

August 10, 2006

Dr. Christopher Ibeh
PSU/NSF – REU/RET Director
Department of Engineering Technology
Pittsburg State University
Pittsburg, Kansas 66762

RE:

Dear Dr. Ibeh:

It is a privilege to present to you my summer research on the AFM phase imaging of Epoxy Resins Nanocomposite.

This paper will review the basics of nanocomposites and Phase Imaging. There is a great potential market for these nanocomposites. The AFM Phase Imaging scans of nanocomposites will help us identify variations of composition of nanocomposites.

Sincerely,

Austin M. Baldwin

Pc: Student, Physics, Pittsburg State University

AFM Phase Imaging of Nanocomposite

August 3, 2006

By

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AFM Phase Imaging of Nanocomposite

**Austin Baldwin, Physics,
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1. Abstract

Rust and corrosion have been a costly problem for steel ships since their conception. The Navy and many commercial shipping companies have begun the search for better alternatives to steel in recent years. Epoxy composite resins and composite materials are the new building for ships that steel was many years ago. By mixing nanoclay with epoxy resin the favorable properties of the material should be improve. Atomic Force Microscopy Phase imaging can be used to identify variations in materials. Although Phase Imaging this is a powerful tool, due to the sample preparation, this method is not ideal for identifying exfoliation.

2. Introduction

Nanocomposites are made by adding nano material into a plastic resin to give it added strength and other desired properties. Nano-size materials are favored for many applications due to their large surface area and at least one dimension on a nanometer scale. An increase in available surface area can increase the reactivity of reagents and catalysts. Nanocomposites that contain small amounts of plate-like nanoclay particles, in this case (Cloisite®10A), have improved mechanical and thermal properties compared to the base resins. They also have better barrier performance and flame retardency without increasing the weight or affecting the transparency according to the manufacture. Also, the nanoclay particles are a naturally occurring form of smectite. The trick is getting them to disperse correctly into composites, which will create favorable conditions for exposure of all its surface area to the polymer. This is referred to as exfoliation, but without proper exfoliation the full application of its properties cannot be realized.

The manufactures say that the insertion of Nanoparticles in the polymer matrix should increase the modulus of elasticity and perhaps the strength. The clay used in the synthesis of the nanocomposite polyurethane was a smectite clay additive with flakes that are only one nanometer thick (one-millionth of a millimeter) and 500-2000 nm long. The surface area of this semctite clay is around 700m²/g. The trade name of this clay is Cloisite®10A, which is natural montmorillonite clay modified with a quaternary ammonium salt. Several testing methods are employed to analyze the samples. Mechanical tests were carried out to estimate the effect of particle concentration along with AFM microscopy.

3. Literature Review

[1] This article gives a brief history on the AFM and discusses the basic principles on how it operates. The strengths and weaknesses of the AFM in comparison to other scanning microscopes. http://en.wikipedia.org/wiki/Atomic_force_microscope

[2] This article reviews market value potential and possible future uses for a soy based plastic. It also discusses that these renewable resources can compete with their petrochemical counterparts.
http://www.unitedsoybean.org/newuses/targets_soybased_plastics.html

[3] This paper discusses the source of natural nanoclay comes from, how it was formed, how much nanoclay there is and how SPC can give us a consistent supply.
<http://www.nanoclay.com/>

[4] This article discusses the ever growing problem of rust and corrosion on ocean going vessels. The main discussion point is the effects of rust and corrosion on oil tankers and the cost and to repair and some protection techniques. The end conclusion is that composite ships are the way of the future. "The New Supertanker Plague" By Richard Martin, http://www.wired.com/wired/archive/10.06/superrust_pr.html

[5] This article says it all in the title. The amazing thing is that the article was written in 1999 and many of the things hold true today. They found that a small percentage on nano clay filler can increase properties by astounding amounts. This potential of improvement in physical properties allow these nanocomposites to be competitive with costlier engineering resins. The other main issue in this article is the possible market for use in the auto industry. By making automobile body panels out of these nanocomposites a automakers can reduce weight and cost while maintaining quality and safety. In one word nanocomposites are the future. June 1999 *Plastics Technology* Nanocomposites: A Little Goes A Long Way By Lilli Manolis Sherman, Senior Editor

[6] This article discusses the military and civilian use of large composite vessels. Many of the ships used by the military are made by one company. Materials like epoxy resin and vacuum bagging are used in the construction. "Composite Ships: Building A New Paradigm": by Karen Fisher Mason, August 2005
<http://www.compositesworld.com/ct/issues/2005/August/950/1>

[7] This article discusses the use of phase imaging to identify the different components in composites. Phase imaging can be used to map different components in composites by measuring the level of adhesion or hardness. "Phase Imaging: Beyond Typography", K.L Babcock, C.B. Prater

4. Background Information

a. Nanoclay

Nanocomposites are made by adding nano material into a plastic resin to give it added strength and other desired properties. nano-size materials are favored for many applications due to their large surface area and at least one dimension on a nanometer scale . An increase in available surface area can increase the reactivity of reagents and catalysts. Nanocomposites that contain small amounts of plate-like nanoclay particles, in this case (Cloisite®10A), and have improved mechanical and thermal properties compared to the base resins. They also have better barrier performance and flame retardancy without increasing the weight or affecting the transparency according to the manufacture. The nanoclay particles are a naturally occurring form of smectite. The clay used in the synthesis of the nanocomposite was a smectite clay additive with flakes that are only one nanometer thick (one-millionth of a millimeter) and 500-2000 nm long. The surface area of this smectite clay is around 700m²/g. The trade name of this clay is Cloisite®10A, which is natural montmorillonite clay modified with a quaternary ammonium salt.

