EFFECT OF HEAT TREATMENT ON THE MECHANICAL PROPERTIES OF NATURAL BAMBOO

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ABSTRACT

Previous studies have shown that heat treatments have the ability to improve mechanical strength and stiffness in hardwood species. Compared to structural hardwoods, bamboo is a more sustainable and globally viable renewable resource. Therefore, *Bambusoides vivax* (timber bamboo) culms were fabricated into flat, rectangular cross-section samples with varying cellulose content to be heat treated and tested for mechanical properties. All samples were heat treated (HT) twice. The first HT was a normalization of all samples at 103 +/- 3 °C. The second HT was performed at various times and temperatures ranging from 130°C to 170°C for 0.5 to 3 hours. Post HT, the samples were loaded until failure in a three-point bend apparatus on an Instron 150 kN load frame based on the specifications of ASTM D790-03. The specimens were loaded to fail under tensile stress along the fiber-dense side of the bamboo sample, which was seen to reduce variance in earlier testing. From initial pilot testing, an increase in flexural strength of 70% and an increase in relative flexural stiffness of 30% were seen in samples that had undergone 1 hour HTs at 103 +/- 3 °C and 160 +/- 3 °C compared with samples that had not undergone HT. Formal testing displayed an average flexural strength of 200 MPa and an average flexural stiffness of 50 MPa compared to the non-heat treated samples which displayed a 100 MPa flexural strength and 30 MPa flexural stiffness. No statistically significant difference was determined between the different heat treatments, although all heat treated samples performed to statistically higher degrees than the non-HT samples.

1. INTRODUCTION

1.1 Stakeholders

The results of this experiment impacts industries which utilize bamboo as a composite material for a range of structural and architectural applications. The stakeholders include those who invest in green building materials and whom desire to improve the frequency that these materials may be used for a wide range of composite applications.

1.2 Broader Impacts

Bamboo is a highly renewable resource that has accumulated a significant amount of attention in the 21st century. As designers, manufacturers, and engineers attempt to create more eco-friendly products, bamboo has been at the forefront of the spectrum for materials in use. Bamboo has several qualities that are favorable for its use in design applications. In the realm of natural materials, it has a low embodied energy, rapid production time, and incredible mechanical properties.

This project aims to optimize the stiffness and modulus of rupture in bamboo species by controlling the phases and composition of bamboo's microstructural constituents. The constituents of interest are cellulose, hemicellulose, and lignin. Although these constituents are similar to the constituents of wood, the natures of their interactions are different in bamboo. There is currently a limited amount of information available about the effect of heat treatments on altering the microstructure of bamboo. Our goal is to fill this void by collecting a range of data on the effect of heat treatment times and temperatures on the microstructure composition and % crystallinity, relating to changes in mechanical properties. If the properties of bamboo can be significantly improved by simple heat treatment, there are multiple positive implications. The heat treatment has potential to improve the longevity of the material, reducing water absorption as well as increasing the resistance to biological attack. This would allow the bamboo, and composites made from the bamboo, to have less toxic additives and preservatives. Also, if the lignin and hemicellulose matrix could be removed from the cellulose fibers, it might be possible to create complex shapes that traditional bamboo structures were previously unable to make.

With these improvements in properties, the applications available to bamboo composite materials increases as well. The theoretical strength of cellulose is similar to that of glass fibers, which could mean the replacement of discontinuous glass fiber composites with a renewable, environmentally friendly, and energy efficient material. Glass fiber composites have many applications in the automotive industry, as well as for products such as poles, panels, boat hulls, and honeycomb applications. There are almost 1,500 species of bamboo, each with varying size, growth rate, and microstructural constituents. With fiberglass rods (fishing poles, bulk fiberglass poles, etc) , bamboo rods could be heat treated without further manufacturing into panels or

complex shapes, which would decrease the cost substantially while eliminating the need to make rods with the more costly fiberglass.

There are possible implications for structural applications using bamboo as a building material. In developing countries and countries with natural bamboo growth, bamboo is the go-to option for simple structures. For example, in rural regions of China, entire villages are built from bamboo structures. Bamboo can be used for decorative and structural purposes, with applications ranging from raised flooring to entire building skeletons and sheathing. If the properties were improved, the safety and lifetime of these structures could be improved, increasing the quality of life with more trustworthy infrastructure. The tug between raw bamboo and treated bamboo may be difficult in developing countries. It is likely that no time and resources will be available for treatment. If the results of this project do show a significant amount of mechanical property improvement, then the demand may begin to shift towards bamboo treatment.

With a broad reach, if the mechanical properties of stiffness and modulus of rupture can be improved through simple heat treatment in bamboo samples, the resulting impact may span with applications in green building materials, composites, renewable material replacement, and multiple environmentally positive alternatives.

1.3 Design Constraints

There are two factors that constrain the use of heat treated bamboo. The first is the variance in properties displayed by natural materials. Since natural materials are not manufactured from purified initial materials which undergo a set series of processing, the properties displayed can have a wide range based on a range of factors including species, agriculture locations, drying conditions, and manufacturing conditions. The second significant factor is the size scale and investment for heat treatment. Countries that utilize bamboo as whole-culm structures would likely not have the capital to invest in large furnaces that could heat treat full bamboo culms. Heat treated bamboo would most likely be used for composite applications where the natural material could be chopped or shredded into a bulk material prior to heat treatment. This includes fiber-reinforced matrix composites, glue-laminated materials, and architectural flooring and paneling among other engineered wood applications.

2. BACKGROUND

2.I - Why use bamboo?

According to the Global Footprint Network[1], the world population currently uses the equivalent of 1.5 planets to provide our resources and absorb our waste. Compare this to a value of 0.75 planets in 1960. The value will continue to increase, with a predicted value of 3 planets by 2050 if the global population continues at the current rate. This is the definition of non-sustainable. Our resources will decline at an increasing rate until a global shift occurs. This scenario suggest a need for an increase in the use of sustainable materials. One of the main reasons that bamboo is a desirable sustainable material is its capacity to grow at extremely fast rates and in diverse locations worldwide.

David Farrelly[2] writes that bamboo is known as the fastest growing plant species with larger species averaging between 3 to 16 inches per day, and a recorded growth for *Phyllostachys eulis* in Japan of 47.6 inches in one 24 hour period. They generally reach their maximum height in a month of fast growing during the wet season with little growth after that, other than some branch and foliage growth. Bamboo diameters range in size from 0.5 inches to almost 6 inches in some species, with height ranging from less than a meter to more than 30 meters high.

