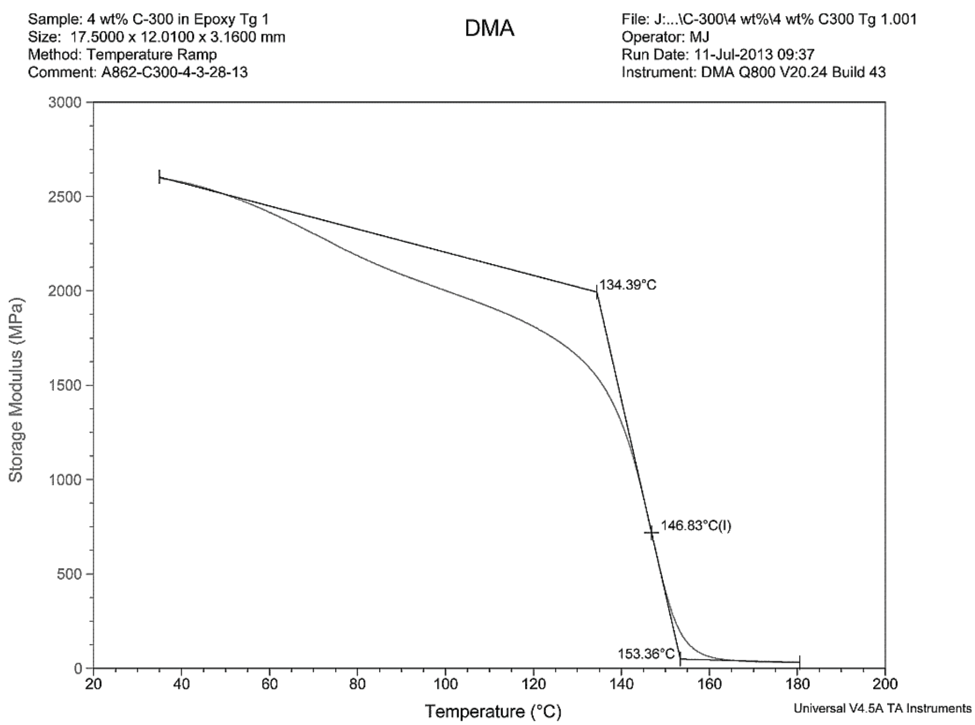


Figure D-49: Storage Modulus for 4 wt% xGnP®-C-300 in Epoxy Test 1

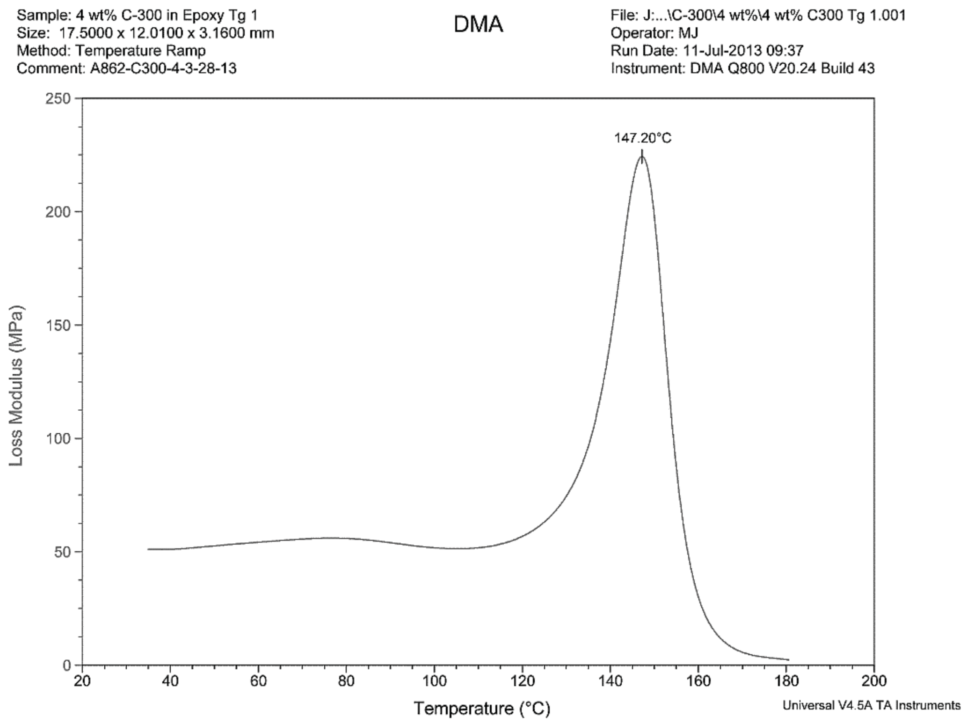


Figure D-50: Loss Modulus for 4 wt% xGnP®- C-300 in Epoxy Test 1

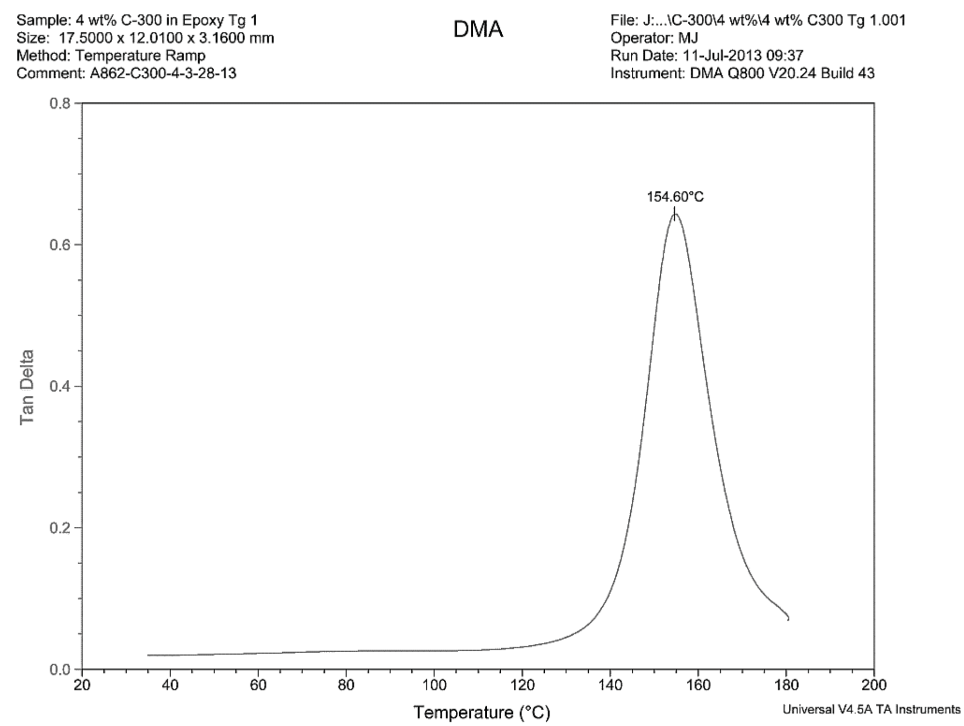


Figure D-51: Tan Delta for 4 wt% xGnP®- C-300 in Epoxy Test 1

Table D-18: DMA Results for 5 wt% xGnP®- C-300 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-C300-5-4-1-13	1	133.13	145.77	153.52
A862-C300-5-4-1-13	2	132.29	145.49	153.02
A862-C300-5-4-1-13	3	133.58	145.61	153.45
Average		**133.0**	**145.6**	**153.3**
Standard Deviation		**0.7**	**0.1**	**0.3**
Count		**3**	**3**	**3**