b. Atomic Force Microscope

Along with the many test that were ran on the samples the main focus of this research was the AFM scans. The (AFM) or the Atomic Force Microscope is a device that is used to take topographical pictures of samples at high magnification. Gerd Binnig and Heinrich Rohrer were two men that made great breakthrough in scanning probe microscopes. They found that if one could keep a metal probe about 1.0×10^{-10} meters from the surface of a conductive sample tunneling forces can be detected. From these forces the displacement of the cantilever could be measured. The other big achievement in AFMs was being able to scan non-conductive materials. The probe tip is attached by a cantilever that has a spring constant less than the atomic force that attaches the atoms of the sample. The amount that the tip moves is equivalent to the force that the sample exerts on the tip which is shown by the equation:

$$F_{spring} = -k \cdot \Delta Z$$

The spring constant of the tip must be less than $\omega^2 m$. Phase imaging is a very useful option of tapping mode on the AFM. Phase Imaging can be used to detect changes in composition adhesion hardness and many other properties. The main application that we are interested in is mapping the different components of the epoxy resin composite. Ideally, this will give us the ability to differentiate between the samples with nanoclay and without; also this would show the distribution of the nano particles on the surface.

5. Main Body

Rust arises from water's oxygen and hydrogen atoms taking electrons from atoms of iron. Saltwater conducts electricity better than freshwater; the iron in steel oxidizes creates rust more quickly in seawater - up to 0.10 millimeter per year. Given enough time, this process can eat through even the thickest hull.

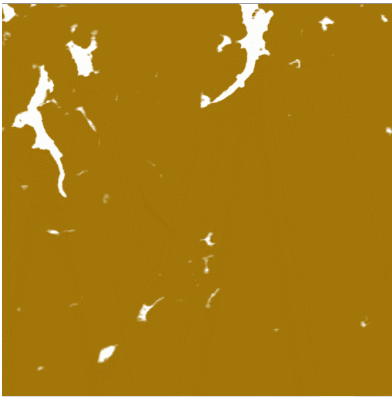
The way corrosion attacks the interior of a tanker is more devastating. This process can be seen in the cargo tanks, which go along the ship's backbone beneath the deck, and in the ballast tanks along their outer edges. In these areas, steel deteriorates at five, ten, even thirty times the nominal rate.

In the ballast tanks, which are normally filled with seawater when the cargo tanks are empty, water conducts electrons between plates on either side, and between separate areas of a single plate - that is, the tanks become huge, if weak, batteries. The increased electrical activity hastens the metal's degradation. To combat the problem, shipbuilders have traditionally installed bars of reactive metal like zinc or aluminum inside the tanks. The added metal becomes a "sacrificial anode," which corrodes in place of the ship's steel. Known as cathodic protection, this method has become less popular as paint manufacturers have developed rust-resistant coatings over the past 20 years or so. In the absence of cathodic protection, however, corrosion sets in when coatings break down.

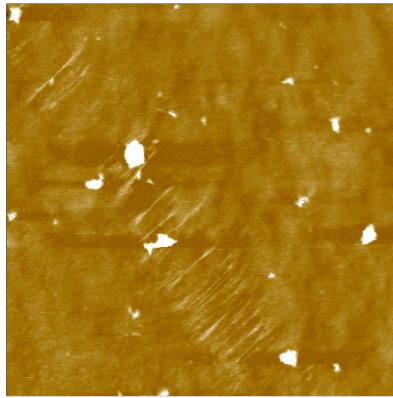
The concept of composite ships is not a new idea. The U.S navy has had a composite mine hunter ship the *Osprey*-class (MHC 51) in their fleet since 1990. Composite navel ships are made in the same way that many consumer boats are made today. The main difference between consumer and industrial ship building is the scale of the project, although some of the same techniques are used. Infusing large panels for industrial ships requires precise timing with regard to resin mixing, controlled resin formulation system from Shar Systems (Fort Wayne, Ind.) can mix as much as 55 gal of resin at a time.

Over the last ten years, polymer composites containing nano-scale layered silicate clay particles have been a big area of study. This is mainly because the addition of small amounts of clay has shown a large increase in mechanical and barrier properties of the final composite. These composites are now being considered in a wide variety of industry. It is generally believed that the improvement of the properties of the nanoclay composites is directly related to proper exfoliation of the nanoclay in the polymer matrix. However the processing technique that produces complete exfoliation being researched. This is due to the high viscosity of the resin and the tendency of nano particles to agglomerate.

