

Fabrication and Testing of Arabinan Cellulose Nanocomposites

A Senior Project

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California Polytechnic State University, San Luis Obispo

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Bachelor of Science, Materials Engineering

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Approval Page

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Materials Engineering Department

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Abstract

Inspired by the structure and composition of cactus spines found in nature, arabinan-cellulose nanocomposites were fabricated into thin films and tested for mechanical stiffness. The composites consisted of varying amounts of nanocrystalline cellulose reinforcement suspended in an arabinan matrix. Both materials are polysaccharides and are known to be biodegradable and food safe. The thin film samples were tensile tested using a dynamic mechanical analysis machine both as-cast and after a heat-treatment. The heat treatment of the arabinan itself resulted in an order of magnitude increase in stiffness, while the cellulose reinforced composites increased roughly six fold. The arabinan-50 wt% cellulose heat-treated sample had a mean stiffness of 6.09 GPa. These results are promising because early trials have already surpassed the stiffness of PET, a common packaging material and are approaching future goals of matching the mechanical properties of fiberglass and eventually dry native cactus spines.

Keywords: materials engineering, arabinan, cellulose, cactus spines, nanocomposite, nanocrystalline, fabrication, dimethylformamide, vacuum oven, dynamic mechanical analysis, heat treatment

1. Introduction

1.1 Motivation

Packaging waste accounts for a large percentage of all waste. Consumer packaging alone has accounted for more than 20% of all US landfill waste over the last two decades, while little of it is actually recycled. Even worse, it can take up to 1000 years for this waste to degrade. It would be ideal to have a stronger, sustainable, biodegradable and food safe material that could mitigate this waste. A candidate material already exists in nature.

Cactus spines have extraordinary specific strength and stiffness properties, with similar values to those of steel and fiberglass. Unfortunately, cactus spines are not a practical structural or packaging material, as their limited size and geometry makes them inadequate for use in most applications.

1.2 Project Goals

The purpose of this project was to create a castable new material that mimics the structure and composition of cactus spines, while retaining their mechanical properties. The solution was arabinan cellulose nanocomposites, or ArCCs for short.

1.3 Realistic Constraints

Obtaining the raw materials from their native state was beyond the scope of this project, thus the components were sourced from credible commercial suppliers. Additionally, due to the high cost of these materials, samples were fabricated and tested for bulk properties as thin films and in small sample sizes, with the hopes of establishing a scalable procedure for future research.

2. Background

2.1 Cactus Spines

Cactus spines are naturally occurring fiber-reinforced composites. They consist of cellulose reinforcement oriented along the spine's major axis, which is suspended in an arabinan matrix. Both components are polysaccharides.¹ Spines of the *Opuntia ficus-indica*, known as "OFI" or "prickly pear cactus", are evenly composed of arabinan and cellulose.

Like most other fiber reinforced composites, cactus spines have extraordinarily high specific properties. Spines of the OFI cactus, for example, have an average flexural strength of

609 \pm 48.1 MPa, and an average elastic modulus of 28 \pm 3.66 GPa. For reference, glass fiber reinforced polymer (GFRP) composites have an average elastic modulus of 26 GPa.^{2,3} Average flexural strength data is not readily available for GFRP, although it should be comparable to its tensile strength, which is around 180 MPa, well below that of OFI spines. GFRP has a density of around 2 g/cm³, depending on the specific matrix material and volume fraction of the components.⁴ The spines of the OFI have an average density of 1.3 g/cm³, making their already impressive strength and stiffness even more impressive.² The strength and stiffness can be normalized to density-independent values, which puts their specific properties on the order of steel (Figure 1).

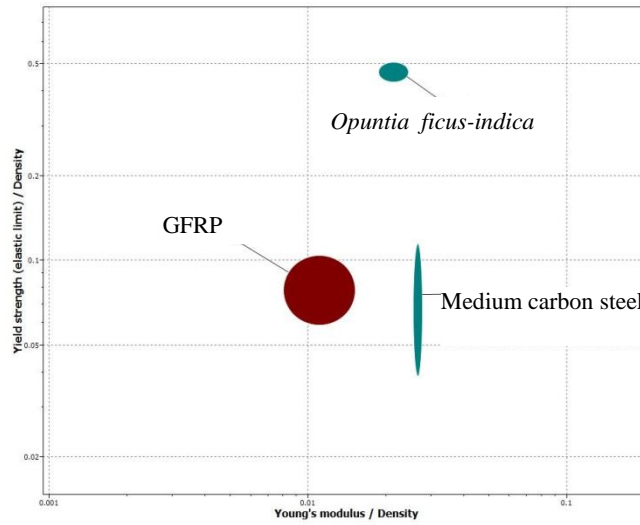


Figure 1. Graph of specific strength and specific modulus of OFI spines in relation to GFRP and medium carbon steel.

Spine structure is characterized by embedding the spines in epoxy, sectioning with a diamond knife and using incident light microscopy or scanning electron microscopy (SEM) to visually inspect the spines. Additionally, X-ray Diffraction (XRD) is used to investigate fiber orientation, fiber diameter and percent crystallinity, which has a loose correlation to flexural strength. The strongest spines tested by Cooper, Goldstein and Tarlton (2013) had the highest percent crystallinity, however; the rest of the spines did not fit this correlation. Percent crystallinity is calculated by taking the percent difference (Equation 1) between the intensity of the crystalline phase, I_{200} , and the intensity of the amorphous phase, I_{am} (Figure 2). I_{200} occurs at 22.5° and I_{am} at approximately 19°.

Eq. 1

$$\text{Percent Crystallinity (\%)} = (I_{200} - I_{\text{am}}) / I_{200}$$

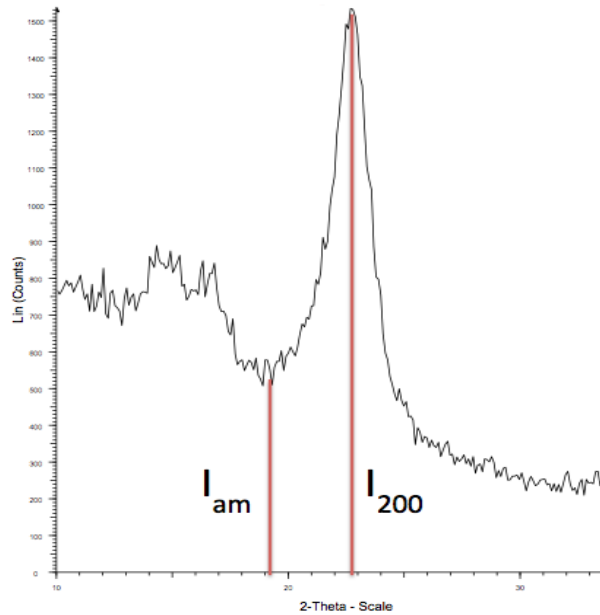


Figure 2. An XRD graph showing the amorphous and crystalline intensity values, I_{am} and I_{200} , respectively. The percent difference is calculated (Eq. 1), and is known as the percent crystallinity.⁵

Limited data exists for the cactus spines, and for some species there is not a large enough sample size to effectively determine the average properties, as shown by the variance within certain species, such as *Stenocactus multicostatus* (Figure 3). Additionally, Cooper et al. investigated 12 of the more than 2,000 species of cactus currently known to exist, each species purposefully selected because of the acicular shape of their spines.⁶ Current data is not representative of cactus spines as a whole, and only provides limited insight into the relationship between the structure and properties of cactus spines, however; it is useful to note that the mean elastic modulus of the various species of cactus spines ranged from roughly eight to nearly 30 gigapascals (GPa).