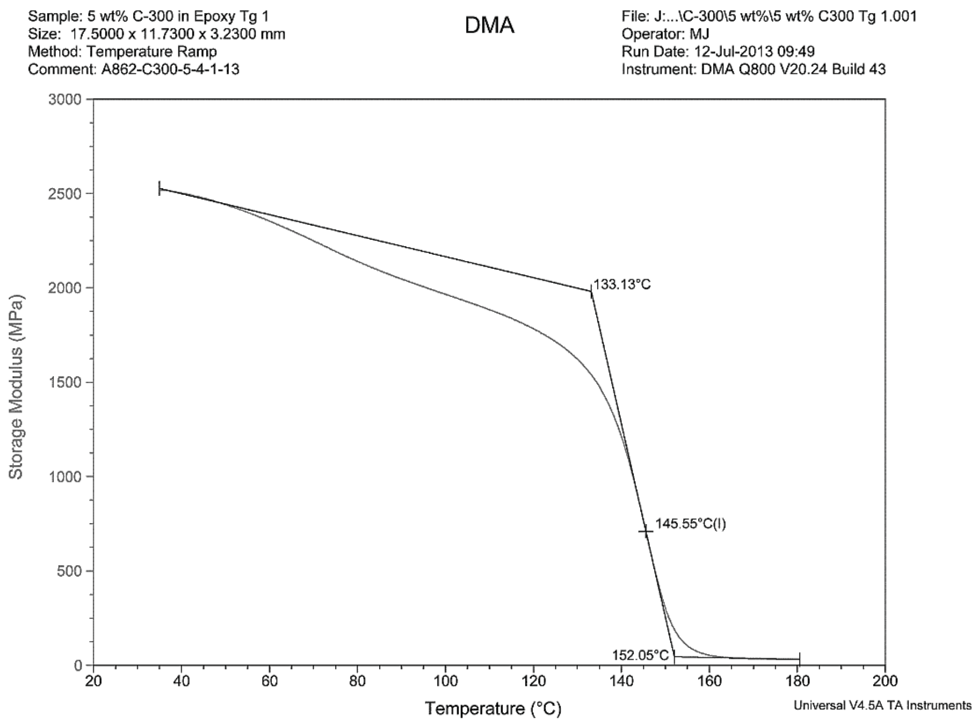


Figure D-52: Storage Modulus for 5 wt% xGnP®-C-300 in Epoxy Test 1

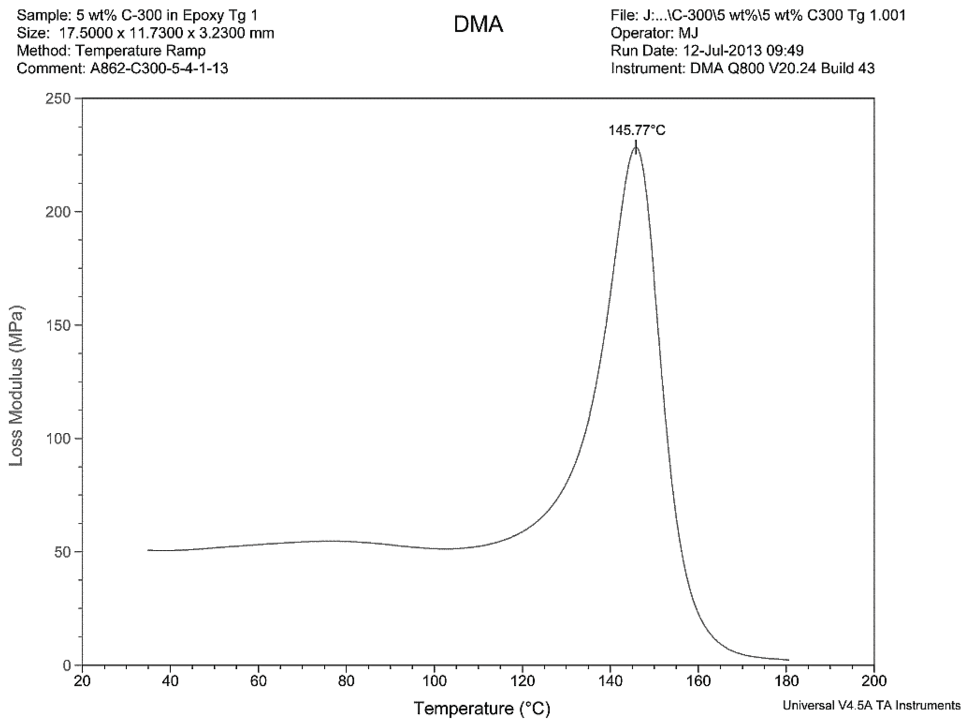


Figure D-53: Loss Modulus for 5 wt% xGnP®- C-300 in Epoxy Test 1

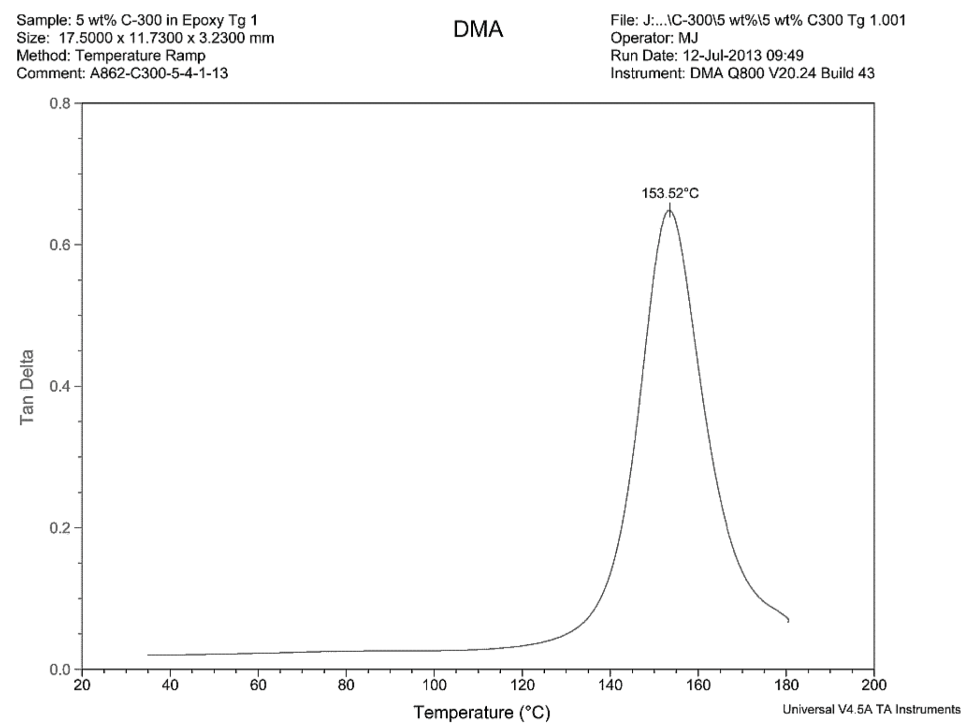


Figure D-54: Tan Delta for 5 wt% xGnP®- C-300 in Epoxy Test 1

Table D-19: DMA Results for 6 wt% xGnP®- C-300 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-C300-6-4-1-13	1	132.28	145.33	153.12
A862-C300-6-4-1-13	2	132.98	145.43	153.49
A862-C300-6-4-1-13	3	133.23	145.32	153.54
Average		**132.8**	**145.4**	**153.4**
Standard Deviation		**0.5**	**0.1**	**0.2**
Count		**3**	**3**	**3**