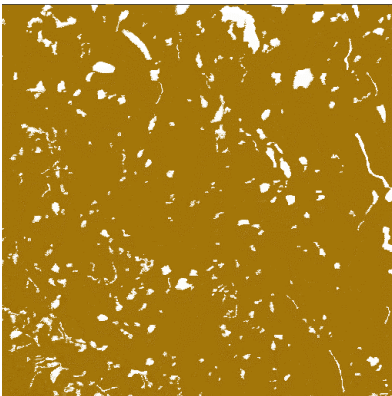
The (AFM) or the Atomic Force Microscope is a device that is used to take topographical pictures of samples at high magnification. The probe tip is attached by a cantilever that has a spring constant less than the atomic force that attaches the atoms of the sample. The amount that the tip moves is equivalent to the force that the sample exerts on the tip. Phase imaging is a very useful option of tapping mode on the AFM. Phase Imaging can be used to detect changes in composition adhesion hardness and many other properties. The main application that we are interested in is mapping the different components of the epoxy resin composite. Ideally, this will give us the ability to differentiate between the samples with nanoclay and with out; also this would show the distribution of the nano partials on the surface.



a.) intercalated



b.) immiscible



c.) disordered

These images taken by Emmanuel P. Giannelis Research Group at Cornell University show that phase imaging has the ability to capture images of the nanoclay in a sample.

6. Methodology

a. Equipment used

1. SPMLab software
3. Vibration Dampening Table
4. Veeco Explorer AFM
5. Diamond Edge Band Saw

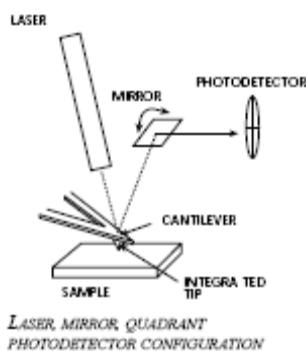
b. Materials used

1. Samples that were used were prepared by Manan Aggarwal of Pittsburg State University. The method used is outlined in Appendix A
2. Double sided tape
3. Steel disks

c. Sample Preparation

1. After obtaining samples of material, it was cut to fit the size of the mount using a diamond edge band saw.
2. A piece of double sided tape was applied to the mount so that the sample piece would adhere to the steel disk.

d. Testing procedure from Explorer Manual Beam Alignment:



LASER, MIRROR, QUADRANT
PHOTODETECTOR CONFIGURATION

Each time a cantilever is exchanged or any alignment adjustments are altered, the laser must be aligned for proper feedback. Three components must be adjusted — the laser, mirror, and photodetector. When properly aligned, the laser light beam bounces off the back side of the cantilever, to the adjustable mirror, then onto the center of the quadrant photodetector. The objective of the alignment procedure is to maximize the signal generated by the beam at the photodetector, and to ensure that nearly equal amounts of light are hitting each quadrant.



Unless engaged, the laser, mirror, and photodetector alignment thumbscrews all rotate freely without affecting adjustments. To engage an alignment screw and make an alignment adjustment, apply slight pressure inward while rotating slowly. When you feel the thumbscrew assembly slip into one of its slots, the knob will move in slightly and there will be more resistance as you rotate. At this point, you will be engaging the adjustment mechanism. Once an adjustment has been completed, disengage the thumbscrew by pulling it straight out ensuring that it rotates freely again. Disengaging serves two functions: the adjustment screw setting cannot be accidentally changed; and the interior scanning tower is isolated for stability.




IMPORTANT: When the photodetector thumbscrew is engaged and rotated to either end of its range, a torque-release mechanism will automatically slip, preventing you from over-torquing the photodetector assembly. To avoid damaging the assembly, do not rotate beyond the point at which the torque release stops the adjustment. When engaged, the mirror and laser alignment screws will come to a hard stop at the end of their adjustment range. Do not attempt to rotate the thumbscrews beyond this point.

IMPORTANT: After any alignment thumbscrew adjustment, always disengage the screw (by pulling it straight out). Failure to do so can prevent the system from scanning properly.


1. Mount a sample.
2. If not already in the SPMLab Acquisition mode, enter it by clicking on the Data Acquisition button on the Tool Bar — .
 - a. The Scanner Select dialog is activated. Select the appropriate scanner file for the scanner installed in your instrument.

IMPORTANT: *Improper selection of the scanner file may damage the scanner.*

- b. Click on OK. You will be reminded to power up the ECU-Plus. Both the ECU-Plus' Power light (red) and the Ready light (green) should be on.

IMPORTANT: *Before starting SPMLab and entering Data Acquisition —  be sure that the cantilever is raised above the sample surface. If the tip is too close to the surface at start-up, it could be broken during the software/hardware initialization sequence.*

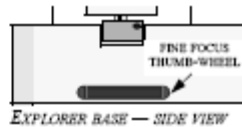
IMPORTANT: *The tip and Z scanner can be destroyed if crashed into the vacuum chuck or sample surface. Lower the head carefully while directly observing the Z scanner and the sample surface. When the Z scanner gets close to the sample surface, observe the final lowering of the cantilevers on the video microscope monitor, ensuring that the cantilevers do not contact the surface. Use the probe height thumb screws to bring the cantilevers' Z position close to, but not touching, their shadows on the sample surface.*

3. The Oscilloscope window and PreScan Acquisition Control sub-panel should be active. If not, open the PreScan sub-panel by clicking on the  button on the Tool Bar.
4. Toggle the laser intensity setting to HIGH.