Lugt et al.[3] (2006) determined that bamboo has positive sustainability aspects compared to wood through the use of LCA. Above all else, they found that the high annual yield of bamboo, the ability to grow bamboo in non-traditional locations, and its durable and continuous root structure, makes it a highly sustainable material for local use. It can grow on slopes and areas where the foresting of wood is not possible. It can replace tropical hardwood, mitigating the decrease of tropical forest area. It can support local economies in the third world. Bamboo is a sustainable solution for local applications, although transportation impacts and costs decrease the level of sustainability. A large issue is the high volume to weight ratio since the bamboo is hollow. Competitive advantage of bamboo use (for sustainability) depends on the application. The application must take advantage of the hardness, bendability, aesthetic properties, and outside durability (impact and environmental). The LCA determined that local use of bamboo for a structural beam has an relative eco cost of 0.086/kg compared to steel at 0.487/kg. Transported bamboo from China to Europe, however, has an eco cost of 0.89/kg.

Lugt et al.[4] (2006) concluded that based on environmental and financial comparison, bamboo can compete with building materials commonly used in Western European countries such as timber, concrete, and steel for certain applications. Suggested applications for bamboo culm are for functions where structural requirements are not precise, due to the inescapable irregularity of structure in natural materials. These possible applications include replacement of wood in oriented strand board (OSB), particle board, flooring, indoor and exterior laminates, and wood composite applications

Huda et al.[5] (2005) concluded that cellulose-reinforced biobased PLA composites have mechanical properties that can compete with conventional thermoplastic composites. This study

is based on recycled cellulose. It gives confidence that the use of bamboo particles in a matrix could improve the properties further due to their high strength compared to a recycled cellulose.

2.II Relationships Between Structure, Chemistry, and Mechanical Properties of Bamboo and Similar Woody Materials

2.II.i - Structure and chemistry of bamboo

According to Lucas et al.[6] macro-structurally, there are two main elements of structure in the bamboo culm wall. These are defined as fibers and the woody matrix that surrounds them. The strength of bamboo comes directly from the stiff fibers that run the length of the culm, supporting the weight of the structure and providing resistance to external stimuli like wind and rain. The woody matrix that surrounds the fibers makes up the majority of the mass of bamboo, and provides the compressive strength that is common with all woody plants. The matrix also helps transmit the applied forces directly to the structural fibers through the dense bonding networks between the two structural elements.

The yield strength of bamboo is directly correlated with fiber content, with increased fiber content having higher yield strength. Fiber content is not constant through the bamboo culm like it is in other woody structures, mainly because the growth of bamboo is primarily upward, rather than upward and outward like trees and other woody plants. This forces each bamboo culm to have a very specific fiber content variation with location. Fiber content increases from the inside of the plant wall to the outside, which makes the outside ring of the culm extremely hard and tough, increasing its strength. This is slightly similar to the growth rings in some softwood trees, where the growth rings are made up of earlywood and latewood which have different mechanical properties.

According to Shmulsky et al.[7] the earlywood in softwoods is fast growing and forms during the wet season making it structurally weaker than latewood, which forms slower during the dry season and is more densely fibrous than earlywood. In softwoods the transition from earlywood to latewood is not instantaneous like it is in hardwoods. The gradual transition of fiber density makes softwood yearly growth rings similar in structure to bamboo. The main reason in the difference in fiber content in bamboo is to reduce weight by having strength only on the outside of the plant, which allows for water transportation through the inside of the plant walls where it is less dense with fiber content. Figure 1 below displays the fiber gradient in a culm wall along with the increasing fiber density highlighted by the arrow.

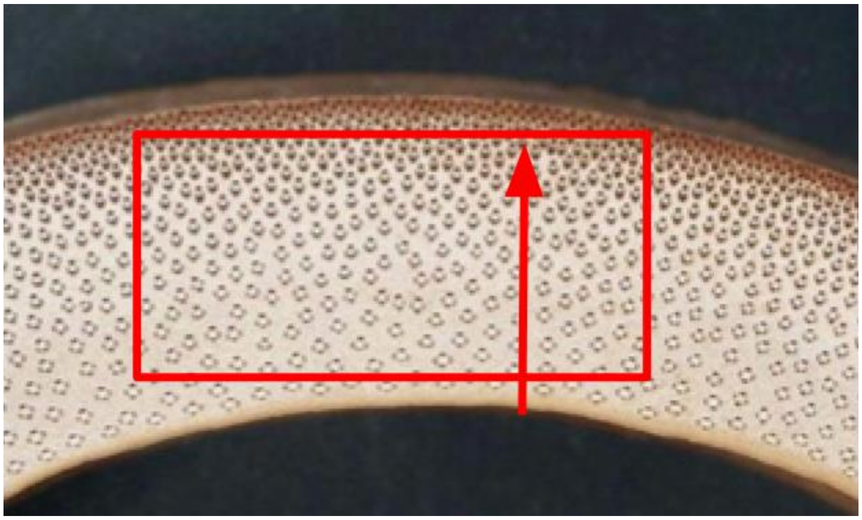


Figure 1. Gradient in fiber density through the culm wall of a bamboo stalk.

According to Lucas et al.[6] fiber content increases from the bottom to the top of each culm internode as well. This is due to the fact that each nodal crosslinking barrier is more connected to the culm above it than the one below it, which leads to a reduction in strength between internodes (Figure 2)[6] . The crosslinking node makes up for the lack in strength due to reduced fiber content. The increased fiber content at the top of each culm increases the total stiffness of the connection between the top of one culm and the bottom of the next at each internode, increasing the strength of the plant as a whole. This means that a sample taken from the bottom of a culm section will have slightly different properties than a sample taken from the top of that culm, due to the change in fiber content. This will have to be taken into account during the fabrication of test samples.