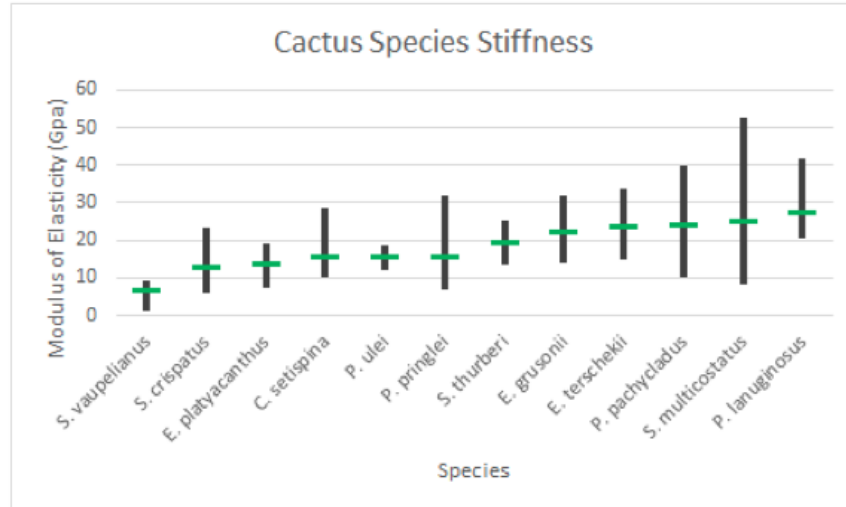


Figure 3. Elastic modulus of 12 different cactus species. Note the discrepancies between both modulus and range among the various species. *Stenocactus multicostratus* for example has a range of nearly 40 GPa, but a mean of only about 20, while *S. vaupelianus* has a modulus of about 8 GPa and a range of about 8 GPa.⁵

2.2 Cellulose

Cellulose is a polysaccharide and is also the most abundant organic polymer on earth. It is the primary structural component of many plant cell walls and exhibits strong crystalline character. It is highly hydrophilic, meaning it is attracted to water and is therefore weakened in its presence. The arabinan forms hydrogen bonds with the cellulose and ties up some of the hydroxyl groups that make cellulose so hydrophilic. Cellulose is a natural fiber that acts as the reinforcement, much like carbon or glass fiber in traditional composites. Figure 4 shows how cellulose is comprised of microfibrils containing nanofibrils, which are individual chains of cellulose.

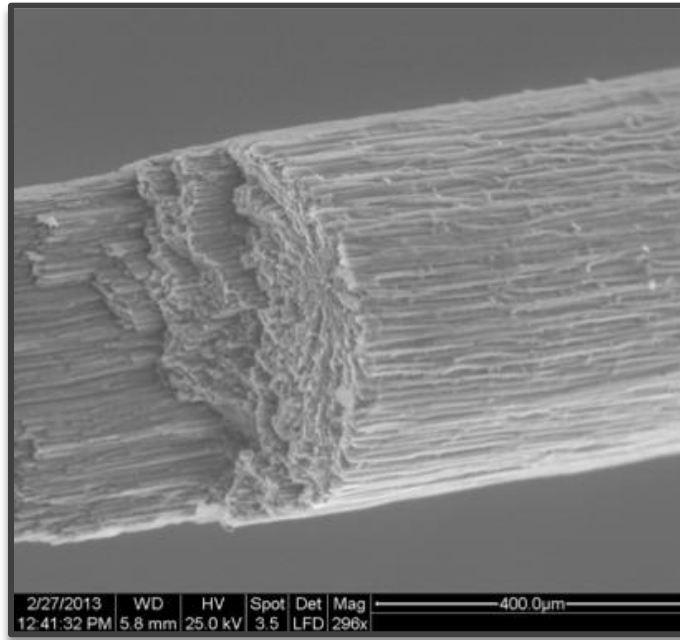


Figure 4. This image shows a scanning electron microscope view of a Mexican Giant Cardon cactus spine. Notice the continuous cellulose fibers running the length of the spine embedded within the arabinan matrix.

2.2.1 Percolation Theory

Unlike the cactus spines themselves, ArCCs are not standard fiber reinforced composites and therefore do not behave like them. Nanocomposites operate under the percolation theory model, whereby nanoscale particles, cellulose in the case of ArCCs, are dispersed throughout the matrix, and if there are enough particles bonding to their neighbors, they will form interconnected networks of cellulose, that will assume the load in all directions. The theoretical percolation threshold is around five weight percent cellulose.

2.2.2 Nanocrystalline Cellulose

For the cellulose networks to be as strong as possible, the particles must be fully crystalline. In normal cellulose nanofibril, there are lower strength, non-crystalline regions connecting the crystalline regions (Figure 5). Acid hydrolysis using sulfuric acid (H_2SO_4) selectively etches the amorphous regions, leaving regions known as *whiskers* in tact. These whiskers range from 10 to 100s of nanometers in length and 1 to 100 nanometers in diameter, with an aspect ratio of around 70. Cellulose whiskers have a theoretical Young's modulus of 65-167.5 GPa. This combined with their low density of 1.58-1.59 g/cm^3 yields a specific modulus of 67

GPa·cm³/g, almost double that of glass fibers. The theoretical tensile strength of cellulose whiskers is 13-17 GPa, which is among the highest of all natural materials.^{7,8} If the cellulose is not crystalline at the nano-scale, however; the composite will not exhibit properties requisite for a structural material.

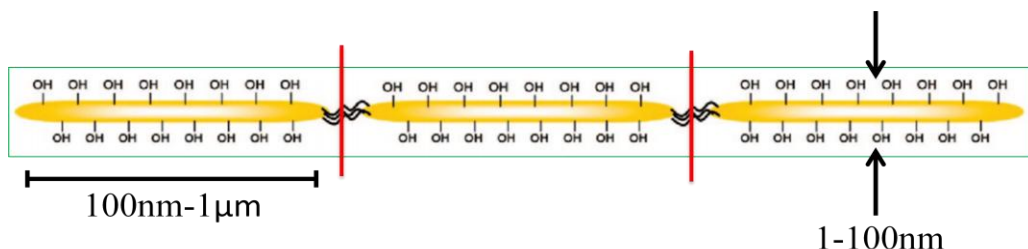


Figure 5. Schematic illustration of cellulose supramolecular structure with crystalline and amorphous regions. Hydroxyl groups are located on the microfibril surface.⁹

2.2.3 Prior Research

The previous research group sought to produce their own nanocrystalline cellulose (NCC) through acid hydrolysis. The whiskers are too small to be imaged properly by an electron microscope, but can be observed in solution by two main methods: Transmission Electron Microscopy (TEM) and Liquid Birefringence (LB). LB will determine if a colloidal suspension of nanocrystalline cellulose was created by showing the formation of a nematic liquid crystalline phase under cross polarized light.⁶ TEM would be able to directly measure whisker size however it requires a surface treatment of the whiskers with uranyl acetate, a mildly radioactive compound of depleted uranium, in order to provide elemental contrast. Both of these test methods were beyond the scope of this study due to a lack of time, funding and proper equipment training. Commercially available, and verified to specification, nanocrystalline cellulose whiskers were obtained from Celluforce, Inc.