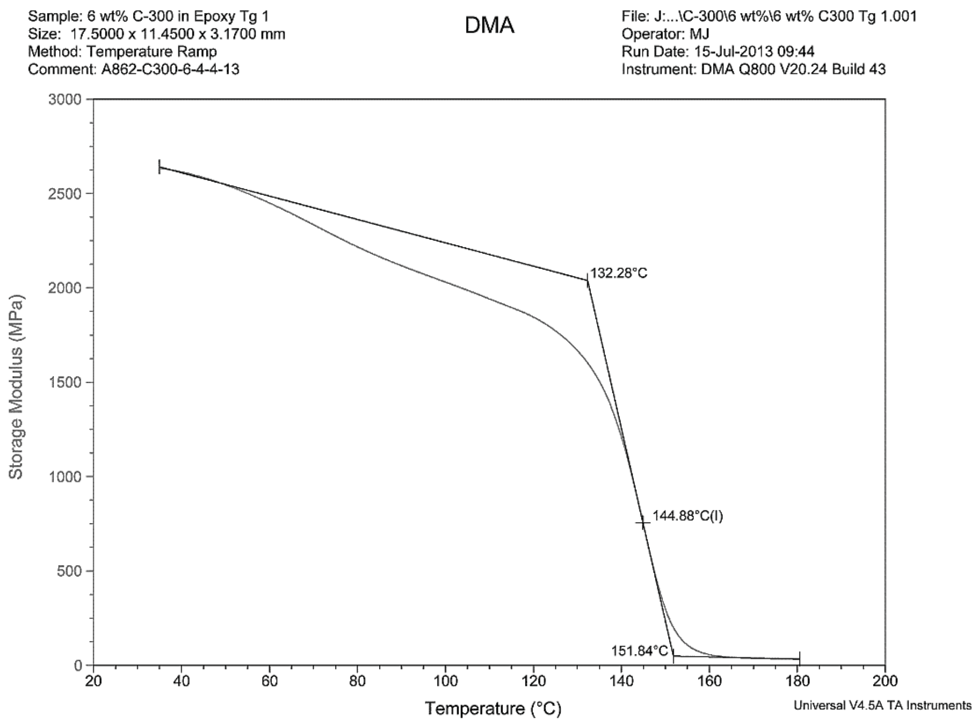


Figure D-55: Storage Modulus for 6 wt% xGnP®-C-300 in Epoxy Test 1

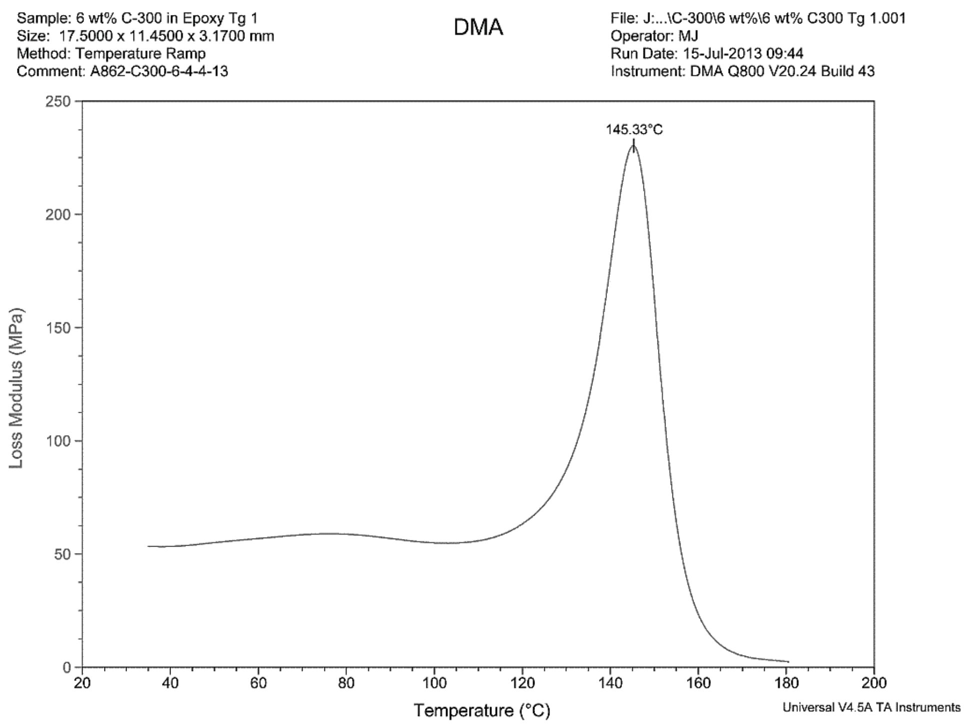


Figure D-56: Loss Modulus for 6 wt% xGnP®- C-300 in Epoxy Test 1

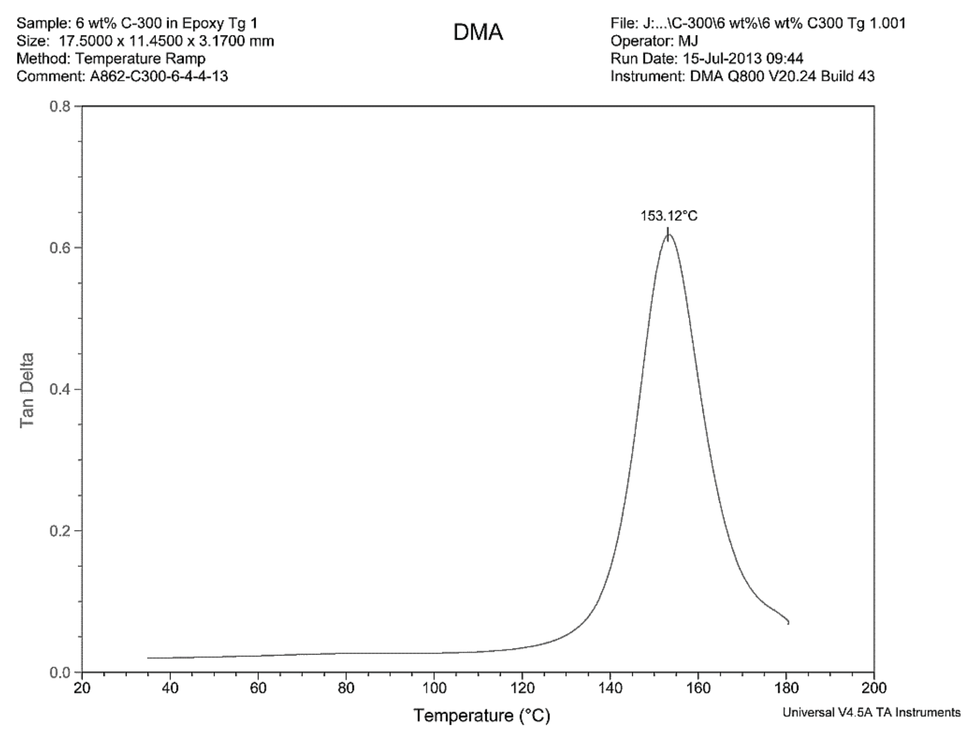


Figure D-57: Tan Delta for 6 wt% xGnP®- C-300 in Epoxy Test 1

Table D-20: DMA Results for 1 wt% TC307 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-TC307-1-1-7-15	1	133.51	146.54	154.41
A862-TC307-1-1-7-15	2	132.46	146.12	153.67
A862-TC307-1-1-7-15	3	132.36	146.36	154.11
A862-TC307-1-1-7-15	4	132.37	145.7	153.87
Average		**132.7**	**146.2**	**154.0**
Standard Deviation		**0.6**	**0.4**	**0.3**
Count		**4**	**4**	**4**

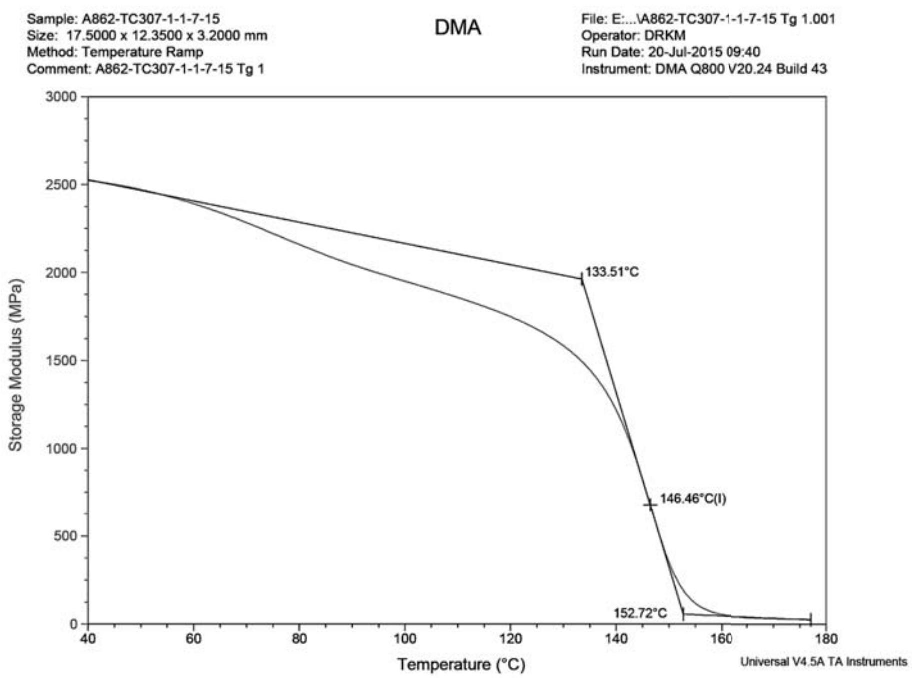


Figure D-58: Storage Modulus for 1 wt% TC307 in Epoxy Test 1

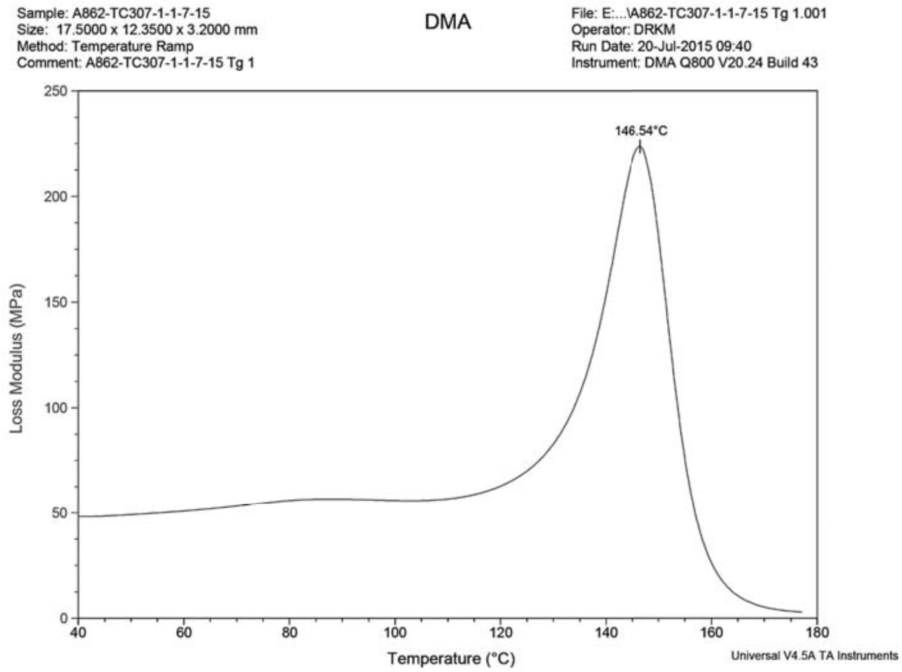


Figure D-59: Loss Modulus for 1 wt% TC307 in Epoxy Test 1

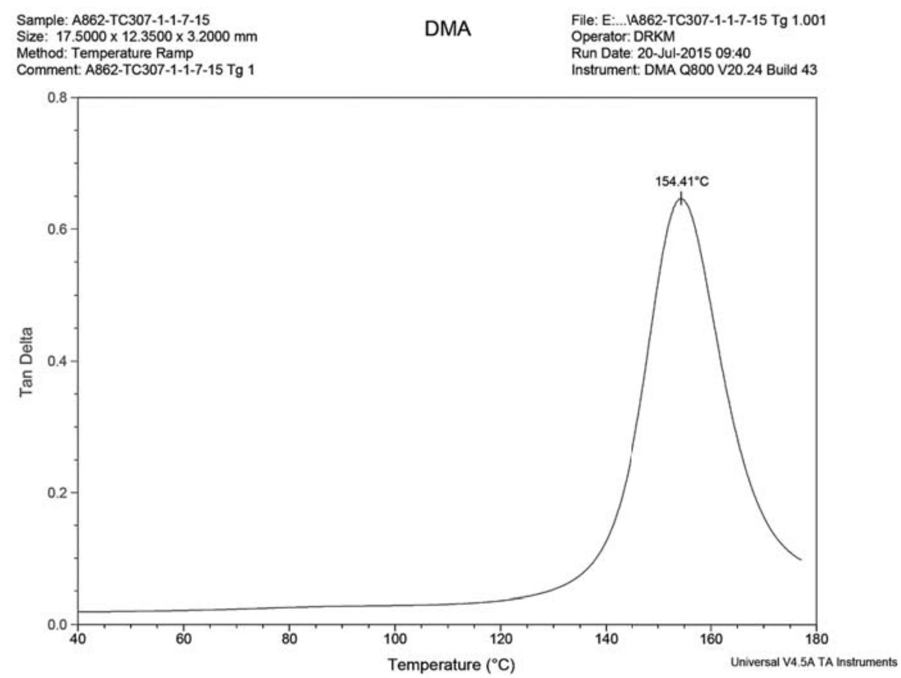


Figure D-60: Tan Delta for 1 wt% TC307 in Epoxy Test 1

Table D-21: DMA Results for 2 wt% TC307 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-TC307-2-12-14-14	1	134.65	147.76	155.33
A862-TC307-2-12-14-14	2	135.24	148.25	155.66
A862-TC307-2-12-14-14	3	134.32	148.64	155.89
A862-TC307-2-12-14-14	4	134.31	148.42	155.84
Average		**134.6**	**148.3**	**155.7**
Standard Deviation		**0.4**	**0.4**	**0.3**
Count		**4**	**4**	**4**