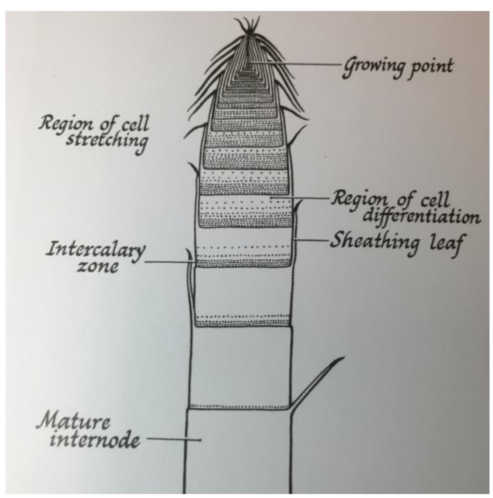


Figure 2. The principal parts of a typical bamboo stem, pertaining to how the stem emerges, elongates and develops. Notice how each internode is connected to the culm above it rather than the one below it, and attaches to the next culm like a sleeve.

Figure 3 shows a typical bamboo cell wall cross section, with cellulose and hemicellulose shown in blue and yellow respectively. The area surrounding the cellulose and hemicellulose fibers is filled by ligneous structures.

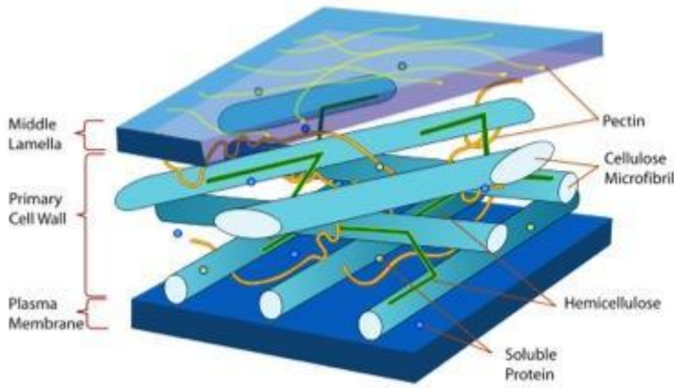


Figure 3. Microscopic structure of Bamboo cell wall showing cellulose and hemicellulose fibers in lignin matrix.

According to Shmulsky et al.[7] (2011) cellulose is the main structural component in the cell wall of bamboo, as it is in the cell wall of all plants that contain it. Cellulose is composed of the monomer glucosan, which is a 5-carbon sugar molecule with an oxygen molecule comprising the 6th spot on the ring, with alcohol and methanol groups on each ring in isotactic locations[5]. Each glucosan monomer is connected to the next through C-O-C linkages, creating an extremely strong structure. Cellulose forms long, highly crystalline chains of 100-10,000 units long. Strength increases with crystallinity, causing structures that contain higher molecular weight (longer) cellulose chains to be stronger than structures that have chains that are shorter due to chain degradation. Cellulose content is extremely high in fibers compared to the woody matrix surrounding them, which is the main reason for their increased strength. Hemicellulose has a structure similar to that of cellulose. However, hemicellulose (characterized as pentosan) is composed of five main C_5 and C_6 sugars, rather than just one. The main sugars are glucose, galactose, mannose, xylose, and arabinose. These form amorphous chains that are generally 50-200 units long. While hemicellulose is not as major a structural component as cellulose, its purpose in bamboo is largely as a crosslinker between different cellulose chains, and as a small-chain reinforcement fiber. The main purpose of lignin in woody materials is as the glue that holds everything together. It fills the remaining space in the cell wall between the cellulose and hemicellulose chains. It has a similar purpose to that of epoxy or other resins in composite applications, in that it transmits the load applied on the structure directly to the structural fibers. It is extremely strong in compression and bending, whereas cellulose provides strength in tension. It is mostly composed of aromatic monomer units including vanillyl, syringyl, and guicyl groups that form an extremely large amorphous random copolymer. The highly crosslinked polymer generally has a molecular weight in excess of 10,000+ molecular weight units.

Data from Higuchi[9] in Table I shows sample constituent percentages in two wood species, a typical softwood (Pine) and hardwood (Birch), as well as a common bamboo species.

Table I: Chemical analysis of 3 bamboo species.[9]

Microconstituent	Common Bamboo (*Phyllostachys heterocycla*)	Common Softwood (Pine) (*Pinus sylvestris*)	Common Hardwood (Birtch) (*Betula verrucosa*)
Cellulose	45%	40%	41%
Hemicellulose	26%	29%	33%
Lignin	25%	27%	22%

It can be seen from the table that overall the microconstituent percentages for woody tree species is similar to that of bamboo, with only slight differences. The cellulose content of bamboo is slightly higher due to the hollow culm and the need for more structural reinforcement. This can also be seen in the high degree of fiber density throughout the culm and the gradient transition through the culm. The hemicellulose content for bamboo is slightly less, because of the lack of woody matrix to make up for the increased fiber content of which hemicellulose is a main component.

2.II.ii Thermal Properties of Constituents - Degradation and Transformation

Bamboo is composed mainly of cellulose, hemicellulose, and lignin, which are the main microstructural elements of all woody materials. The range of each component is relatively wide compared to other plants, with cellulose ranging from 26-43% dry weight, hemicellulose ranging from 15-26% dry weight, and lignin accounting for 21-31% dry weight. The thermal degradation characteristics of these natural polymers are shown in Figure 4.[9]

From the image, it can be seen that lignin and hemicellulose degrade first and cellulose remains at a constant mass until about 300°C, after which it degrades all at once due to its regular repeating structure of the sugar glucose. Hemicellulose also degrades all at once but at a temperature lower than that of cellulose, due to it being comprised of a 5 main types of pentosan sugars that are less stable than the regular order of glucose. Lignin does not have a fast degradation, which is accounted for by its complex structure, which is comprised of many aromatic carbon groups that form a highly cross-linked polymer.