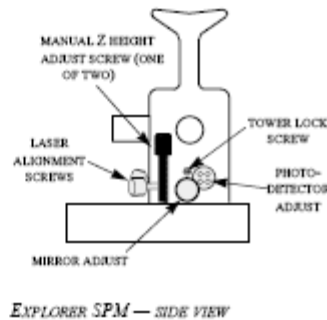
WARNING: *NEVER LOOK DIRECTLY INTO THE LASER BEAM.*

- If necessary, adjust the fine focus thumb wheel on the Explorer base to achieve optimum focus of the scanning cantilever.

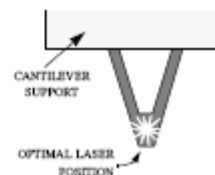


- By adjusting the laser alignment screws, move the laser beam so it is visible on the tip of the cantilever (as seen in the video monitor).

For most applications, alignment and scanning should be performed with the cantilever closest to you as viewed on the video monitor. This is the cantilever which is mounted and tested for optimal alignment.



If you are unable to see the laser beam reflecting off the cantilever, it is likely that it is nearby, but difficult to detect because it is not reflecting off the cantilever or its support. If this is the case, it is best to move the laser beam to the cantilever support, then use the laser adjustment screws to move the beam to the apex of the V-shaped cantilever you are using for the scan. You may need to lower the optical microscope's bulb brightness level (with the control at the TMX 1010 stage) to adequately see the laser beam on the tip of the cantilever.



- In the Oscilloscope window, select **INTERNAL FEEDBACK** in the **SIGNAL** field of one of the traces.
- At the **INTERNAL FEEDBACK** signal window, select **FULL SCALE**.

IMPORTANT: The **NON-CONTACT ACTIVE** option in the **Non-Contact Control** window (**SETUP** ⇒ **NON CONTACT**) must be **OFF** to properly perform beam alignment.

- On the PreScan sub-panel, toggle the Detector Signal setting to **T-B**.

The system is set to the top minus bottom alignment mode.

- Adjust the mirror until the Internal Feedback signal trace crosses the 0 nA level. (You should be able to adjust the mirror to bring the signal level into the negative and positive regions).

If you reach one extreme of the mirror alignment screw's range without being able to bring the signal level into both the negative and positive regions, try adjusting the screw in the opposite direction.

- b. Adjust the mirror until the signal is at or near the 0 nA level.

This adjusts the beam to center it between the top and bottom halves of the quadrant photodetector.

If mirror adjustment has little or no effect on the signal, the laser may not be properly hitting the apex of the cantilever. Repeat step 6; then repeat step 9.

If after repeated attempts to align the laser signal on the tip, you can not get an appropriate response, the tip you are using may be damaged or out of alignment. Turn the laser off; install a new tip, then repeat steps 4, 6, and 9.

- 10. On the PreScan sub-panel, toggle the Detector Signal setting to L-R.

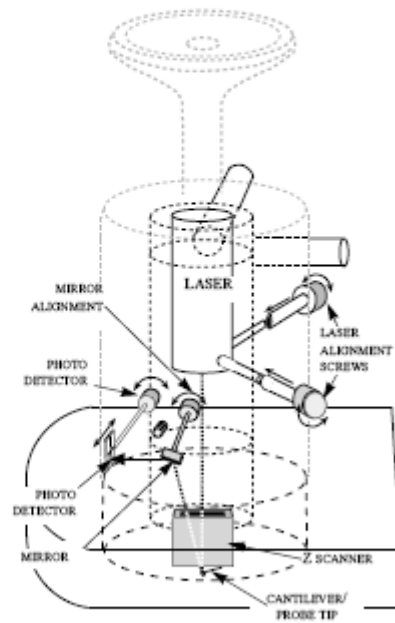
The system is put in lateral force (left minus right) alignment mode.

- a. Adjust the photodetector thumbscrew until the Internal Feedback signal trace crosses the 0 nA level. (You should be able to adjust the photodetector to bring the signal level into the negative and positive regions).

If you reach one extreme of the photodetector alignment screw's range without being able to bring the signal level into both the negative and positive regions, try adjusting the screw in the opposite direction.

- b. Adjust the photodetector until the signal is at or near the 0 nA level.

These operations center the beam on the photodetector quadrants.



EXPLORER HEAD

11. On the PreScan sub-panel list, toggle the Detector Signal setting to **SUM**.
In this mode, the photodetector quadrants are summed, allowing fine adjustments of the laser alignment on the cantilever.
 - a. Carefully make very fine adjustments to the laser alignment screws. (one at a time) to maximize the sensor current, as displayed on the Oscilloscope's internal sensor current trace.

12. On the PreScan sub-panel, toggle the Detector Signal setting to **L-R** again.
 - a. Adjust the photodetector until the Internal Feedback signal trace crosses *over* the 0 nA level. (You should be able to adjust the photodetector to bring the signal level into the negative *and* positive region).
 - b. Adjust the photodetector until the signal is at or near the 0 nA level.
This re-adjusts the beam to center it between the right and left halves of the photodetector again.

13. On the PreScan sub-panel, toggle the Detector Signal setting to **T-B** again.
 - a. Fine adjust the mirror until the Internal Feedback signal trace crosses the 0 nA level. (You should be able to adjust the mirror to bring the signal level into the negative and positive regions).
 - b. Adjust the mirror until the signal is at or near the 0 nA level.