2.3 Arabinan

Arabinan is a general term for polysaccharides, which are generally found as lateral side chains on pectin molecules. In the context of cactus spines, arabinan is mainly composed of arabinose.¹⁰ Pectins are polysaccharides commonly found in cell walls of plants. They are a highly complex heterogeneous group of polysaccharides composed of distinctive domains, which are believed to be covalently linked to one another. There is known to be a strong molecular interaction between the arabinan and cellulose, which is important because unmodified cellulose is highly hydrophilic and susceptible to water damage.

Pectins with arabinans attached have been documented in apples, sugar beets, rapeseed, apricots, tomatoes, carrots, cabbage, horse bean roots, onions and pears.¹⁰ The arabinan currently used in production of laboratory-derived arabinan-cellulose composites was purchased commercially at a cost of \$25/g, which limits the number of samples that can be tested. Sugar beets contain significant amounts of arabinan, which can be extracted as a less expensive alternative to commercially available arabinan.

Arabinan has the potential to solve the problem of water absorption in cellulose under the principles of conventional polymer reinforced composites. In fiber reinforced composites, the matrix serves several functions: Transferring load to the fibers, fixing the shape factor, protecting the fibers from abrasion and moisture, separating the fibers, and determining the thermal range of the composite. Pectins, like arabinan, “act as a binder between cellulose fibers and improve the mechanism of load transfer towards fibers when the sample is subjected to a mechanical stress. This is either via hydrogen bonding or covalent connections between hemicelluloses and cellulose fibers.”¹¹

3. Experimental

3.1 Fabrication

ArCC composites were fabricated with compositions of 0, 2.5, 15, 25, 50 and 75 wt% cellulose by dissolving NCC and arabinan powder into dimethylformamide (DMF). This was performed using an ultrasonic mixing machine as indicated by steps [1] and [2] in Figure 6. Ultrasonic mixing is an important step because cellulose is hydrophilic, and therefore it tends to form agglomerations, which are detrimental to the properties of the composite. Agglomerations are defects in the structure of the composite, where the cellulose come together and are not fully wetted by the matrix. Ultrasonic mixing ensures that individual cellulose whiskers are interacting with the matrix, a critical condition for the percolation model of reinforcement. The resulting solution was cast in a silicone mold and dried in a vacuum oven at 130 °F for 24 hours to remove the DMF, as seen in steps [3] and [4]. Step [5] was the removal of the composite from the mold in the form of a thin film. In step [6], the ArCC films were cut into 0.25” x 1.0” rectangular test coupons. Half of the coupons were then heat treated for an additional 17 hours at 170 °F prior to testing, while the rest were tested in the as cast condition.

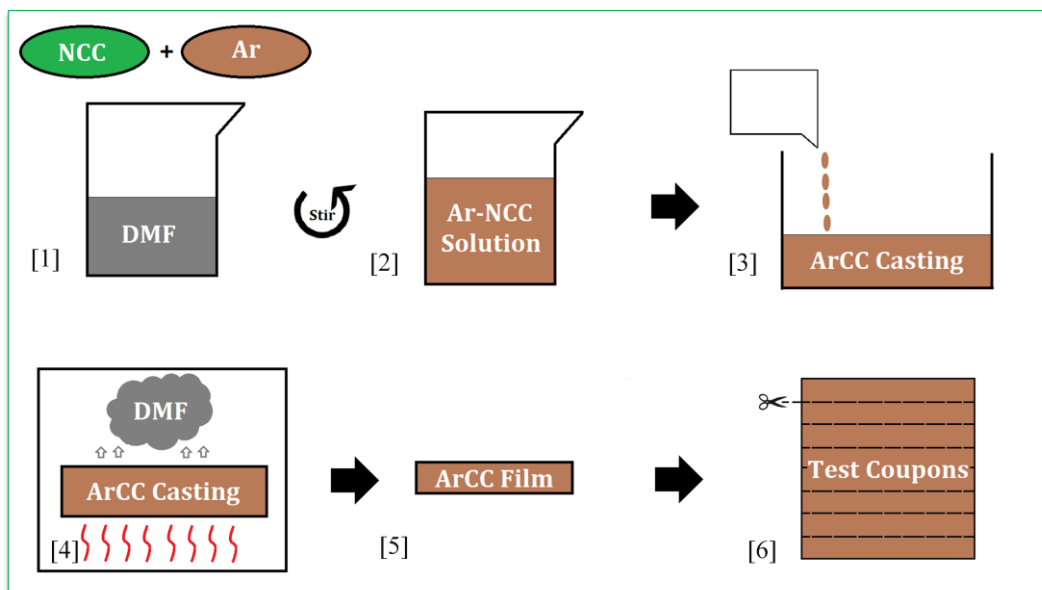


Figure 6. Diagram outlining the ArCC fabrication process.

Refer to **Appendix A** for standard operation procedure regarding composite fabrication.

3.2 Curing and Heat Treatment

Refer to **Appendix B** for standard operating procedure regarding composite curing and heat treatment.

3.3 Mechanical Testing

To test mechanical properties a simple tensile test was performed from 0-10% strain at 1% strain per minute. A Dynamic Mechanical Analysis, or DMA, machine was used to test our thin film samples due to its small grip size and low load capacity of 19 newtons. The ArCC samples would not fit in traditional tensile testing grips, as they were short, thin film coupons. The DMA output stress versus strain plots, which can be analyzed for various properties. Stiffness was analyzed because it is a bulk property, meaning that it will be the same value regardless of size or amount of material, and the properties of the thin films can therefore be scaled to the bulk materials. Stiffness is the ratio of stress divided by strain as known by Hooke's Law.

Refer to **Appendix C** for standard operating procedure for mechanical testing.

4. Results and Discussion

4.1 Properties

Stiffness values were calculated for all sample compositions, except for 75 wt% NCC. The NCC to arabinan ratio was too high, preventing the arabinan from fully wetting the reinforcement. This caused the samples to disintegrate upon curing. The 2.5 wt% sample did not exhibit a substantial increase in stiffness beyond pure arabinan. This suggests that the 2.5 wt% composition is below the percolation threshold as predicted. There were manufacturability issues with many of the compositions. 50 wt% appeared to be the ideal composition for quality of fabrication and maximization of stiffness. This became the focus of testing.

Stiffness is seen visually in our plots as the slope of each line. A dramatic increase in stiffness with heat treatment was clearly seen in both pure arabinan and arabinan-50 wt% NCC (Figure 7).