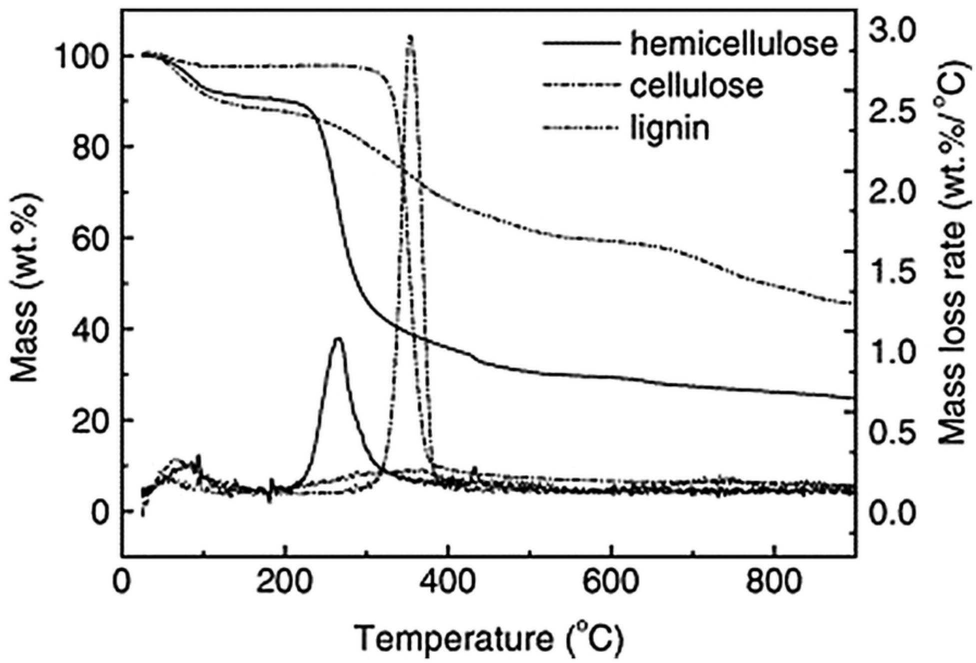


Figure 4. TGA curves of cellulose, hemicellulose, and lignin representing the mass loss and mass loss derivative.[9]

From Dickerson et al.[9] Figure 5 shows possible degradation pathways for Xylan, which is one of the more common molecular constituents of hemicellulose, and its various degradation pathways through pyrolysis.

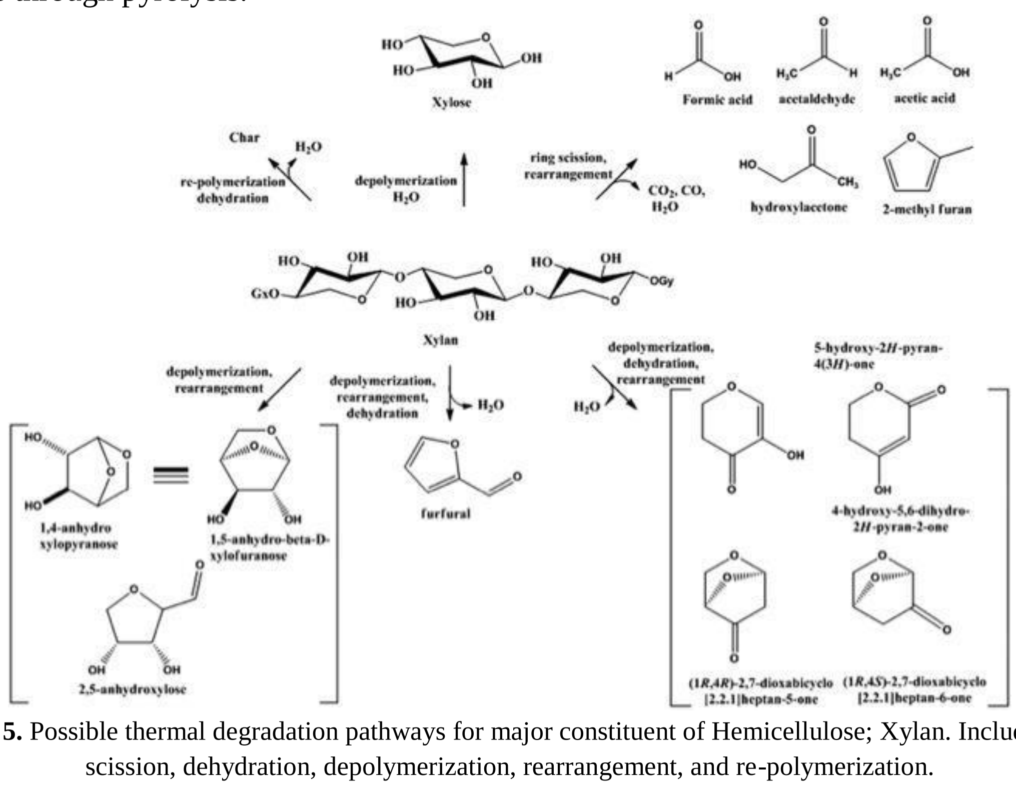


Figure 5. Possible thermal degradation pathways for major constituent of Hemicellulose; Xylan. Includes ring scission, dehydration, depolymerization, rearrangement, and re-polymerization.

According to Dickerson[10] (2013) reactions under pyrolysis conditions are complex and not fully understood due to the range of reaction temperatures and the complex biomass composition, but they can be generally classified as a simultaneous mix of dehydration, depolymerization, re-polymerization, fragmentation, rearrangement, crystallization, and condensation, as represented by some examples in Figure 5**.**

2.II.iii Heat Treatment Effects on Woody Structures

Bhuiyan et al.[12] (2000) determined that nearly twice as much crystallization occurred in wood cellulose under wet condition compared to oven-dried condition. They hypothesized that this was because lower stresses were acting on the wood components in the moist condition, so the cellulose molecules had a greater ability to rearrange. The study also observed that more crystallization occurred in wood cellulose compared to pure cellulose, hypothesizing that the other constituents play a role in aiding crystallization. A weak correlation that the ease of crystallization (based on activation energy) and decrystallization in moist condition is greater than in dry condition.

Boonstra et al.[13] (2007) observed a significant increase in modulus of elasticity in most softwood samples, tracing it to the crystallization of the polymeric structural wood constituents. It was also observed that heat treatment decreased tensile strength while increasing compressive strength.

Bhuiyan et al.[14] (2001) tested bending strength, compressive parallel, compressive radial, compressive tangential, tensile parallel, and hardness of multiple specimens. In Scots pine samples (a softwood tree), the heat treatments resulted in increased property values for compressive parallel strength and hardness (parallel and perpendicular). A slight increase in compressive tangential strength was observed. The other properties decreased, with a minimal decrease in bending strength. Observed significant decreases in impact strength. Greater variation in bending strength was seen in samples with a longer heat treatment dwell time, although average values were fairly consistent. This article supports the concept that the degradation of cellulose is limited below 210°C.