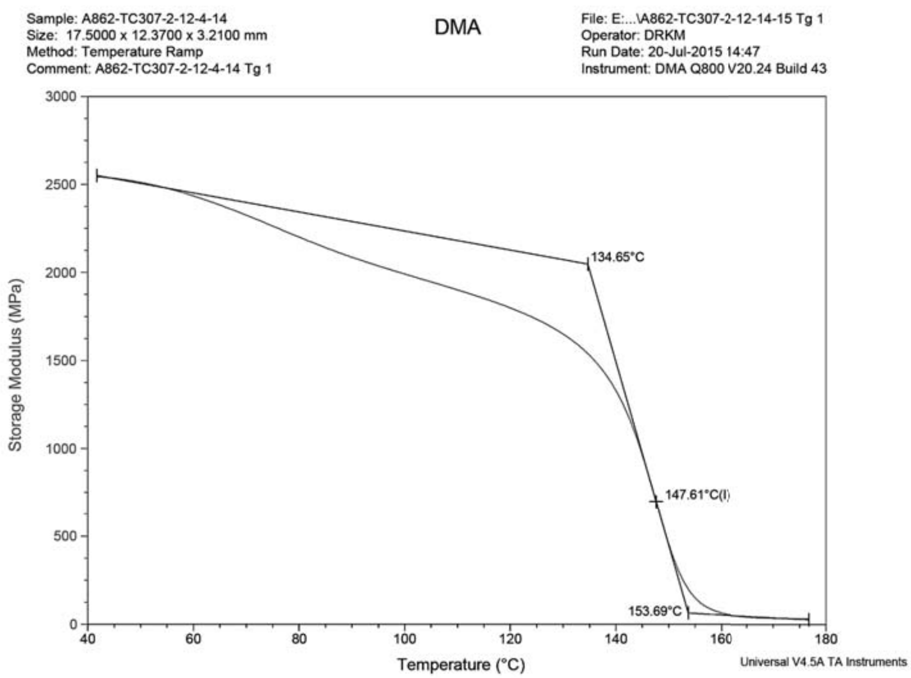


Figure D-61: Storage Modulus for 2 wt% TC307 in Epoxy Test 1

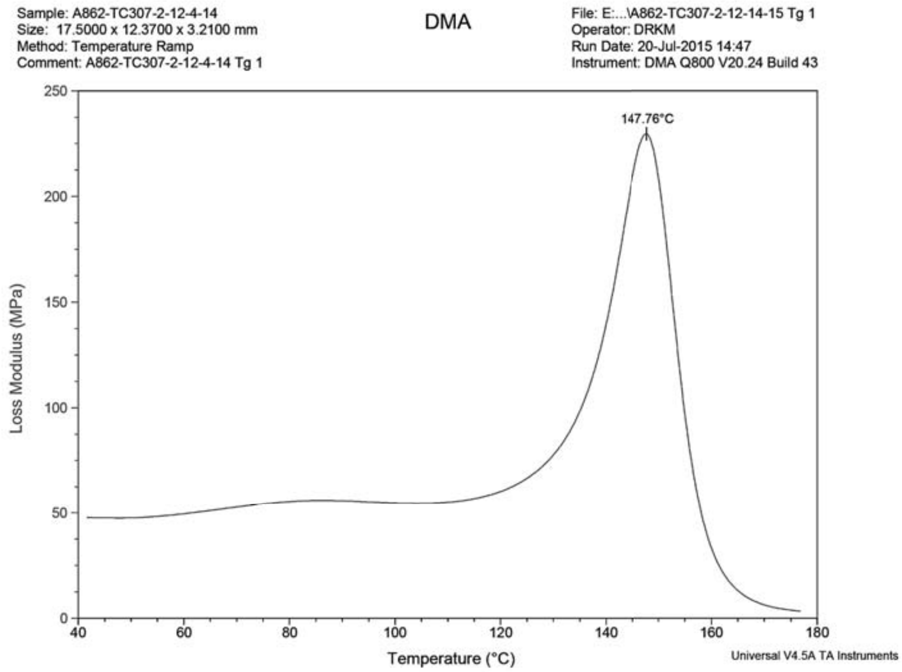


Figure D-62: Loss Modulus for 2 wt% TC307 in Epoxy Test 1

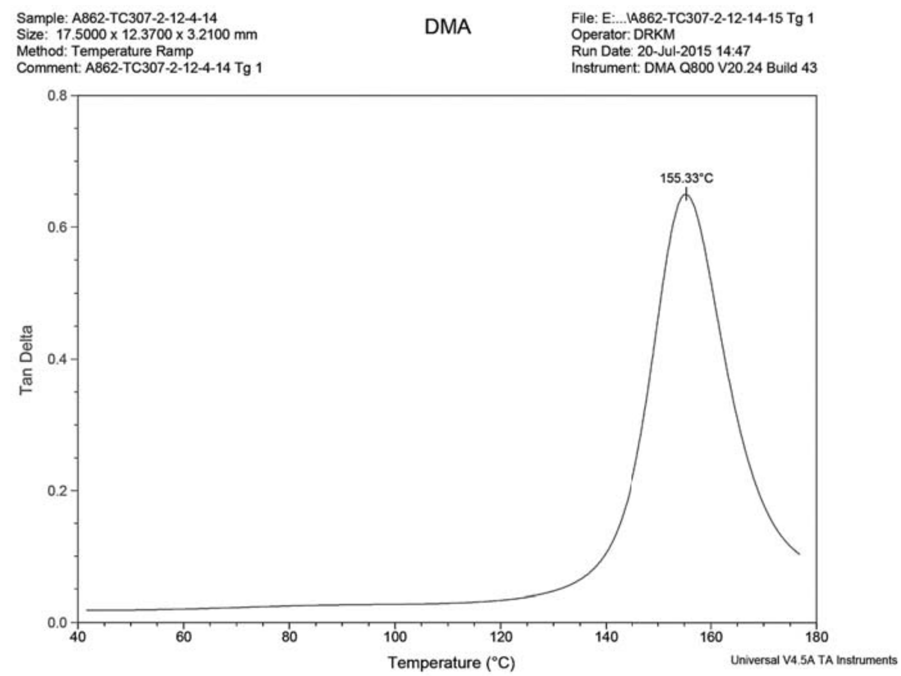


Figure D-63: Tan Delta for 2 wt% TC307 in Epoxy Test 1

Table D-22: DMA Results for 3 wt% TC307 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-TC307-3-1-9-15	1	132.80	145.45	153.46
A862-TC307-3-1-9-15	2	131.88	145.49	153.18
A862-TC307-3-1-9-15	3	132.05	145.44	153.18
A862-TC307-3-1-9-15	4	132.96	145.53	153.39
Average		**132.4**	**145.5**	**153.3**
Standard Deviation		**0.5**	**0.0**	**0.1**
Count		**4**	**4**	**4**