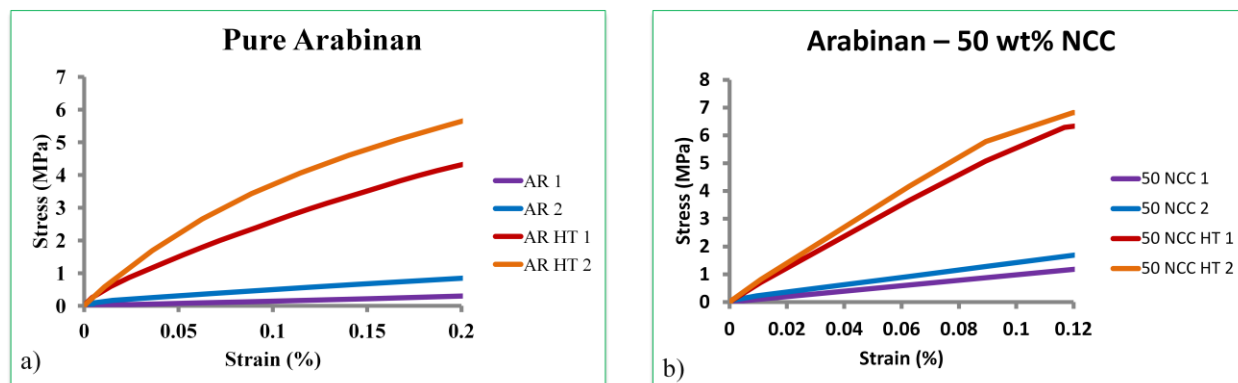


Figure 7. A simple tensile test was performed at 0-10% strain at 1% strain per minute. For both compositions as cast and heat-treated samples were tested. Image a) shows the linear elastic region of the stress-strain curve for Pure Arabinan, focused on 0-0.2 percent strain. Image b) shows the linear elastic region of 50-wt% cellulose in an arabinan matrix, focusing on the 0-0.12 percent strain.

Average stiffness values were calculated in GPa for the pure arabinan and arabinan-50 wt% NCC samples in both the as cast and heat treated (HT) condition (Table I). A fourfold increase in stiffness was seen between the pure arabinan and arabinan-50 wt% NCC. This confirmed that NCC was an effective reinforcement component. The pure arabinan sample exhibited approximately an order of magnitude increase in stiffness with heat treatment. Furthermore, the stiffness of arabinan-50 wt% NCC increased by about a factor of six with heat treatment. This proved that heat treatment was an effective processing technique for increasing stiffness. This

performance is promising because the arabinan-50 wt% NCC sample is approximately three times more stiff than polyethylene terephthalate (PET), a common packaging material. ArCC stiffness values are approaching that of glass fiber reinforced polymers (GFRP). The ultimate goal is to reach values of representative cactus spines found in nature, such as the *Opuntia ficus-indica* (OFI), or Prickly Pear.

Table I. Stiffness Values of ArCC Samples Compared to Reference Materials

Material	Stiffness (GPa)
Pure Arabinan	0.26
Pure Arabinan , HT	3.24
Arabinan – 50 wt% NCC	1.09
Arabinan – 50 wt% NCC, HT	6.09
PET	2.4
GFRP	17
Dry OFI Cactus Spine	33

4.2 Heat Treatment Effects

Previous research in Differential Scanning Calorimetry (DSC) of cactus spines caused a subsequent, unforeseen increase in stiffness among ArCCs. This indicated heat treatment as a possible stiffening process. The current procedure is occurs at a temperature below both the glass transition temperature (T_g) of arabinan and the melting point of cellulose. A representative time of 17 hours was chosen to allow for adequate heat transfer into the composite. The nature of the stiffness increase, however, is unknown. In order to optimize the heat treatment, the mechanism behind the increase must be further investigated.

4.3 Supplementary Testing

It was believed that the stiffness increase after heat treatment was due to an increase in the crystallinity of arabinan. This hypothesis stems from the correlation between that strongest cactus spines having the highest crystallinity as observed by Cooper et al (2013). Additionally, the presence of nanocrystalline cellulose could have provided heterogeneous nucleation sites for

the arabinan crystals to grow upon during heat treatment. The high surface area to volume ratio of the cellulose whiskers provide many interphase interfaces, off of which new solid arabinan crystal phases could nucleate and grow.

4.3.1 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to investigate the fracture surfaces of pure arabinan and arabinan-50 wt% NCC samples (Figure 8). The films were fractured after a liquid nitrogen quench to achieve an optimal fracture surface. Image a) shows a glassy, brittle fracture indicative of low energy absorption. In contrast, image b) shows the convoluted fracture surface of arabinan-50 wt% NCC indicating the absorption of far more energy. This is evident by the long fracture paths and multiple layers of features visible. Additionally, there was no evidence of cellulose agglomeration, proving that the ultrasonic mixing procedure was successful in dispersing the cellulose whiskers. These observations correlate well with the results from DMA, confirming that pure arabinan was far less stiff than arabinan-50 wt% NCC.

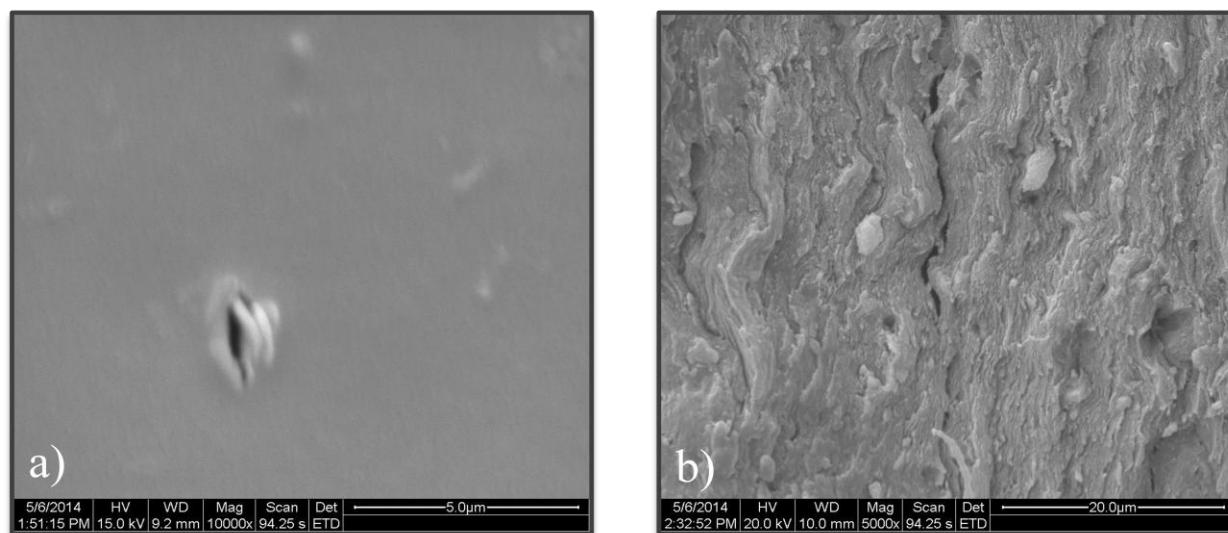


Figure 8. SEM images of ArCC fracture surfaces. Image a) is of pure arabinan and image b) is of arabinan-50 wt% NCC.