NOTE: The laser should now stay in alignment as long as the tip is in place. Unless you accidentally change the laser, photodetector, or mirror adjustments — raising and lowering the head for sample replacement should not affect laser alignment.

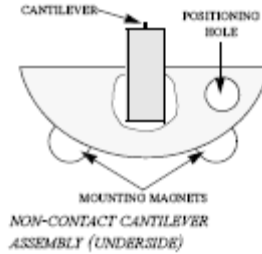
14. Disengage all of the alignment thumbscrews (by gently pulling them straight out).


Phase Imaging Procedure:

1. Simultaneously rotate the (two) probe height thumb screws clockwise at least one half rotation to ensure that the probe is elevated above the stage and sample surface.
2. If your system does not already have the Z scanner installed, install the appropriate one in accordance with the procedure described in "Z Scanner Selection/Installation," on pg. 2-18.

- Mount a #1660 (LRF)-type or #1650 (HRF) cantilever/probe assembly in the magnetic seat of the Z scanner assembly. (See "Cantilever/Tip Specifications," on pg. 2-43 for more information on non-contact probes).

If you are not familiar with the cantilever/probe mounting procedure, refer to "Tip Mounting," on pg. 2-23.

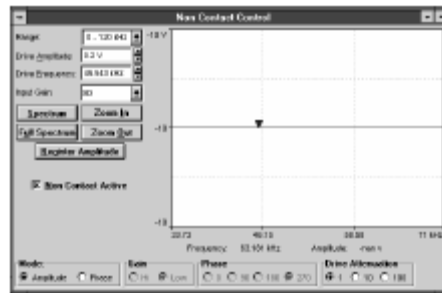


- Align the laser beam to the tip by following the Laser Alignment procedure described in "Beam Alignment," on pg. 2-38 — making sure that the final mirror adjustment brings the Internal Feedback signal to approximately 0 nA.
- If your system is not in the Data Acquisition mode, enter it by selecting WINDOW ⇒ IMAGE ACQUIRE or by clicking on the  button on the Tool Bar.
- Mount the sample.
- Select SETUP ⇒ NON CONTACT).

The Non-Contact Control window appears.

- In the Non Contact Control window, select the NON CONTACT ACTIVE option.
- Click to select the AMPLITUDE button in the Mode group box*.

The system is set to the amplitude detection non-contact mode.



**NOTE: This portion of the Phase Detection procedure must be run in the Amplitude mode. The Phase mode will be selected later.*

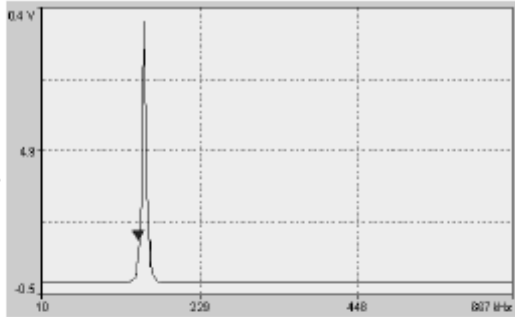
- Depending on the installed Z scanner, set the initial value in the DRIVE AMPLITUDE field to the appropriate setting in accordance with the table found in "Initial Settings — Drive Amplitude, Input Gain, Drive Attenuation," on pg. 5-5.
- Depending on the installed Z scanner, set the initial value in the INPUT GAIN drop-down list to the appropriate setting in accordance with the table found in "Initial Settings — Drive Amplitude, Input Gain, Drive Attenuation," on pg. 5-5.
- Set the RANGE field to the widest range in the drop-down list.

13. Click on the **SPECTRUM** button.

The system sweeps the full frequency range, displaying the cantilever oscillation amplitude.

In the specified range, the spectrum is displayed as probe oscillation amplitude versus frequency, with a resolution of 200 sampling points at 2 kHz.

Because the normal FWHM (full width at half maximum) of a resonance peak is typically about 30 Hz ~ 2 kHz, the full frequency sweep is only used to locate the resonance. The zoom function allows a higher resolution display at the probe's resonant frequency peak.



14. After the spectrum is displayed, click on the **Zoom In** button.
- Identify the sharp, narrow peak within the resonant frequency range of your cantilever. This is your cantilever's resonant frequency peak.
 - Position the cursor anywhere in the spectrum display, and click on the left mouse button.
A vertical line marker is attached to the cursor.
 - Position the line marker to the immediate left of the resonant frequency peak, and click the left mouse button.
The lower limit of the reduced frequency range is set.
 - Position the line marker to the immediate right of the peak, then click the left mouse button again.
The upper limit of the reduced frequency range is set.
 - Click on the right mouse button.
The zoom function is exited and the system sweeps the newly specified frequency range. You can perform this zoom function again if necessary. A frequency range of 5 kHz should be adequate for the final zoom.

15. Click to select the **PHASE** button in the Mode group box.

16. Click on the **SPECTRUM** button.

The system begins the sweep of the frequency range and displays the cantilever phase shift.