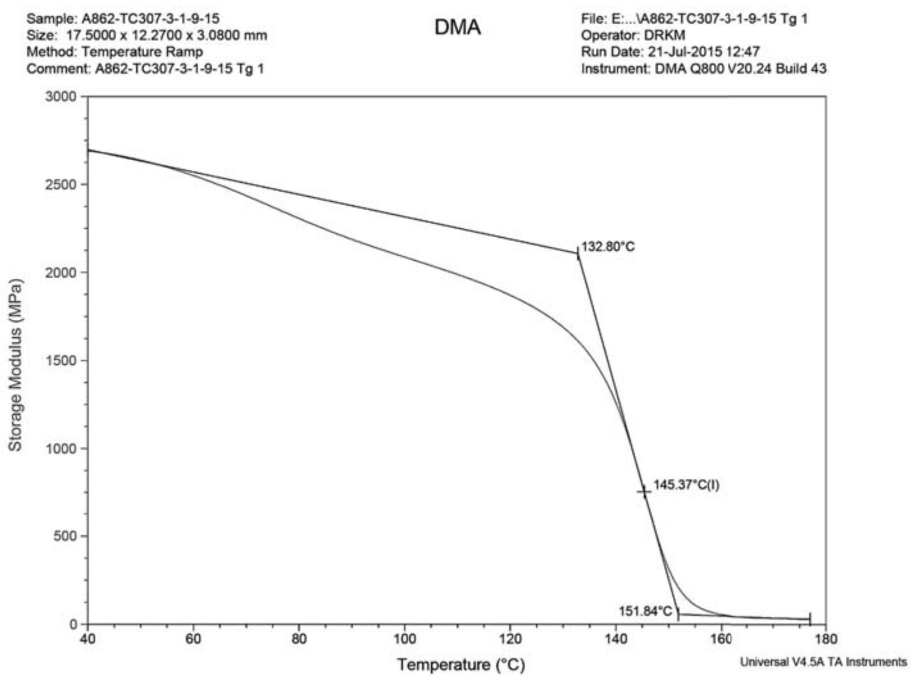


Figure D-64: Storage Modulus for 3 wt% TC307 in Epoxy Test 1

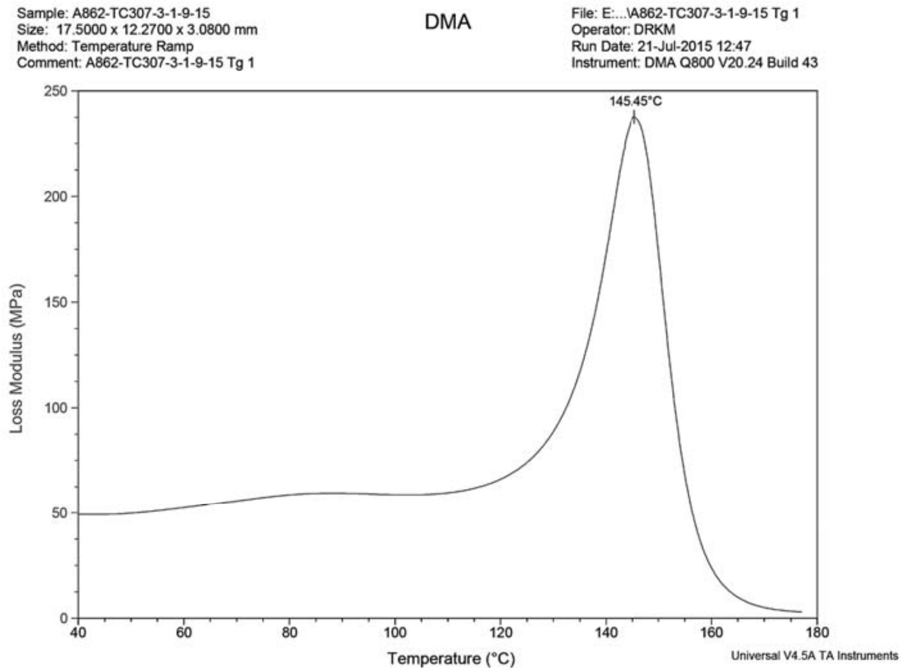


Figure D-65: Loss Modulus for 3 wt% TC307 in Epoxy Test 1

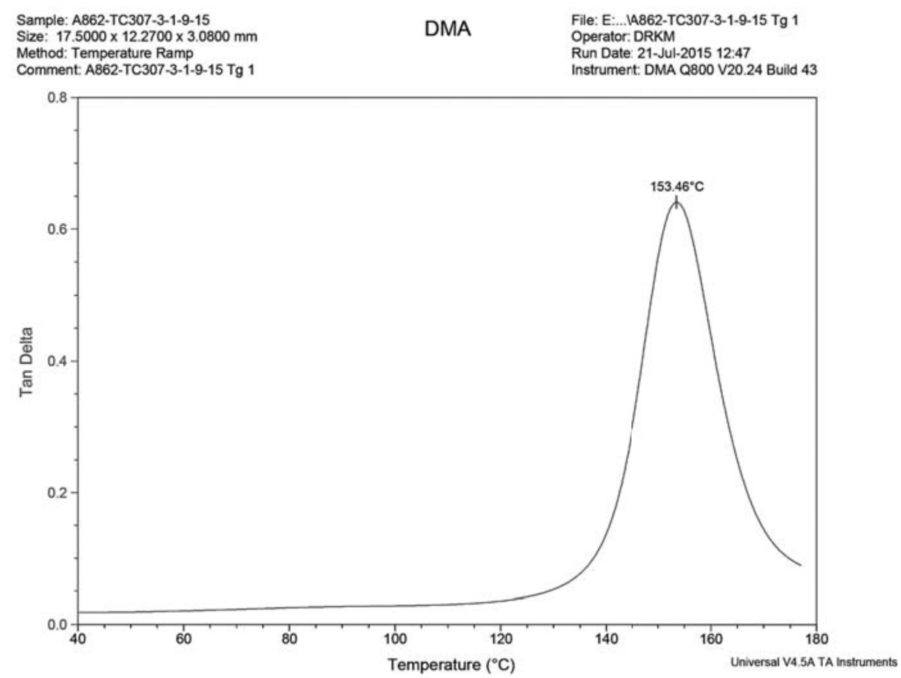


Figure D-66: Tan Delta for 3 wt% TC307 in Epoxy Test 1

Table D-23: DMA Results for 4 wt% TC307 in Epoxy

Sample	No.	Storage Modulus Onset (°C)	Loss Modulus Peak (°C)	Tan Delta Peak (°C)
A862-TC307-4-12-8-14	1	135.79	148.82	156.34
A862-TC307-4-12-8-14	2	135.58	148.71	156.38
A862-TC307-4-12-8-14	3	135.08	148.66	156.21
A862-TC307-4-12-8-14	4	135.14	148.87	156.57
Average		**135.4**	**148.8**	**156.4**
Standard Deviation		**0.3**	**0.1**	**0.1**
Count		**4**	**4**	**4**

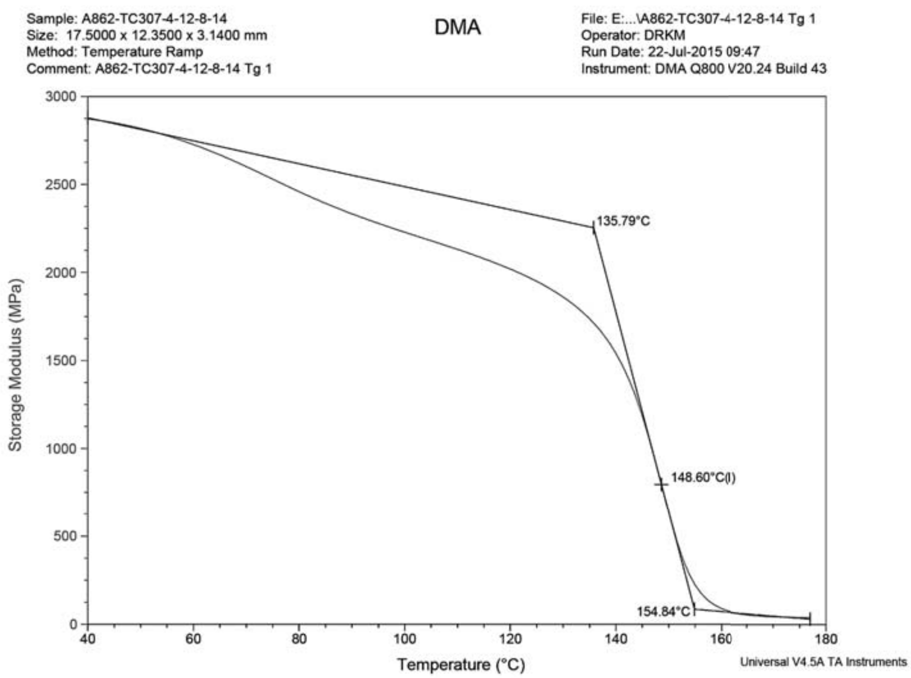


Figure D-67: Storage Modulus for 4 wt% TC307 in Epoxy Test 1

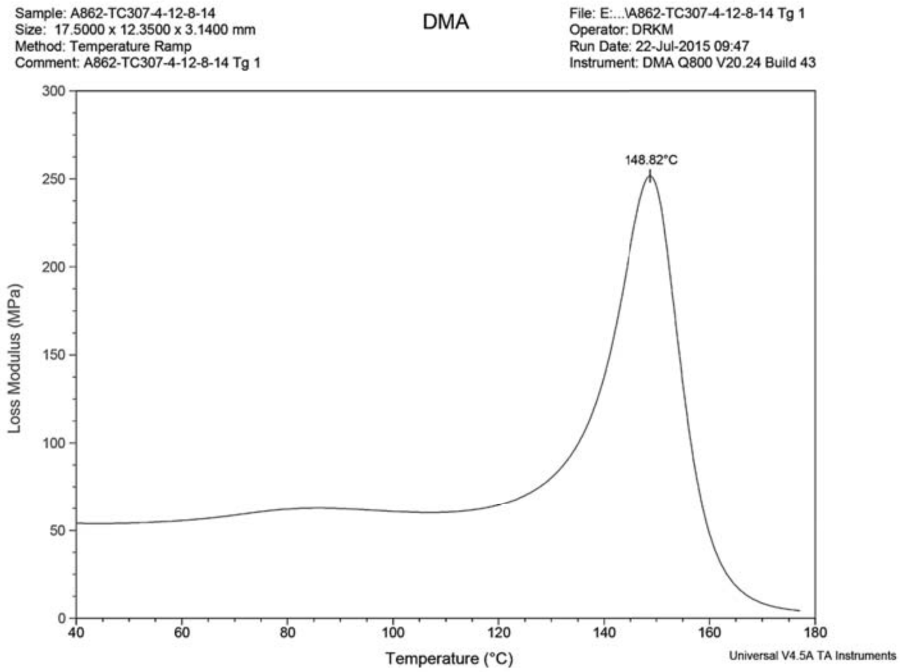


Figure D-68: Loss Modulus for 4 wt% TC307 in Epoxy Test 1

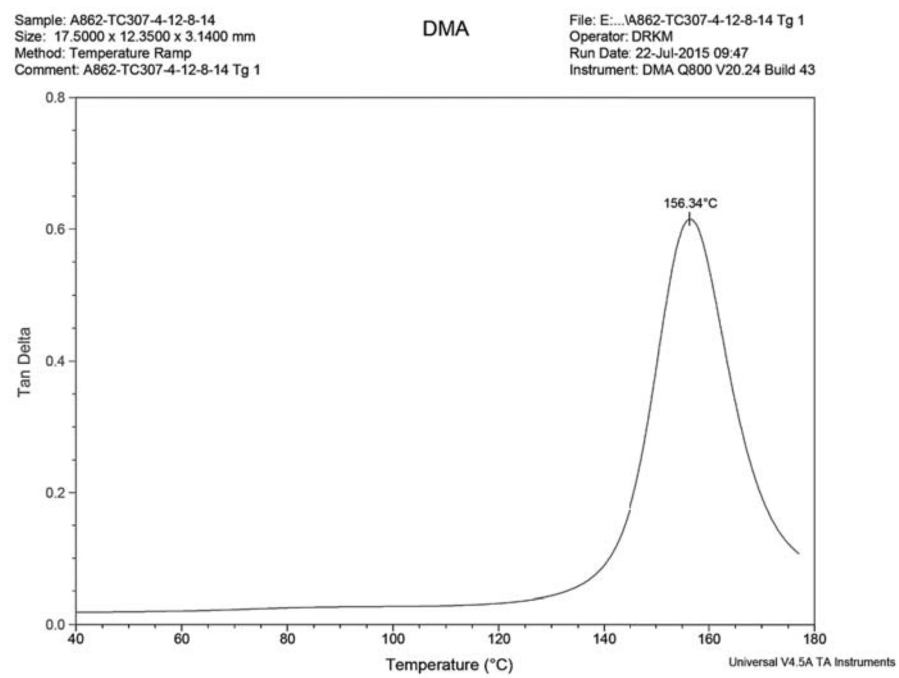


Figure D-69: Tan Delta for 4 wt% TC307 in Epoxy Test 1

Appdenix E: DSC Results

Table E-1: DSC Results for Neat Epoxy

Sample	No.	Tg (°C)
A862-9-30-11	1	142.89
A862-9-30-11	2	142.95
A862-9-30-11	3	143.95
A862-12-6-11	1	142.61
A862-12-6-11	2	144.23
A862-12-6-11	3	142.58
A862-12-6-11	4	144.12
Average		**143.3**
Standard Deviation		**0.7**
Count		**7**