4.3.2 X-Ray Diffraction

It was believed that the stiffness increase observed by DMA after the heat treatment process was due to an increase in the crystallinity of arabinan. X-Ray diffraction (XRD) was used to investigate the arabinan crystallinity of pure arabinan and the arabinan-50 wt% NCC in both the as cast and heat treated conditions. Arabinan has crystalline peaks in a small range

between 14° and 17.3° 2θ . The samples showed diffuse, rather than defined peaks over this range. Therefore, Eq. 1 cannot be applied to the arabinan region. Cellulose, on the other hand, exhibits a strong peak at 22.5° 2θ . Figure 9 shows the XRD plots both samples with the as cast condition in black and the heat treated condition in red, however, no increase in peak intensity was observed in either condition, indicating that no increase in crystallinity of arabinan occurred. The arabinan-50 wt% NCC sample XRD graph showed a crystalline peak for cellulose as expected. These results suggest that arabinan crystallization is not responsible for the dramatic stiffness increase produced by heat treatment.

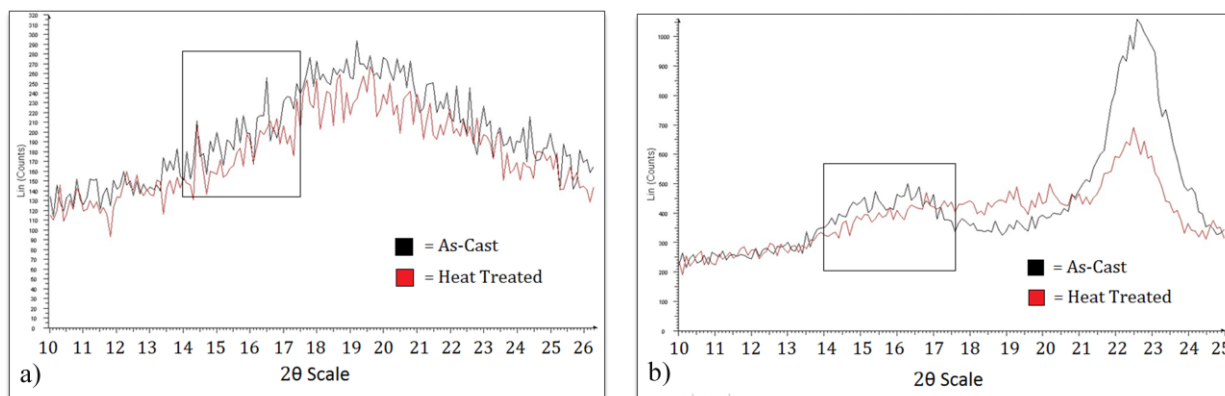


Figure 9. Images showing the scan results of XRD testing. Image a) is of pure arabinan and image b) is of arabinan-50 wt% NCC. The black boxes highlight the diffuse peaks for arabinan from 14 - 17.3° 2θ .

5. Conclusion

This project set out to fabricate natural nanocomposites inspired by cactus spines. This material is of great interest due to its biodegradable and renewable nature. ArCCs have already surpassed the stiffness of PET, and are approaching that of fiberglass and dried *Opuntia ficus-indica* cactus spines. This shows great promise in its use as a packaging material to replace conventional unsustainable materials. As strength and stiffness properties are optimized, less material can be used leading to even greater savings. As future groups continue this research, the goal is to replicate what nature has already perfected in the cactus spine.

6. Recommendations for Future Studies

In continuation of this project, supplemental testing should first be addressed. DSC must be performed to confirm the XRD results regarding crystallinity and stiffness increase from heat

treatment. If confirmed, the mechanism of stiffness increase must be determined in order to optimize the desired properties. Additionally, the glass transition temperatures (T_g) of arabinan and the composites themselves can be determined via DSC. This information will provide insight to the optimization of the heat treatment procedure. The current HT time of 17 hours is not substantiated by research.

The fabrication procedure could be improved upon in several ways as well. The solvent DMA needs more containment to minimize operator exposure. Ultrasonic mixing should be performed under a fume hood to capture particulates generated through the process. Some type of exhaust or ventilation system needs to be coupled with use of the vacuum oven. When removing samples after curing, vaporized DMF enters the room creating a health hazard.

The Chemistry department's DMA machine was not designed for simple tensile testing and has resulted in some experimental difficulties. Increasing coupon size would allow for testing on the Materials Engineering department's own low load cell, which requires roughly three inch long coupons. Additionally, the low load frame would be located within the department, allowing for more frequent and faster testing with greater sample size, n . A larger number of samples will allow for an increase in statistical significance of the stiffness data. Having statistically significant data will bring greater meaning to the data. For example, a doubling in stiffness between compositions could be significant or merely a function of the variance within the data.

In order to accomplish this, a new casting procedure will also be needed to allow for the larger coupons. The current silicone molds limit the coupon size to one square inch. A custom mold needs to be investigated. A metal or polymer die could be manufactured in house to facilitate the new coupon geometry. Furthermore, this could be coupled with a flat and flush cover to maintain a uniform surface finish. The silicone mold would create samples with uneven cross-sectional thickness because it was open to the atmosphere, leading to testing difficulties, such as premature fracture. Compositions with less solvent to solid ratio tended to be more viscous, and gelled, which made casting them uniformly very difficult. However, these ratios cured better, with less defects.

Lastly, the price of arabinan and cellulose raw material restricts the amount of samples that can be made. The acid hydrolysis technique should be optimized so nanocrystalline cellulose can be produced in house. Extraction of arabinan from sugar beets should also be explored. This will dramatically drive down the cost of the materials, as a single thin film sample can cost up to \$50.

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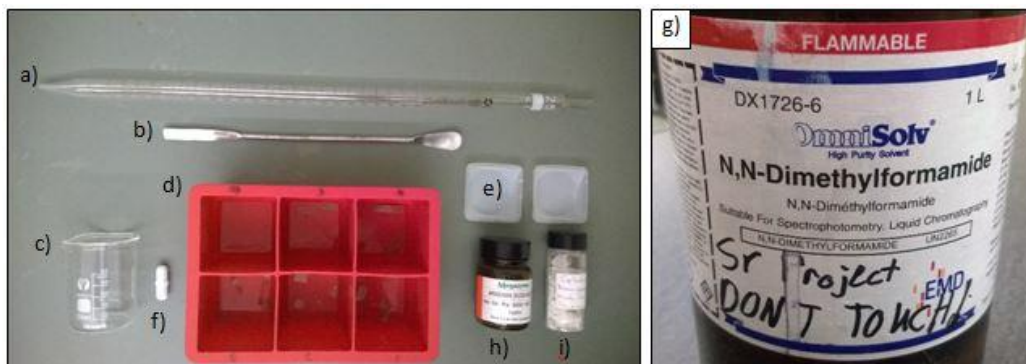
8. Appendices