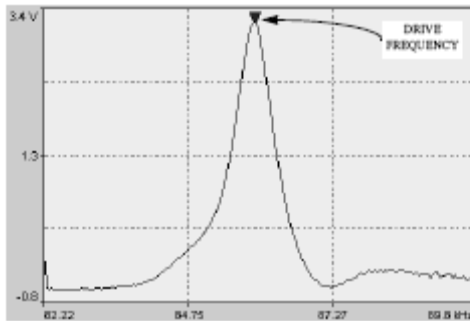
17. Position the cursor anywhere in the spectrum, and left-click.

A vertical line marker is attached to the cursor.

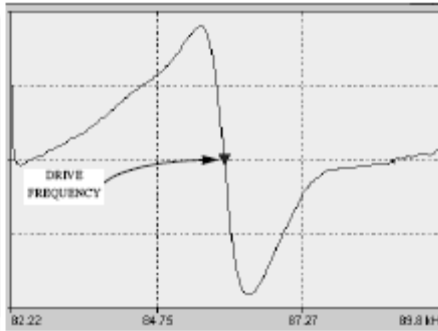
- Click to select the 0 phase angle option in the **PHASE** group box.

The wave form should look like one of the following four examples:

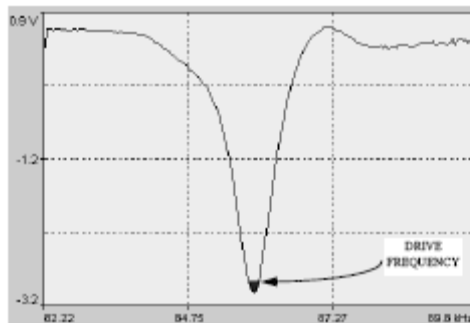
Example 1



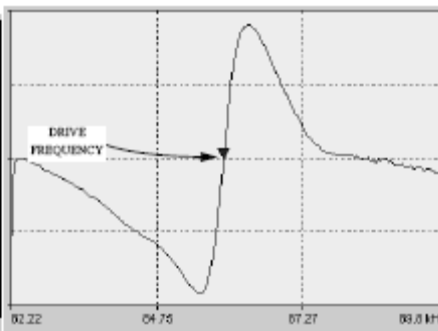
Example 2



Example 3



Example 4



- b. After determining which example matches your setup, right-click set the drive frequency on your wave form at the same point as the appropriate example illustration.

If necessary, you can reset the drive frequency by repeating steps a and b.

(If you need better resolution to monitor the oscilloscope signal change, click to select the **AUTO SCALE** option button in the Oscilloscope window.)

18. While monitoring the **INTERNAL FEEDBACK** signal in the Oscilloscope window, click to select each phase angle button in the Phase group box

(0, 90, 180, 270). Select the phase angle option button that gives the most negative signal on the **INTERNAL SENSOR FEEDBACK** trace on the oscilloscope.


19. Place the cursor on the **INTERNAL FEEDBACK** trace in the Oscilloscope window, click the left mouse button, then take note of the signal level (in nA) as the *internal feedback signal* — for reference in step 20. (The signal level is displayed above the trace.)
20. At the Acquisition Control Panel, adjust the **SET POINT** field to a setting 25%-50% more positive than the actual internal feedback signal recorded in step 19 — e.g., if the signal is -6nA, adjust the set point value to ~ -3nA.


WARNING: Do not use the **Sensor Response** button/function (on the Acquisition Control panel) when performing the computer-controlled non-contact operation. Using Sensor Response in this mode will damage the probe tip.

21. Minimize the Non Contact Control window so you can easily access the rest of the interface — but **DO NOT** close the window.
22. Ensure that the **HIGH** button is selected in the Laser group box.
23. Ensure that the **T-B** button is selected in the Detector Signal group box.
24. Select **SETUP** ⇒ **ACQUIRE**. The Image Acquire Setup window will be invoked.
 - a. Click to select the **TOPOGRAPHY** mode in the Data Channels group box.
 - b. Click **EXIT** to close the window
25. Click on the **TIP APPROACH** button.
 - a. Once in feedback, select the **LINE SCAN** option on the Oscilloscope.

The **INTERNAL FEEDBACK** signal should be stable and have low noise. If not, move the tip away from the sample surface by decreasing the set point (more negative), or move the tip closer to the sample surface by increasing the set point.

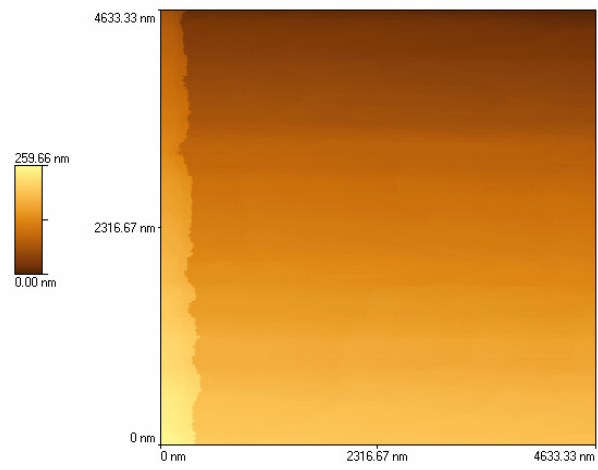
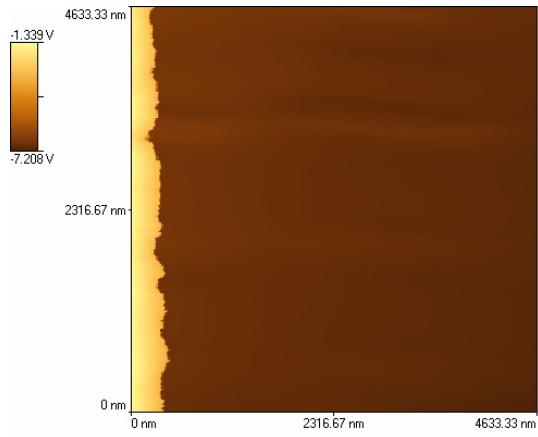
WARNING: Decreasing the set point too much risks tip damage.