Adewopo et al.[9] (2011) tested the effects of heat treatment on various mechanical properties for Pine, Sweetgum, and Oak. The heat treatments were conducted at 93°C, 104°C, and 204°C, for 2 hours, 5 hours, and 8 hours resulting in a total of 27 data points for each mechanical test. The tests include Modulus of Rupture, Modulus of Elasticity, Compression Perpendicular to grain, Compression Parallel to grain, Shear parallel to grain, and Janka hardness for Pine. The results of these tests are shown below in Figure 6.

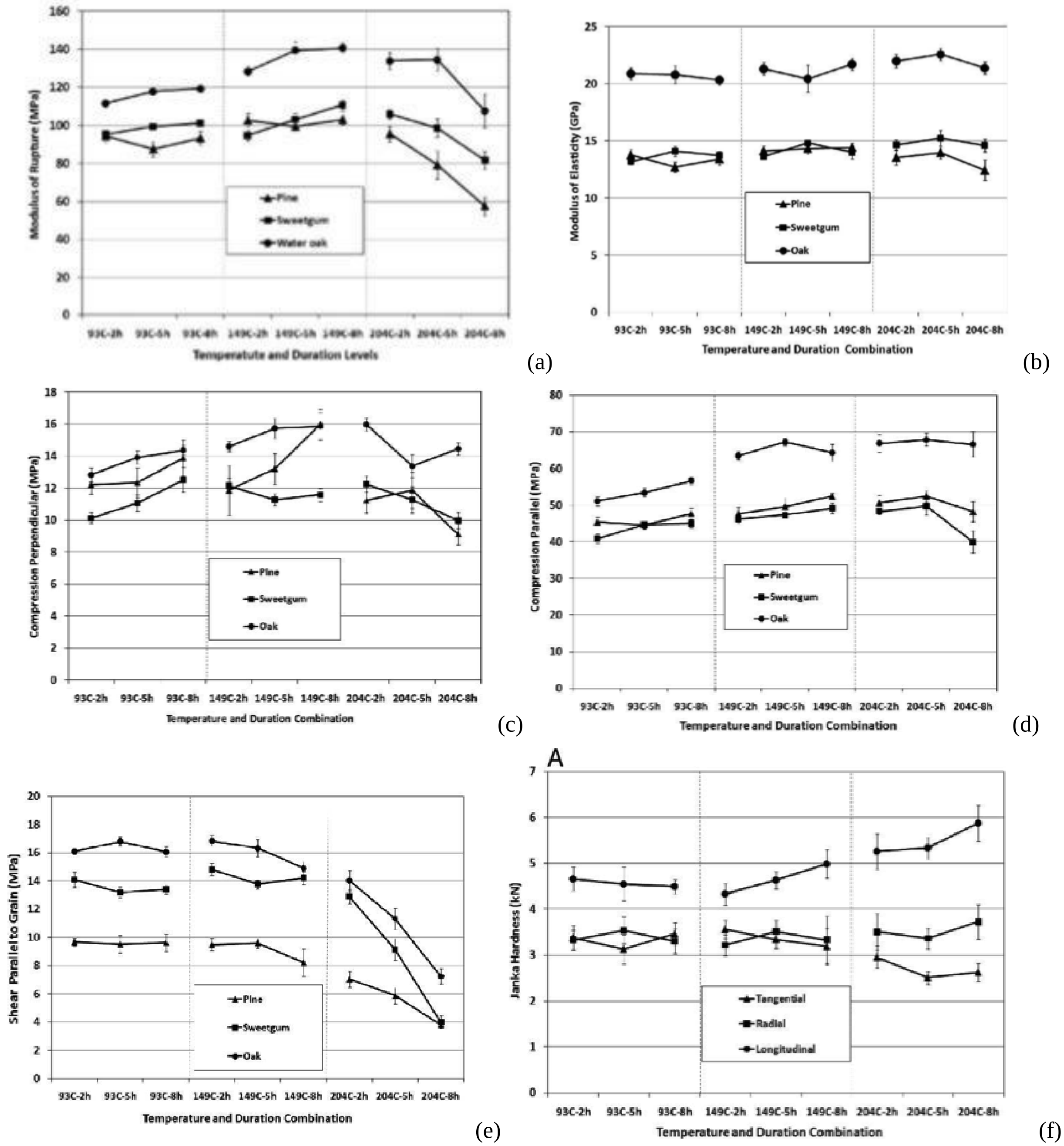


Figure 6. Changes in mechanical properties Pine, Sweetgum, and Oak with 3 heat treatments (93 C, 149 C, and 204 C) for 3 different lengths of time (2 hour, 5 hour, and 8 hour) (a) Modulus of Rupture (b) Modulus of Elasticity (c) Compression Perpendicular to grain (d) Compression Parallel to grain (e) Shear parallel to grain (d) Janka hardness for Pine.

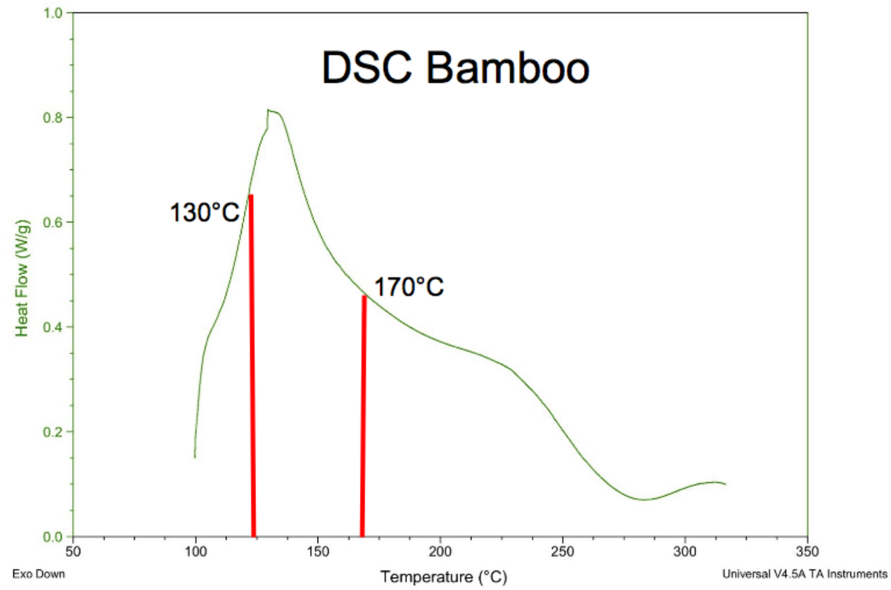
Source: Adewopo, Julius B., and David W. Patterson. "Effects of Heat Treatment on the Mechanical Properties of Loblolly Pine, Sweetgum, and Red Oak." *Forest products Society* (2011): 526-35. Print. [9]