Appendix A: Fabrication

Composite Fabrication Procedure

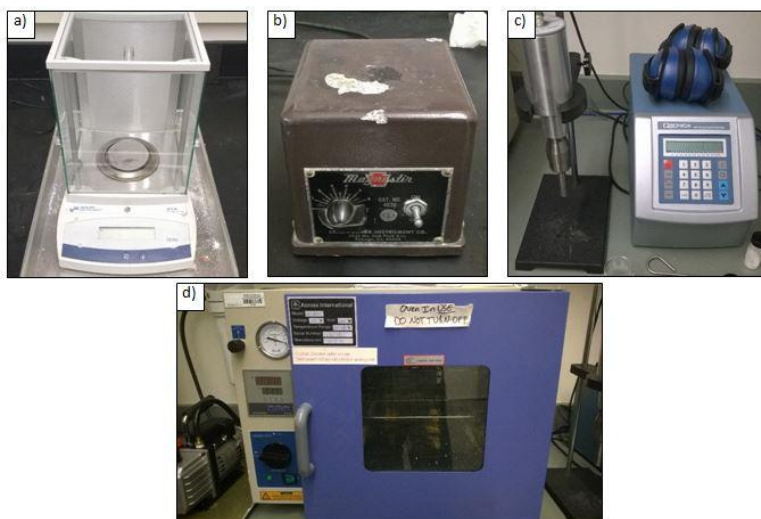
1. Materials

- a. 25 mL pipette
- b. Flat spoon
- c. 50 mL beaker
- d. Silicone mold
- e. Plastic weigh boats
- f. 1" magnetic stir bar
- g. N,N – Dimethylformamide (DMF)
- h. Megazyme: arabinan (Ar)
- i. Cellulforce: Nanocrystalline cellulose (NCC)



2. Equipment

- a. Analytical balance: 0.0001g resolution
- b. Stir plate
- c. Sonicator
- d. Vacuum oven



3. Method

- a. Determine the desired weight percent composition of the composite (e.g. Ar-35wt% NCC)
- b. Based on 1g of total dry material (arabinan + NCC), calculate mass of each component (e.g. Ar-35wt% NCC: $0.6500\text{g arabinan} + 0.3500\text{g NCC} = 1.000\text{g total}$)
- c. Weigh out components into separate plastic weigh boats using the analytical balance. NOTE: Reduce material loss by putting material only in the corner of weigh boats.



- d. Place a 50mL beaker with a 1" stir bar inside on the balance and press "ZERO".
- e. Use the 25mL pipette to measure out DMF in a 10:1 ratio to the dry components. (e.g. 10g DMF for 1g of total dry material)



- f. Place beaker on brown stir plate and start at the slowest speed possible
- g. While stirring, pour NCC, then Ar powder into beaker, avoiding the top of the stir bar. Stir for 3 minutes.
- h. Tape beaker to base of ultrasonic mixer stand.
- i. Position ultrasonic mixing head close to bottom of the beaker, without touching. NOTE: Ultrasonic mixer can shatter beaker if they come in contact.
- j. Set ultrasonic mixer to pulse 3 sec on, 3 sec off and run cycle for 5 min. NOTE: Sonicator will not turn off automatically, even when using the timer. CAUTION: Ultrasonic mixer produces significant heating, sample will be hot
- k. Pour sample into one of six pockets in silicone mold

Appendix B: Vacuum Oven Operation

Standard Cure and Vacuum Oven Operation

1. Preheat Vacuum oven at least 30-45 minutes prior to curing
 - a. Set to 130 °F using arrow buttons, see figure
 - b. Temperature will ramp past 130 °F and then stabilize
2. Once stabilized, place silicone mold in oven
3. Pull a vacuum to 0.7 MPa
 - a. Turn to close *Vacuum Release Valve*, see Figure A
 - b. Open *Vacuum Valve* (Figure A)
 - c. Turn on vacuum pump with switch located on the back
 - d. Once gauge reads 0.7 MPa, close *Vacuum Valve*
 - e. Turn off vacuum pump

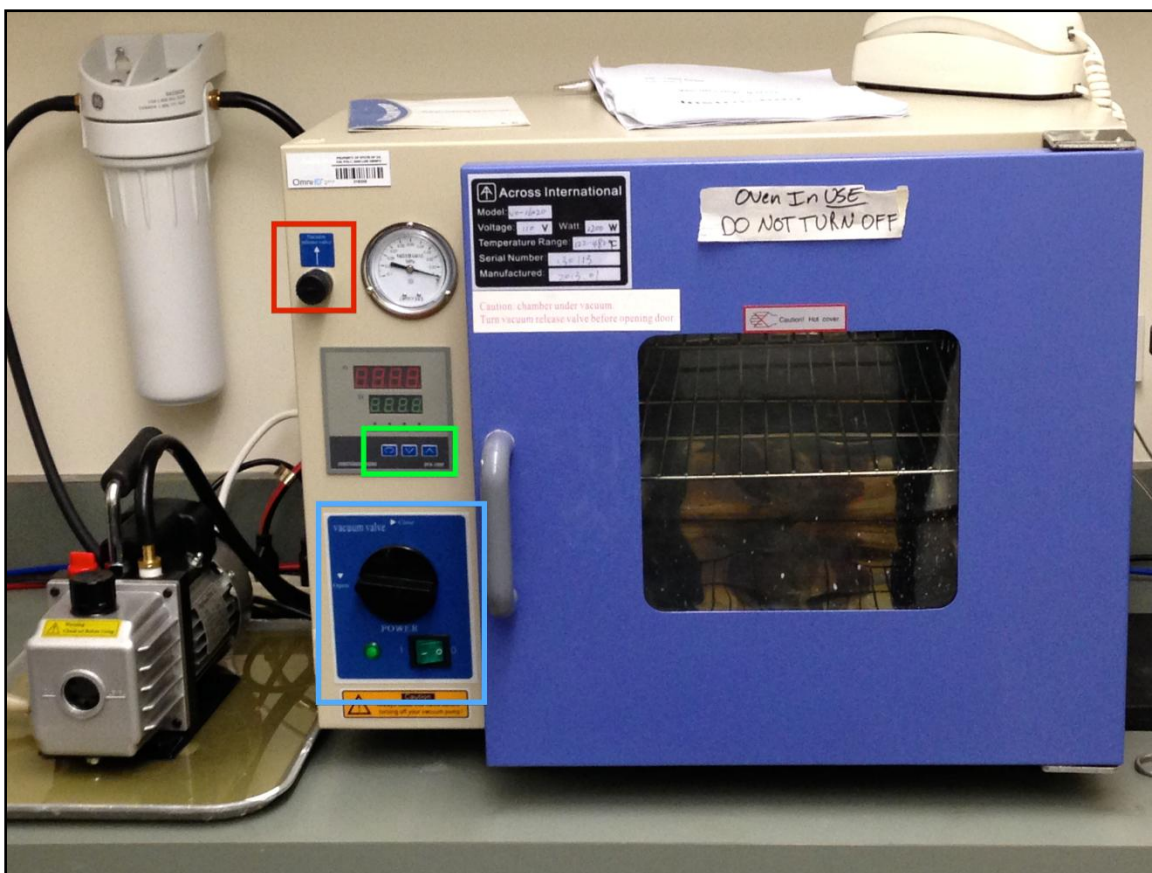


Figure A. Vacuum Oven: Highlighted are the key features, Vacuum Release Valve (Red), Temperature Buttons (Green) and Vacuum Valve (Blue).