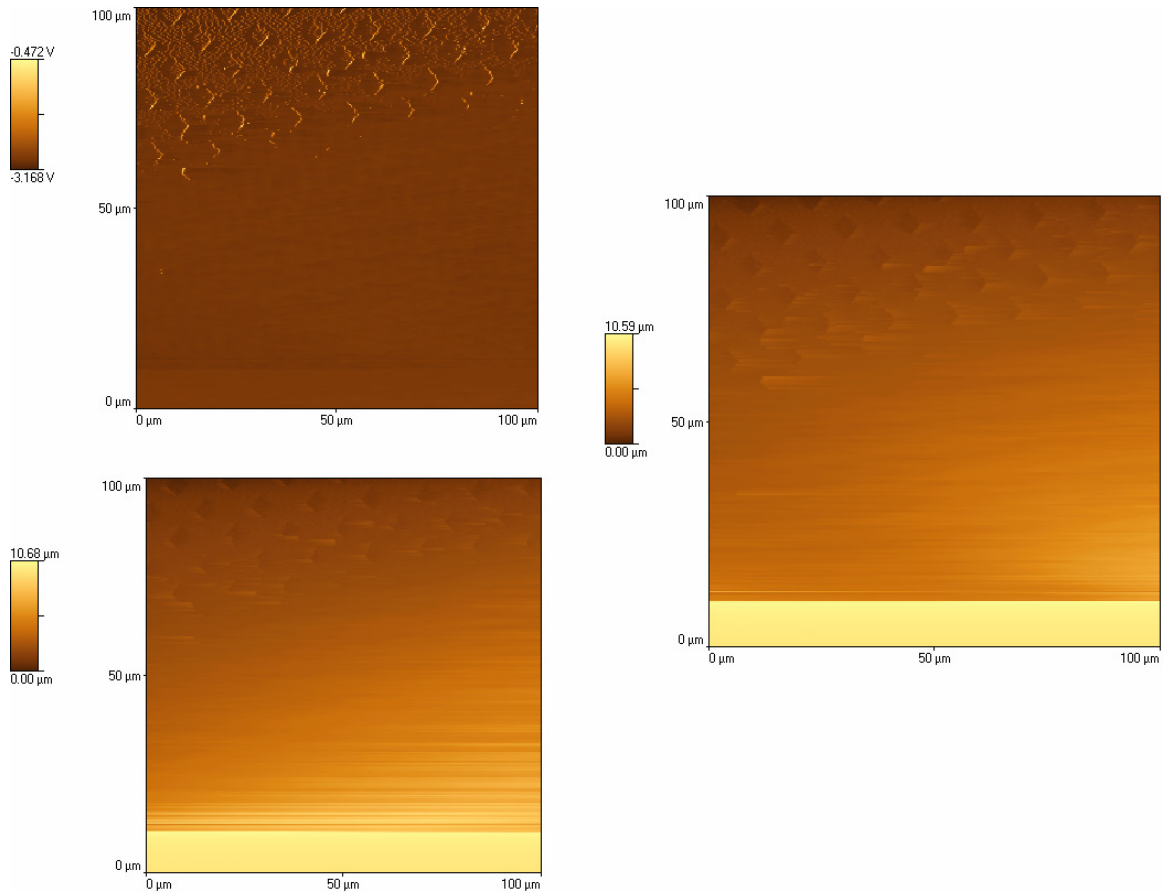
26. Click on the Instant Scan button —  — to initiate a single scan.

You can also click on the Repeat Scan button — , initiating a continuous scan mode. This will cause the scan to repeat after the last scan line in the scan area is reached, overwriting the previous image. Click on the button again to resume single-scan operation.
27. Adjust your feedback parameters to optimize system performance (*described in "Chapter 4 — Optimizing Feedback Parameters"*).

7. Results and Evidence

Evidence of Damaged Tips





8. Discussion of Results

As you look at the first set of images you will notice that there is a very distinguishable artifact on the left side of the image. This feature appeared each time I zoomed in on a different section of the scan. This shows that the tip is probably damaged. The second set of pictures is of the calibration grating and the image should look like a waffle. As you can see the tip didn't get a proper image of the grating which also points toward the conclusion that the tip is damaged.

9. Conclusion

The exfoliation of the material could not be determined due to damaged tips and possibility sample preparation. Making a sample that may give information about the exfoliation would not give accurate data about samples made using different methods. A Transition Electron Microscope (TEM) would work much better than an AFM due to the fact it works much like a slide projector. A projector shines a beam of light through (transmits) the slide, as the light passes through it is affected by the structures and objects

on the slide. These effects result in only certain parts of the light beam being transmitted through certain parts of the slide. This transmitted beam is then projected onto the viewing screen, forming an enlarged image of the slide.