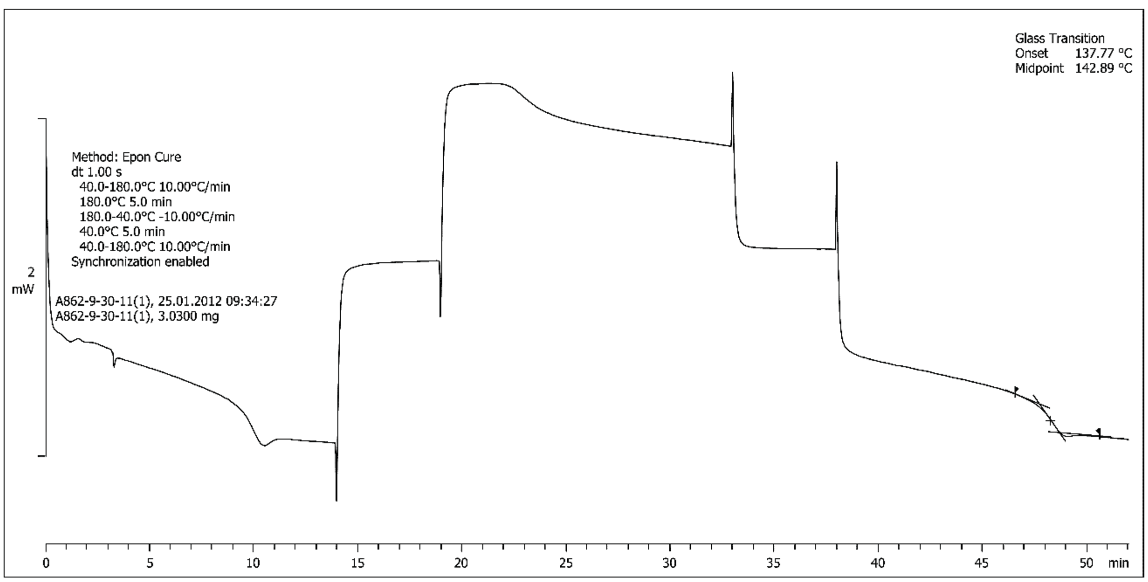


Figure E-1: DSC for Neat Epoxy (A862-9-30-11) Run 1 vs. Time

Table E-2: DSC Results for 1 wt% xGnP®-M-15 in Epoxy

Sample	No.	Tg (°C)
A862-M15-1-10-7-11	1	146.07
A862-M15-1-10-7-11	2	147.00
A862-M15-1-10-7-11	3	143.64
A862-M15-1-10-7-11	4	142.63
A862-M15-1-12-12-11	1	144.66
A862-M15-1-12-12-11	2	146.34
A862-M15-1-12-12-12	3	146.44
A862-M15-1-12-12-12	4	145.95
Average		**145.3**
Standard Deviation		**1.5**
Count		**8**

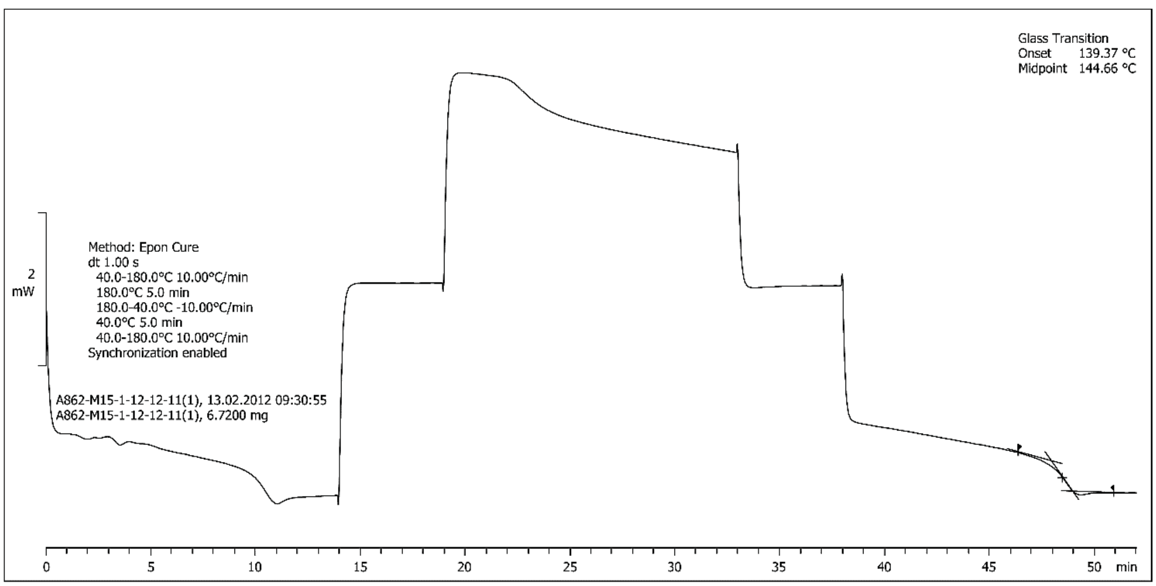


Figure E-2: DSC for 1 wt% xGnP®-M-15 in Epoxy (A862-M15-1-12-12-11) Run 1 vs. Time

Table E-3: DSC Results for 2 wt% xGnP®-M-15 in Epoxy

Sample	No.	Tg (°C)
A862-M15-2-10-4-11	1	142.45
A862-M15-2-10-4-11	2	142.25
A862-M15-2-12-13-11	1	142.64
A862-M15-2-12-13-11	2	144.21
A862-M15-2-12-13-11	3	143.76
A862-M15-2-12-13-11	4	143.55
A862-M15-2-2-22-12	1	142.30
A862-M15-2-2-22-12	2	144.87
Average		**143.3**
Standard Deviation		**1.0**
Count		**8**

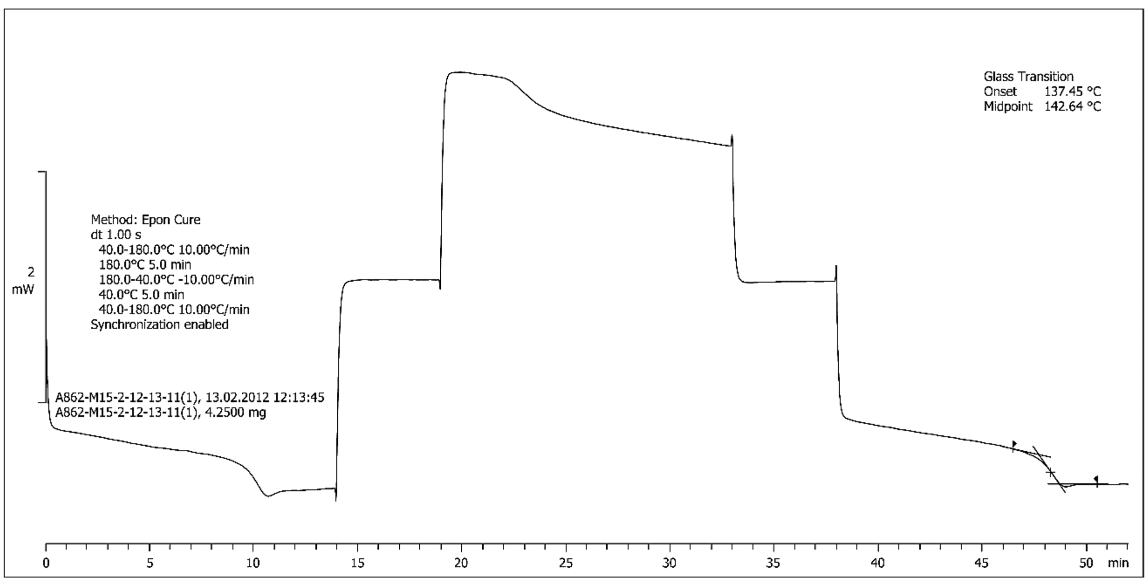


Figure E-3: DSC for 2 wt% xGnP®-M-15 in Epoxy (A862-M15-2-12-13-11) Run 1 vs. Time

Table E-4: DSC Results for 3 wt% xGnP®-M-15 in Epoxy

Sample	No.	Tg (°C)
A862-M15-3-10-11-11	1	140.04
A862-M15-3-10-11-11	2	142.23
A862-M15-3-12-14-11	1	136.07
A862-M15-3-12-14-11	2	137.10
A862-M15-3-12-14-11	3	136.71
A862-M15-3-12-14-11	4	136.33
A862-M15-3-3-14-12	1	140.73
A862-M15-3-3-14-12	2	140.71
Average		**138.7**
Standard Deviation		**2.4**
Count		**8**

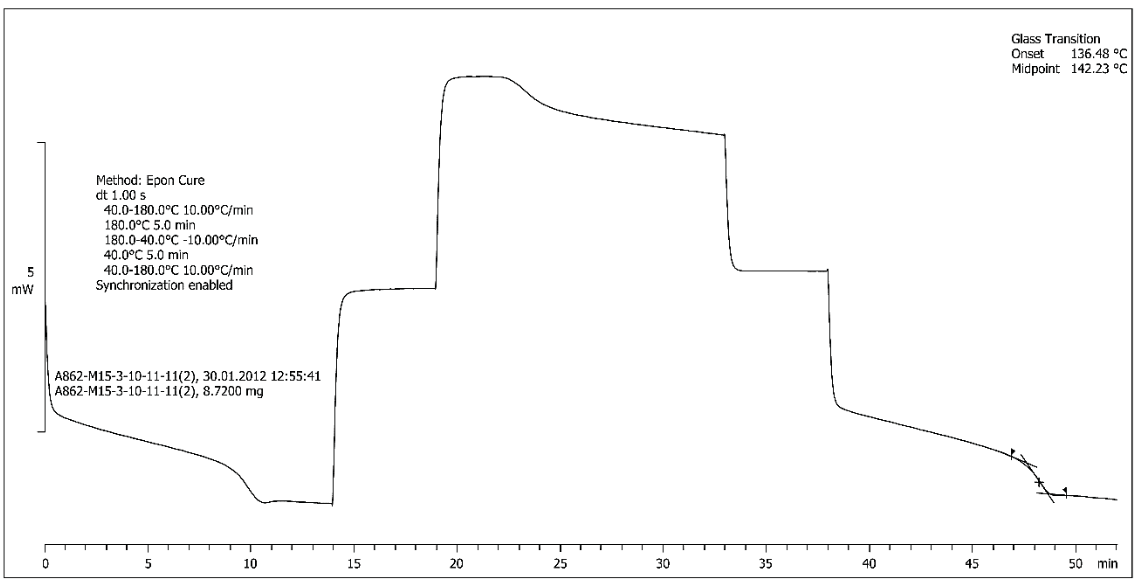


Figure E-4: DSC for 3 wt% xGnP®-M-15 in Epoxy (A862-M15-3-10-11-11) Run 2 vs. Time