From Figure 6 and according to Adewopo et al.[9] (2011) under dry conditions a heat treatment of 149°C for 8 hours can be safely applied to pine, sweetgum, and oak without posing a significant negative impact on their essential mechanical properties. It should also be noted that

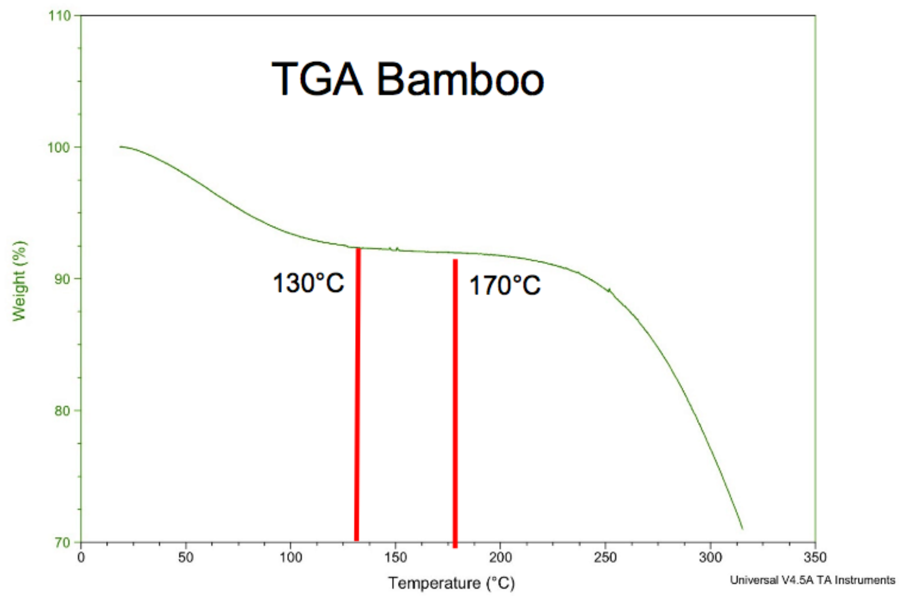
the high degree of variation seen in samples treated at 204°C indicates higher variability in the strength properties of wood treated at this temperature. This might be explained by the weakening of the internal bonds within the structure of the wood as a result of extensive degradation of the lignin matrix and the hemicellulose constituents. However, there were significant increases in MOR in heat treatments up to 149°C for 8 hours, significant increases in MOE, intermediate increases in compressive strengths, and insignificantly improved or sustained hardness values and shear strength up to 149°C for 8 hours. According to the literature, the crystallinity of cellulose, which is strongly correlated with the strength, is not changed or can even improve up to a certain temperature, which may be as high as 200°C depending on the conditions involved.[9] Also the short-term cross-linking and other modification of the lignin complex when wood is heat treated can essentially improve its strength properties, thus explaining the observed increases. For our testing, if similar improvements to the woody structures due to heat treatments could occur in our bamboo samples, heat treatment temperatures should be in the range of 100-200°C for a duration of 1-8 hours. Above 200°C and after 8 hours a large decrease in mechanical properties was seen, which is the reason for the limits in our testing.

Rajulu et al.[11] (2005) observed the thermal degradation of bamboo fibers from the *Dendrocalamus strictus* family, noting a significant amount of weight degradation beginning at 250-300°C. This suggests an extreme upper limit for heat treatment.

Thermal analysis was performed by Mackenzie Kirkpatrick during his research in CHEM 547 Polymers and Coatings Lab, on *Pseudosasa amabilis* or Tonkin cane bamboo (from Home Depot). The tests performed were differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) (Figure 7). DSC analysis involves measuring the heat required to increase a sample’s temperature, and it records heat flow as a function of temperature. This graph can be seen as an enthalpy change against temperature, with phase transitions requiring more energy and therefore having a measurable enthalpy value. TGA, alternatively, measures the mass loss with increased temperature due to decomposition, oxidation or loss of volatiles. The decrease in mass is measured in order to supply complementary and supplementary characterization information to the DSC. With Bamboo, if a temperature could be found where large amounts of energy is absorbed by the structure (DSC), but the structure is not degrading (TGA), this could be a temperature where a high degree of crosslinking is occurring between the lignin, hemicellulose, and cellulose polymers. Thermal holds at these temperatures would allow the structure to increase the connections between matrix and fiber, leading to an increase in strength and stiffness.



(a)



(b)

Figure 7. (a) DSC analysis curve of *Pseudosasa amabilis* or Tonkin cane bamboo (from Home Depot), (b) TGA analysis curve of *Pseudosasa amabilis*
Source: Mackenzie Kirkpatrick 2014

From Figure 7a, it can be seen that there is a transition peaks at about 140°C and 215°C with maximums heat flows of 0.85 W/g and 0.40 W/g respectively. The two distinct peaks show two phase transitions that occur in the bamboo structure. The second peak corresponds with the degradation curve (Figure 7b), where a rapid mass loss occurs at 235°C. The initial small mass loss of 7% is due to the decrease in moisture content in the bamboo sample. The rapid mass loss at 235°C corresponds with the theoretical degradation curve of hemicellulose (Figure 3). The sharpness of the second DSC peak is also similar to the degradation of hemicellulose which

occurs all at once, signifying the upper limit of temperature for our heat treatment tests due to the rapid loss of material. For our testing, the first peak signifies that there is a large amount of energy absorbed by the bamboo microconstituents from 100-200°C that does not directly correlate with a loss in material, meaning that the energy input is causing a change in structure. These changes could be similar to those described in Figure 5, i.e: depolymerization, bond formation, fragmentation, rearrangement, and crystallization. From this temperature range and from previous studies on woody materials, an ideal temperature range for our heat treatments will be from 130-170°C, as shown in Figure 7 above. As discussed, these temperatures are a thermally stable range for bamboo, while displaying a maximum heat flow into the material. Heat treatments done at these temperatures should display an increase in strength and stiffness compared to samples that have no heat treatment.