4. Allow Samples to cure for 24 hours in oven
5. Remove sample
 - a. Turn to open *Vacuum Release Valve* (Figure A)
 - b. Once vacuum is released, open oven and remove samples
 - c. Note: Temperature may far exceed target before slowly returning
6. If sample is fully cured, turn off oven

- a. If not fully cured, return to *step 3*, and leave in oven for shorter increments of time, depending on degree of cure

Heat Treatment Procedure

1. Follow *Standard Cure and Vacuum Oven Operation* with several exceptions:
2. Preheat oven to 170 °F instead of 130 °F
3. Place pre-cut test coupons (0.25" x 1") in petri dishes with lids upside-down on top of samples to prevent warping
 - a. Note: Make sure to pre-cut the coupons because samples are much harder to cut after heat treatment
4. Allow to sit in oven for 17 Hours instead of 24

Appendix C: Mechanical Testing

DMA Operation

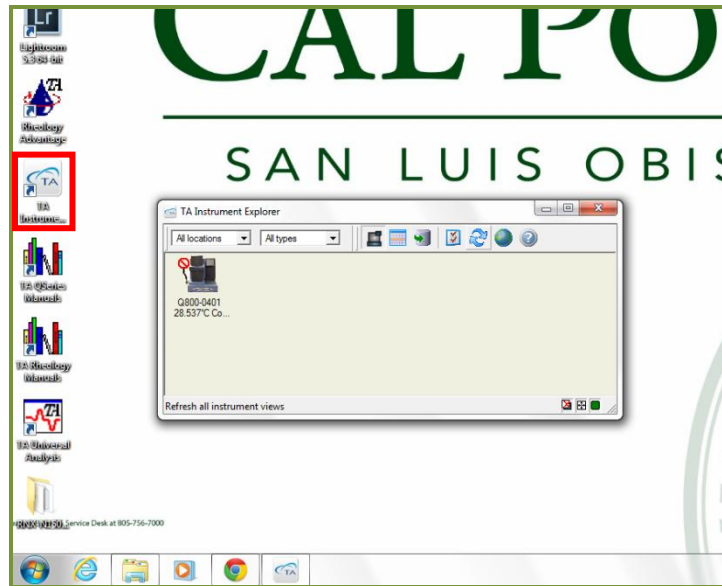
1. Turn on Dynamic Mechanical Analysis (DMA) machine by setting power switch to “I”, found on back right side of the machine [Red Box]



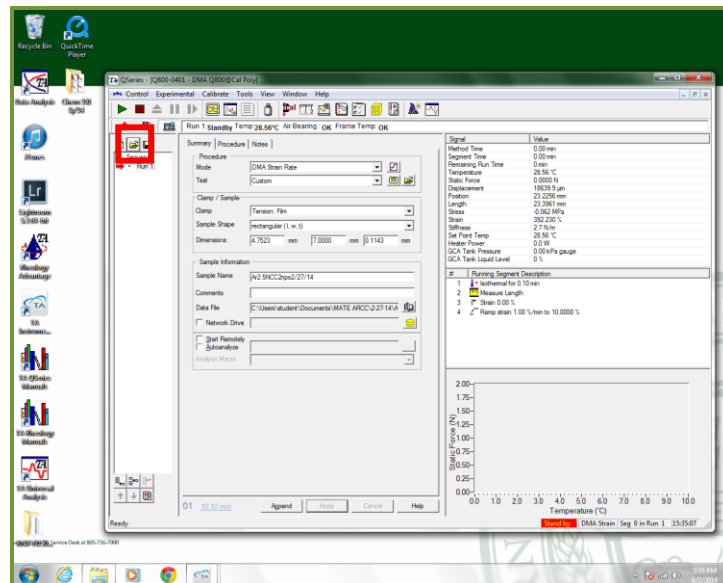
2. Open Nitrogen tank valve, found directly on top of tank, until gauges stabilize
 - a. Do NOT adjust the regulator [Red Circle]
 - b. Consult lab technicians for proper pressure and tank levels/refilling
 - c. Close tank when finished testing



3. Turn on computer
4. Open *TA Instrument Explorer* found on the computer desktop [Red Box]
5. Double click *Q800* icon to connect to the DMA machine
 - a. If red circle logo is visible on the icon, click refresh [Blue Arrow Logo] until it disappears, then double click icon
 - b. This opens the *QSeries Test Control Window*



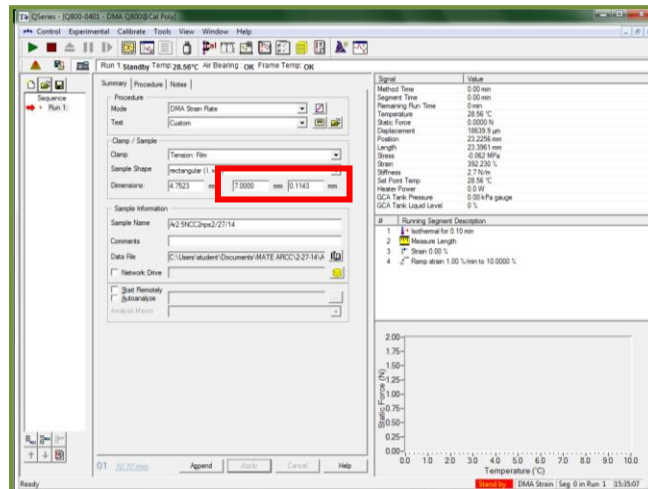
6. Click Open File icon to load *DMA Tensile Procedure* [Red Box]
 - a. File located under: Desktop→Libraries→Documents→MATE ARCC
 - b. Open *DMA tensileprocedure.seq*



7. Verify sequence steps in the *Running Segment Description* box are as follows:
 - a. Isothermal for 0.10 min
 - b. Measure Length
 - c. Strain 0.00%
 - d. Ramp Strain 1.00%/min to 10.00%
8. Section composite sample into 0.25" x 1.0" coupon using scalpel
9. Measure width (w) and thickness (t) of coupon in millimeters using micrometer



10. Input width and thickness in proper *Dimensions* boxes [Red Box]
 a. Note: Length is auto-measured by machine



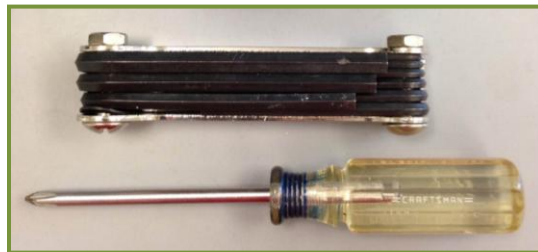
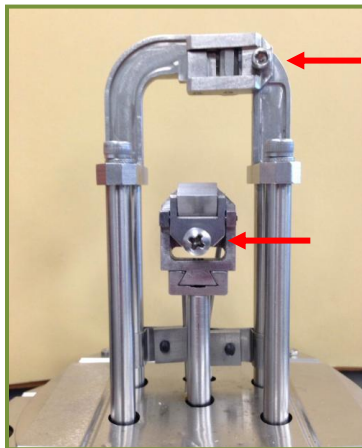
11. Press *Control Menu* on the DMA touch screen [Red Box]



12. Press *Furnace* to open the DMA test chamber [Red Box]



13. Press *Drive* to free clamp movement
14. Load sample in clamps
15. Press *Drive* to lock clamp in place
16. Tighten clamps with allen wrench and screwdriver [Red Arrows]
 - a. Do NOT over-tighten clamps as this can fracture the sample