TEMs work the same way except that they shine a beam of electrons (like the light) through the specimen (like the slide). Whatever part is transmitted is projected onto a phosphor screen for the user to see. With this technology one could view the nanoclay in the sample and not be limited to the surface layer. The AFM is a powerful tool but is limited to imaging the surface of the material. Even though Phase Imaging can determine the different components in a sample it is not suitable to find if a sample is exfoliated. Although the Cornell group seem to have gotten images that show dispersion patterns. I am still not clear on how their samples were made. I tried to contact the principal investigator but he did not recall how their samples were made. Although phase imaging should be able to show us the answers we must first find the best way to make samples suitable for imaging.

10. Acknowledgement

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- ❖ Stefano Bietto
- ❖ Manan Aggarwal
- ❖ Dr. Chuck Blatchley
- ❖ Dr. Christopher Ibeh C, Director, CNCMM

11. Reference

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12. Appendices:

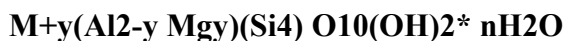
Appendix A

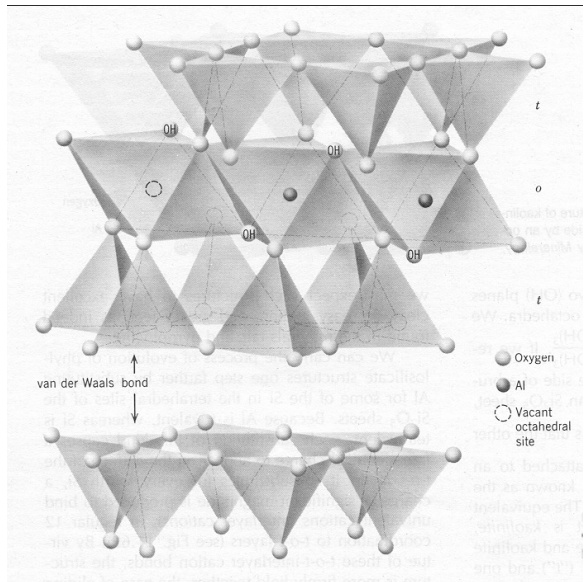
Procedure to produce nanoclay composite

"The epoxy resin used was liquid diglycidil ether of bisphenol A (DGEBA) and it is purchased from the System Three inc. Silvertip Laminating Resin is a medium-low viscosity, liquid epoxy resin system that has been optimized for coating and reinforcing fabric. It has superior wet-out characteristics with little tendency to foam or trap air. Both the resin and hardener are nearly colorless and are used in an easy 2:1 volumetric ratio. Silvertip Laminating Resin cures to a brilliant blush-free film with either the fast or slow hardener eliminating secondary bonding concerns as experienced with other epoxies. The two part A and part B are mixed in the volume of 2:1. epoxy is highly branched aliphatic polyester backbone with average of 11 epoxy groups per molecule.

The clay used for synthesis of nanocomposites was commercially treated clay manufactured by Nanocor which contains Montmorillonite. Montmorillonite is classified as the magnesium aluminum silicate which can be used to make a new class of clay/polymer. Montmorillonite has a sheet type or platy structure. Although their dimensions in length and width directions can be measured in hundred of nanometers, the mineral thickness is only one nanometer. As a result individual sheets have aspect ratios (L/W) varying from 200-1000, with a majority of platelets in the range 200-400 after purification.

The structure and the formula of the montmorilliate is shown in the figure 1.





Preparation of the epoxy-nanocomposites:-

The first step in the preparation of the composite is to dry the nanoclay in oven at 80 degrees for 24 hours. After drying the clay, part A of the epoxy is heated in the oven for 30 min at 65 degree so that the viscosity of the part A reduces and the clay can be dispersed in it properly, after removing from the oven nanoclay in the desired percentage (2%, 4%, 6%) is mixed to the part A by mechanical mixing for 24 hours using the mechanical drill head stirrer or the mixture can also sonicated for the 30 min at 65° and the amplitude is set at 35 and the energy supplied was set to 6% using the -----sonicator. The temperature probe (which is provided with the sonicator) is inserted in the mixture to know the temperature increase in the mixture due to the sonication. A sonication result in increase in temperature of the mixture, to speed up the sonication process the mixture is placed in a water + ice beaker so the heat dissipate into the surroundings at the faster rate. Then the mixture is placed in the vacuum oven and degassed it for the 2 hours at 70° (depend upon the content of clay). The degassing is required so that the bubbles trapped inside the mixture comes to the surface and escape. Mixture is then placed at the room temperature to cool down and part B (hardener) is added to the mixture in amount to 44% of the part A, the part B is mixed by mechanical mixing by hand for 2-3 minutes till the two parts mixed properly and the mixture is poured in the aluminum molds, before the mixture is poured in to the molds the mold must be clean and the mold release must be applied to the molds so that the samples can be removed easily. Leave the mixture for curing for 24 hours at room temperature. After 24 hours the samples are removed from the molds and placed in the oven for post curing at 100 degrees for 2 hours. Once the samples get cooled, they are ready for testing.” (Manan Aggarwal , cncmm summer 2006)