3. Experimental Procedure

3.I Safety Protocol

3.I.1 Mechanical

3.I.1.1 Machining

Make sure to wear eye protection (safety goggles with side of eye coverage), clothes that won't get caught in machines (long sleeves pulled back, jacket hoods down, jackets without strings, and closed toed shoes.

3.I.1.2 Hand Tools

When using chisel, mark out splitting locations with pencil to ensure equal sample size. If cutting culm in half, start split on both sides before cutting all the way through. Keep sample clamped in vice, or have partner hold remaining culm securely keeping hands at least 2 inches from cutting edge. Make sure your hands are clear from top of chisel, so they aren't hit by mallet.

When using saw, make sure your off hand is at least 2 inches away from cutting location, the cutting path is clear from clamp/vice or table support, and there is enough room to safely cut piece without hitting surroundings.

3.I.1.3 Planer

Ear protection is needed as well, either over-ear headphones or small in-ear plugs. Clear debris from planning surface to eliminate danger of kickback. Do not stand directly behind planer, in case kickback does occur. Make sure you have a push-stick that is smaller in thickness than the minimum thickness for the samples, in case a sample gets stuck or disengages from rollers. Before turning on the planer, set the height with the knob to the approximate height of the samples, then test by placing a sample into the planer to make sure it will engage with the rollers. Maximum cut depth should be ⅓ turn on the knob, or 1/32 inch. When planing multiple samples to the same final thickness, start with thickest, and plane until it equals the next thinnest and include that in that pass, then the next thinnest, etc, until

you are passing every sample through the planer at each height. (Don't plane down 1 sample to final thickness then start the next, work all at the same time/rate).

Use flat/smooth board instead of normal base surface of planer. This will ensure that small samples will not shift when moving through the planer and will keep a better surface.
Check samples between each pass. Beware inconsistencies (splintering) on surface that may occur during planing, coordinate planing direction to prevent further splitting or sample failure.

3.I.1.4 Sanding

If power sanders are utilized, handle the sample carefully, ensuring that your hands will consistently have enough clearance between them and the abrasive surface. If there is not sufficient space, use grips to add distance between hands and abrasive surface. Sand with smooth, even strokes that cover the whole sample, so no edges or irregularities occur.

When utilizing a sanding jig, prepare the jig to minimize sharp edges that your hands may accidentally intercept during use. Possibility of splinters in the case of wooden jigs.

3.I.1.5 Furnace

Prior to use, ensure no odd contaminants are present in the furnace. Remove anything that may have been left behind or may interact with the bamboo.

When interacting with furnace, use thermal gloves, face shield, and apron if available. Open furnace while standing to the side so the hot vapors do not damage skin/face/clothes. When inserting/removing samples have partner open/close door for you. Use tongs to place/remove samples on heating rack. Close furnace door if not inserting/removing to ensure least amount of heat loss as possible.

Do not leave the furnace unattended for extended periods of time. Ensure proper power down of the furnace when use is over.

3.I.1.6 Testing Properties - 3 Point Bend Test

Testing frame - 10K Instron Tensile Tester

Ensure tensile tester and components are free of debris for accurate measure and proper function. Do not touch any part of the fixture or sample during testing.
Use a testing shield in front of the fixture in case splintering or other dangerous failure modes occur.

Beware and stay clear of pinch areas:

1. Contact/moving points on machine, stay clear of dynamic motion areas.
2. 3 point bend fixture - Roller Edge (~¼" diameter)
3. Fixture / saddle contact point

If sample does not fail and bending causes sample to slide off of test frame, stop test immediately and return frame to starting location. If sample gets pushed off bottom supports, it may get thrown and injure machine or people.

3.I.2 Psychological

Stay clear headed and calm during the entire project process. Keep the big picture in mind, and be open minded about changing processes to improve final results. There WILL be issues that will sink time or ruin samples. Do not be discouraged or angered, but rather be optimistic and creative about what

methods you may take to bypass these obstacles. This is a standard in the real world, and becoming upset will not solve any issue.

Think logically through problems and discuss with other people if stuck at any point. Input from an outside voice can help solve problems that seem unsolvable.

In case of monetary worries, contact faculty advisors, Lisa, or department head.

This project may just save humanity, and thats a lot of pressure. Also, don’t let success go to your head. Captain, failure is not an option.

Do not hesitate from saying bad puns or jokes for fear of ridicule. Everyone needs a good laugh.

3.I.3 Chemical

If extreme dust occurs while planing or sanding, use a respirator so that no dust is inhaled.

H20 vapors removed during bamboo drying process. If the bamboo was treated, we could encounter other chemicals volatizing during drying process, but we are utilizing raw, outdoor dried bamboo. Dried ~1 year outdoors.

Possible inclusion of drying agent in air-tight container after first heat treatment to remove excess water content - silica gel? Promote a low-humidity environment. Ensure that any additions will not volatize in a harmful manner before use. If required, use of respirators and lab goggles will be utilized.

Possibility to require utilizing small amounts of epoxy to create hard surface on the samples for bend testing. When using 2-part epoxy, ensure curing is done in a well-ventilated area. Nitrile or latex gloves will be used to prevent epoxy-skin contact.

3.I.4 Ergonomic

When utilizing tools, use the correct tools for the designed function. Handle tools carefully, use clamps when necessary to secure jigs and samples for optimal precision. If doing repetitive work, make sure to be in a comfortable position where no cramping/discomfort will occur.

Clean areas before use - do not force a jig to sit where other items clutter the area.

3.II Raw Sample Preparation

Samples were cut from the thickest part of the culm to allow rectangular cross-section beams to be cut without curved surfaces. Select a bamboo culm to be divided in half. One half of the culm will be heat treated, the other half will not, to compare the differences from within a comparable sample. Carefully split the bamboo longitudinally using a mallet and chisel, incrementally cutting deeper into each side of the culm to avoid irregular splitting. Once divided, measure the required sample widths along the cross-section of the bamboo and split each half into respective sample beams.