17. Press *Furnace* to close test chamber



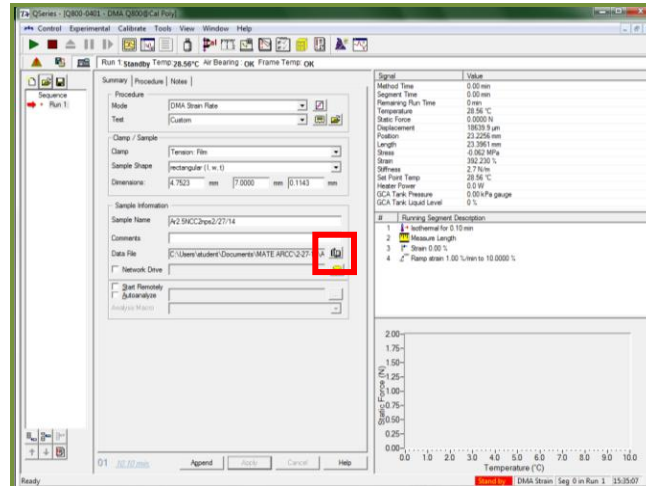
18. Input *Sample Name* according to the format:

[“Matrix” - “wt%” “Type” - “Heat Treat” - “Test #” “Tester’s Initials” “Date”]

HT Example: AR - 35 NCC - HT - 2 ABC 4-25-14

Non-HT Example: AR - 50 NCC - 1 XYZ 3-20-14

19. Click on *Book Icon* to the right of the *Data File* line [Red Box]

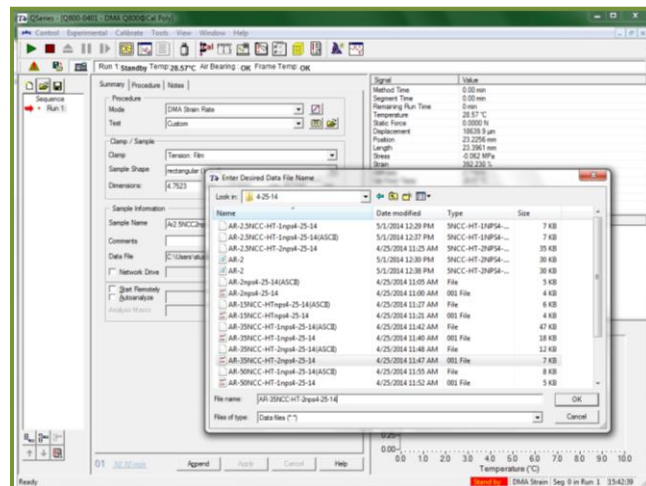


20. Title the *File Name* the same as the *Sample Name*

21. Save the file in a *new folder* within the *MATE ARCC* folder named as the current date

a. For example: 5-10-13

22. Click *OK*



23. Verify all dimensions, file names, and parameters

24. Press the green triangle “play” button to start the test run

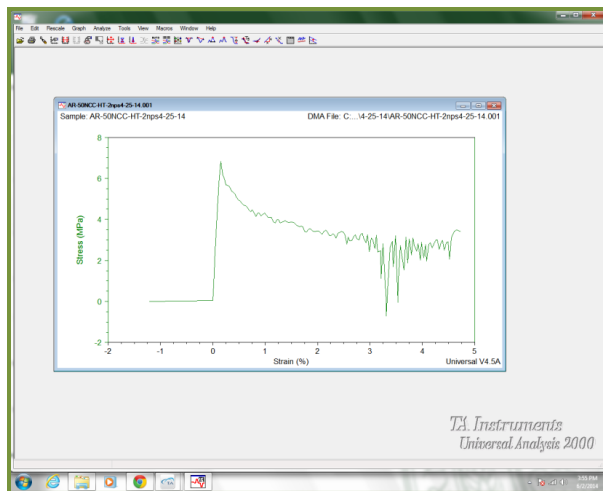
25. If the sample breaks or the run ends, press *OK* to clear the menu



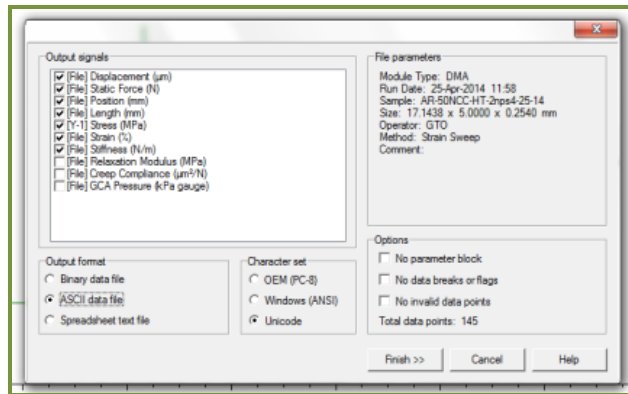
26. If the Nitrogen gas continues to flow, press *Control Menu*, then press *Air Cool* to stop
 - a. Note: Nitrogen flow can be heard as an indication
27. Press *Furnace* to open test chamber
28. Use allen wrench and screwdriver to remove sample
29. Test file is auto-saved to the specified location

Data Analysis

1. Open *TA Universal Analysis* icon found on the desktop
2. Click on the *Open File* icon to load DMA test file data
3. Change *Signals* to: $Y1 = \text{Stress (MPa)}$ and $X = \text{Strain (\%)}$
4. Click *OK* - This displays the stress vs. strain plot of the DMA test



5. Click *File* → *Export Data File* → *File and Plot Signals...*
6. Check all boxes except *Relaxation Modulus*, *Creep Compliance*, and *GCA Pressure* under *Output Signal*
7. Select *ASCII data file* as the *Output Format*



8. Click *Finish*
9. Title the file the same as the *Sample Name*, but with “(ASCII)” at the end

Example: AR - 50 NCC - 1 XYZ 3-20-14 (ASCII)

10. Save file in the same folder as the original DMA test file
11. This ASCII data file can be opened in Notepad and Excel
12. Email the file to the desired computer
 - a. Note: No flash drives are allowed in the CHEM labs

Data Correction

The grips on the DMA machine have an offset built into them. When the clamps are touching, the machine does not register zero for the displacement. This introduces an error into the sample length data collected by the computer. Through measuring samples before and after tensile testing, it was determined that the DMA grip offset is approximately 4.7mm. Therefore, the collected data must be adjusted in order to obtain proper results. See basic instructions below.

1. Open the *ASCII* data file in Excel
 - a. The raw *ASCII* data is compiled in one column
2. Select the entire column of data and click *Text to Columns*, under the *Data* tab
 - a. This opens the *Convert Text to Columns Wizard*
3. Click *Delimited*, then click *Next*
4. Select the *Space* option under *Delimiters*, then click *Next*
5. Click *Finish*
 - a. Data should be separated into individual columns
6. Re-label columns appropriately

Strain is calculated as the change in length divided by original length, or $\Delta L / L_0$. Since the DMA machine incorrectly measures the length of the sample, the offset must be added to L_0 . The new equation for *adjusted strain* should be $\epsilon = \Delta L / (L_0 + 4.7)$. Plot stress vs. *adjusted strain* for proper results.