Table E-5: DSC Results for 4 wt% xGnP®-M-15 in Epoxy

Sample	No.	Tg (°C)
A862-M15-4-10-13-11	1	145.32
A862-M15-4-10-13-11	2	144.73
A862-M15-4-10-13-11	3	143.63
A862-M15-4-10-13-11	4	144.23
A862-M15-4-12-15-11	1	143.94
A862-M15-4-12-15-11	2	144.07
A862-M15-4-12-15-11	3	142.40
A862-M15-4-12-15-11	4	145.77
Average		**144.3**
Standard Deviation		**1.0**
Count		**8**

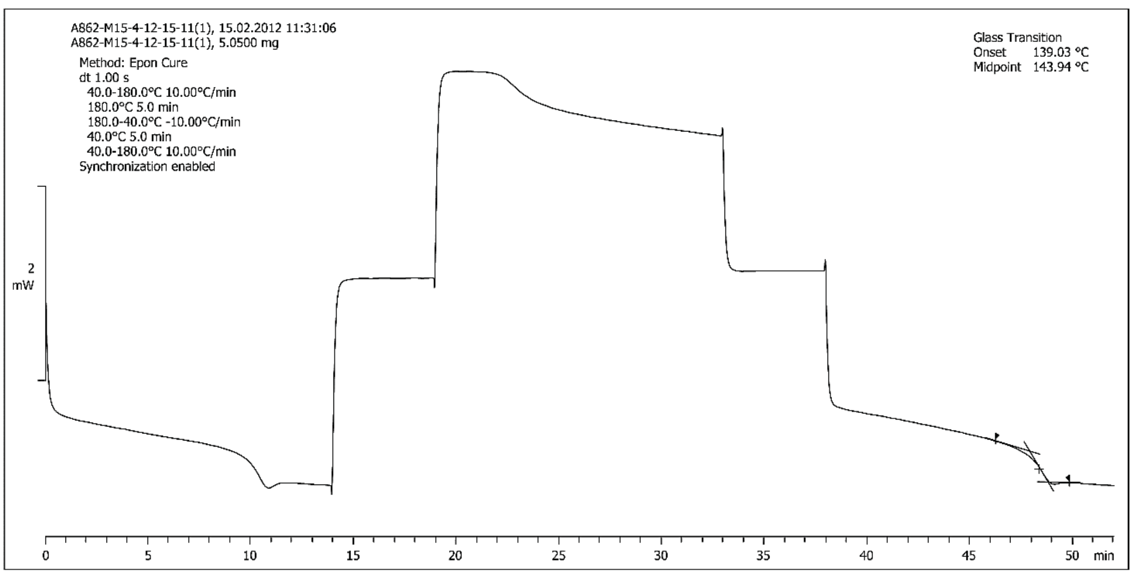


Figure E-5: DSC for 4 wt% xGnP®-M-15 in Epoxy (A862-M15-4-12-15-11) Run 1 vs. Time

Table E-6: DSC Results for 5 wt% xGnP®-M-15 in Epoxy

Sample	No.	Tg (°C)
A862-M15-5-10-18-11	1	143.58
A862-M15-5-10-18-11	2	143.29
A862-M15-5-10-18-11	3	143.59
A862-M15-5-10-18-11	4	143.63
A862-M15-5-12-16-11	1	148.27
A862-M15-5-12-16-11	2	146.94
A862-M15-5-12-16-11	3	145.84
A862-M15-5-12-16-11	4	146.55
Average		**145.2**
Standard Deviation		**1.9**
Count		**8**

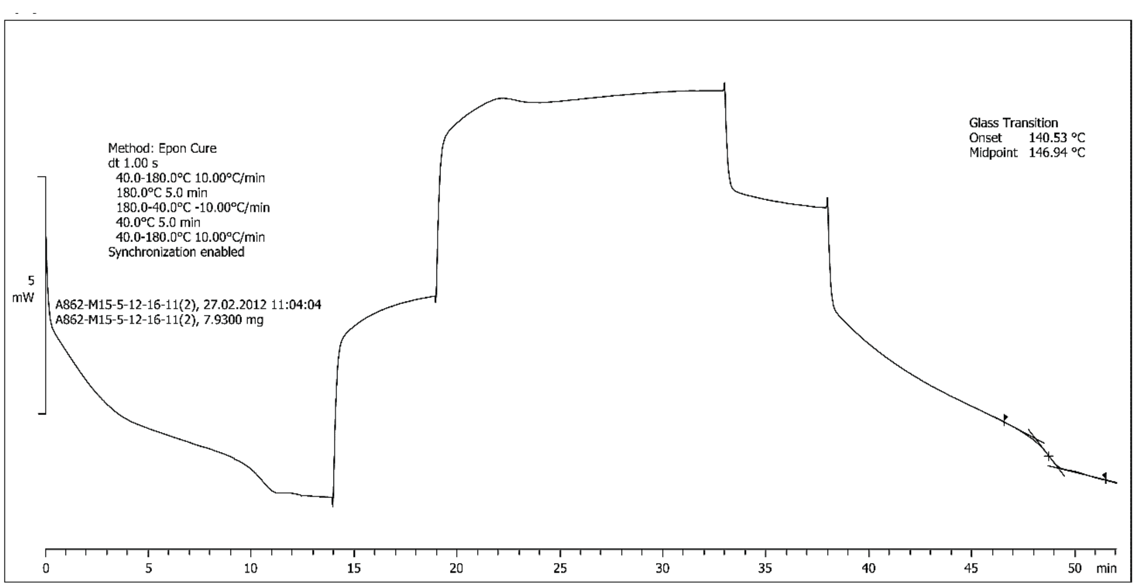


Figure E-6: DSC for 5 wt% xGnP®-M-15 in Epoxy (A862-M15-5-12-16-11) Run 2 vs. Time

Table E-7: DSC Results for 6 wt% xGnP®-M-15 in Epoxy

Sample	No.	Tg (°C)
A862-M15-6-10-26-11	1	141.08
A862-M15-6-10-26-11	2	143.07
A862-M15-6-1-19-12	1	147.12
A862-M15-6-1-19-12	2	145.44
A862-M15-6-1-19-12	3	146.19
A862-M15-6-1-19-12	4	147.47
A862-M15-6-1-19-12	5	148.04
A862-M15-6-1-19-12	6	146.05
Average		**145.6**
Standard Deviation		**2.4**
Count		**8**

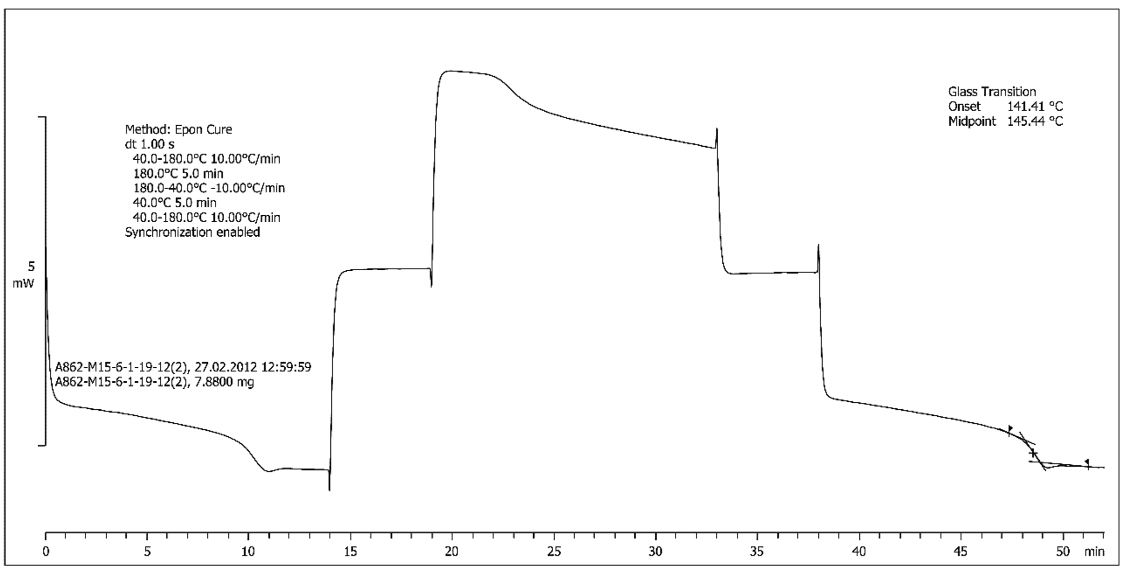


Figure E-7: DSC for 6 wt% xGnP®-M-15 in Epoxy (A862-M15-6-1-19-12) Run 2 vs. Time

Appdenix F: Acoustic Absorption

F.1 Impedance Tube Method

The standard used is ASTM E1050, this is an impedance tube method [1]. This method was chosen because the set up at Michigan Technological University was capable of testing in the frequency range of interest. The measurement of interest is acoustic absorption coefficient at a frequency range of 500-6400 Hz, this frequency was chosen because it shows the most absorption as opposed to lower frequencies, i.e. 50-500 Hz, even highly absorptive foams do not absorb much in this range. How much the sample absorbs will directly indicate how well the materials will dampen sound at the frequency of interest.

The impedance tube consists of a ridged tube with a specimen holder at one end and a speaker at the other with two microphones in between. There are a few different places for the microphones to be placed into depending on what frequency range is of interest. The speaker is connected to a white noise generator the microphones are connected to a computer with a MatLab graphical user interface (GUI) for the data acquisition. Figure F-1 below shows the setup of the tube.

First the microphones must be calibrated, a highly absorptive foam is used to do this. In the MatLab GUI use the normal configuration calibration first, then switch the microphones and use the switched configuration calibration, the configuration is based on the frequency desired for testing. To test the composite samples, the samples' edge is coated with petroleum jelly to keep any sound waves from escaping. The microphones are placed back into the normal position. The MatLab GUI takes data measurements at all the frequencies between 500 and 6400 Hz. The measurements recorded are absorption coefficient, the real and imaginary parts of the reflection coefficient, the real and imaginary parts of the specific impedance ratio, and the real and imaginary parts of the specific admittance. Only the absorption coefficient is used to indicate dampening.

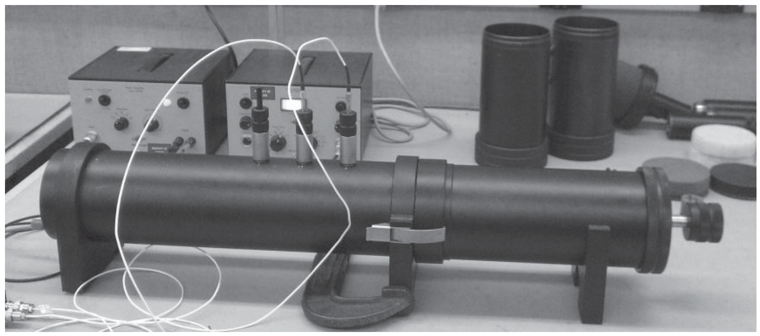


Figure F-1: Impedance tube set up

F.2 Dynamic Mechanical Analysis Method [2]

Dynamic mechanical analysis (DMA) can be used to measure tan delta. Tan delta is the measure of maternal damping, such as acoustic damping. Acoustic dampening is dominated by the polymer T_g. Equation F-1 shows the relationship between acoustic absorption (αλ, units of dB) and tan δ.

$$\alpha\lambda = 8.686 * \pi * \tan\delta \qquad \text{(F-1)}$$

Acoustic damping is dependent on the temperature and the frequency of the measurements. The measurements were done at a frequency of 1 Hz over a temperature range of 40 °C to 180 °C.

F.3 Acoustic Absorption Results

The impedance tube method works well for porous foam-like materials since they have a high absorption coefficient. The acoustic absorption for solid polymers and polymer composites have a very low absorption coefficient, with this method the acoustic absorption for the polymer could not be differentiated from the absorption of the impedance tube. Figure F-2 shows the sound absorption coefficient over a frequency range of 500-6400 Hz for the empty impedance tube and Figure F-3 shows the absorption coefficient over the same frequency range for neat epoxy and 1-6 wt% xGnP®-M-15. The profile of the empty tube is very similar to the profile obtained for the epoxy and the GNP/epoxy composites.

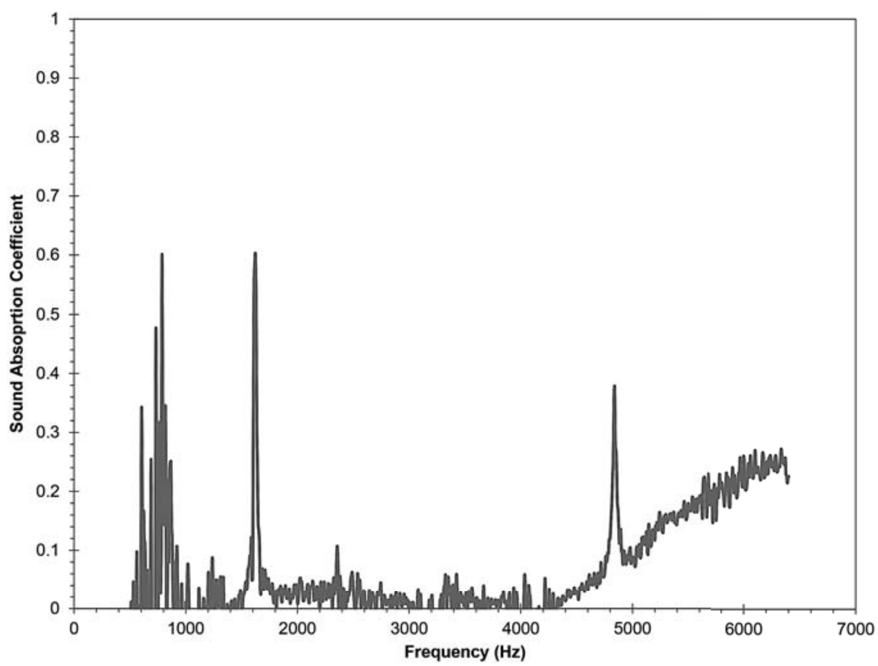


Figure F-2: Sound Absorption Coefficient for the empty impedance tube 500-6400 Hz

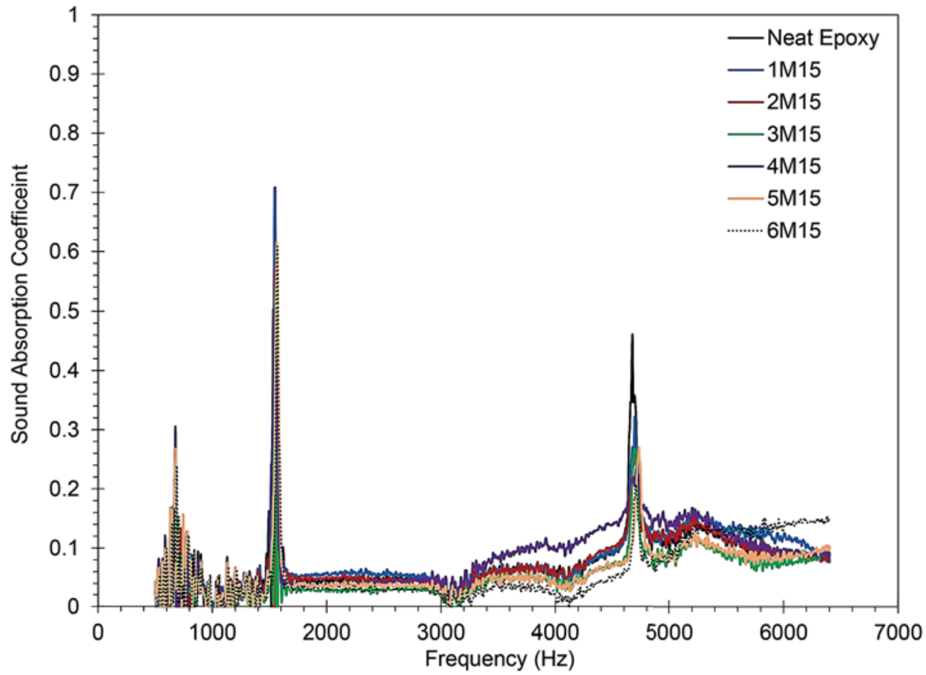


Figure F-3: Sound Absorption Coefficient for neat epoxy and 1-6 wt% xGnP®-M-15 in epoxy 500-6400 Hz

Since directly measuring the sound absorption was not possible, a DMA was used to try to relate mechanical properties to sound properties. Table F-1 shows the alpha-lambda (acoustic absorption) at 1 Hz frequency for neat epoxy and 1-6 wt% xGnP®-M-15/epoxy, xGnP®-M-5/epoxy and xGnP®-C-300/epoxy. The acoustic absorption measured at 1 Hz did not have a very clear trend. The results are scattered, but the overall trend is a decrease in acoustic absorption as more GNP was added to the epoxy, 6 wt% for all three GNPs had the lowest alpha-lambda.

Table F-1: Maximum alpha-lambda with tan delta T_g results for xGnP®-M-15 in epoxy, xGnP®-M-5 in epoxy and xGnP®-C-300 in epoxy measured at 1 Hz

Formulation	**Max Alpha-Lambda (dB)**	**Tan Delta Peak T_g (°C)**
Neat Epoxy	20.69 ± 0.17 n=6	155.8 ± 0.4 n=6
xGnP®-M-15		
1M15	17.99 ± 0.09 n=3	153.5 ± 0.5 n=3
2M15	17.30 ± 0.46 n=5	152.6 ± 0.8 n=5
3M15	18.28 ± 0.15 n=3	150.4 ± 0.3 n=3
4M15	16.81 ± 0.14 n=3	152.4 ± 0.3 n=3
5M15	17.40 ± 0.12 n=3	156.0 ± 0.4 n=3
6M15	16.61 ± 0.13 n=5	155.9 ± 0.5 n=5
xGnP®-M-5		
1M5	19.36 ± 0.14 n=3	154.3 ± 0.3 n=3
2M5	20.00 ± 0.67 n=3	153.1 ± 0.4 n=3
3M5	18.52 ± 0.09 n=3	154.7 ± 0.3 n=3
4M5	18.40 ± 0.31 n=3	156.5 ± 0.4 n=3
5M5	18.20 ± 0.02 n=3	157.2 ± 0.4 n=3
6M5	17.55 ± 0.11 n=3	156.4 ± 0.1 n=3
xGnP®-C-300		
1C300	18.46 ± 0.14 n=3	152.7 ± 0.3 n=3
2C300	19.75 ± 0.11 n=3	143.4 ± 0.2 n=3
3C300	18.02 ± 0.30 n=3	152.7 ± 0.4 n=3
4C300	17.40 ± 0.17 n=3	154.9 ± 0.3 n=3
5C300	17.60 ± 0.16 n=3	153.3 ± 0.3 n=3
6C300	16.87 ± 0.06 n=3	153.4 ± 0.2 n=3

F.4 References

[1] American Society for Testing and Materials, Standard Test Methods for Impedance and Absorption of Acoustical Materials Using a Tube, Two Microphones and a Digital Freqency Analysis System:ASTM Standard E1050-08, (2008).

[2] Carsaro, R. D., Sperling, L. H., *Sound and Vibration Damping with Polymers*. Washington DC: American Chemical Society, (1990) In print.

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