The samples were hand-sanded on a flat surface, concave down, to yield a “flat enough” (no rock) surface to be passed through the planer. The samples were subsequently passed through the planer in the same concave down orientation to yield a flat side on the outer surface

of the bamboo. The bamboo samples were sent multiple times through the planer, maximum ⅓ turn (~3/64 inch), incrementally removing material until the thickness meets the specifications.

When the thickness requirement is met, the samples are sanded on their sides in a jig to yield precise rectangular cross-section samples. The samples are further cut to specified length, and a neighboring piece of the sample length is kept for further characterization and analysis.

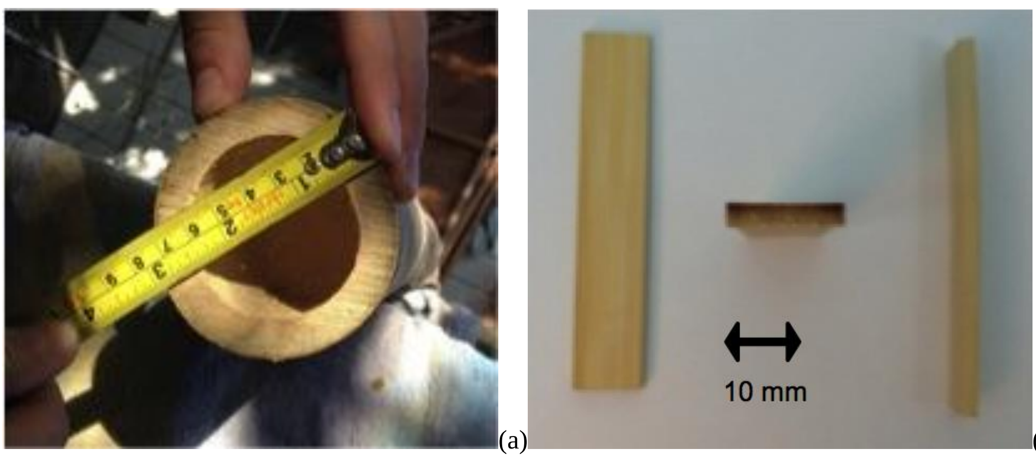
All samples were fabricated to be within the following dimensions.

Length = 84 mm

Width = 18 +/- 1.5 mm

Thickness = 3.5 +/- 0.7 mm

Figure 8 below displays the whole culm before sample fabrication, with culm location near the bottom of the stalk, as well as the final samples before heat treatment. A total of 250 samples were fabricated, and each culm length could produce about 10 bamboo samples, resulting in 25 culm lengths being utilized for sample fabrication. Of the 250 samples produced, a total of 207 samples were used in experimental tests and were chosen based on dimensions within experimental tolerance, sample uniformity, and lack of defects.



(a) (b)

Figure 8. (a) Bamboo culm before fabrication and (b) samples after fabrication.

3.III Mechanical Testing

To give consistency to data, the samples were fabricated with more length than the bend testing requires to accommodate the planer's dimensional requirements.

Modulus of Rupture and Stiffness - 3 Point Bend Test

ASTM D790-03 - Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

Each group of samples were tested on the Instron 150 kN load frame. Based on the specifications from ASTM D790-03 and the capacity of the load frame 3 point bending jig, a constant support span length of 70 mm was determined to be used in coordination with constant sample dimensions as stated previously. The sample lengths were fabricated to be 10% longer than the support span to eliminate the possibility for samples to be pulled off supports due to high flexural elongation. The support and indenter radius of ~5mm hopefully didn't cause early rupture due to indentation. A crosshead movement of 2 mm/min was utilized. The three-point bend test yielded data on the samples' strength in MPa and elongation in mm, from which the maximum flexural strength and relative flexural stiffness values were calculated. The flexural stiffness was calculated from the slope of the linear-elastic region at 1 mm of extension.

3.IV Pilot Testing

3.IV.1 Pilot 1

In Pilot Test 1, half of the samples were tested "fiber up" and half were tested "fiber down." This relates to the mode of failure in relation to the fiber density of the samples. "Fiber down" relates to the fiber-dense side of the sample failing in tension, while "fiber up" relates to the fiber-dense side of the sample failing in compression. Figure 9 displays a schematic for a sample tested in the "fiber down" position.

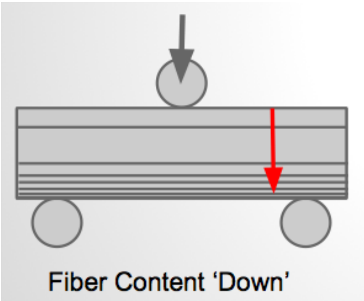


Figure 9. Sample in the 3-point bend fixture in a fiber down orientation.

From this we expected to see which orientation of the sample would display a greater strength and lower variance for 3 point bend testing. In addition to this, we desired to test whether there was a significant variance in strengths between different culms along the length of the bamboo. Three culm sections were tested to see if strength changes based on location up or down the culm stalk. We tested this in conjunction with the fiber up and fiber down test, using 10 samples from 3 different culm pieces. Of which, 5 were tested fiber up and 5 were tested fiber down, as seen in Table II below. A total of 30 samples will be tested in Pilot test 1.

Table II: Schematic representation of sample locations and testing orientation for Pilot Test 1.

Length of time	Bottom Culm	Middle Culm	Top Culm
Fiber Up	5 Samples	5 Samples	5 Samples
Fiber Down	5 Samples	5 Samples	5 Samples

3.IV.2 Pilot 2

Several batches of samples were prepared that had different dimensions and were subjected to different heat treatments. Each batch contained 6 bamboo samples. The different dimensions include the maximum thickness and width, the minimum thickness and width, and a thickness/width combination in the middle range. Table III shows the heat treatments schematically, with a total number of 46 samples. The bamboo samples were divided so that 1 culm piece had a section in each heat treatment block, due to the first round of pilot testing which determined that there is not significant variance between culm positions, but all source of variance were attempted to be minimized. This helped eliminate the relative variation in strength between culms by ensuring that each heat treatment block is an average of all of the culms used in the experiment.

Table III: Schematic representation of heat treatment temperatures and times for Pilot Test 2.

Length of time	No Heat Treatment	103 +/- 2 °C	160 +/- 2 °C	103 +/- 2 °C and 160 +/- 2 °C
0 hour	10 Samples			
1 hour		6 Samples	6 Samples	6 Samples
4 hour		6 Samples	6 Samples	6 Samples

3.V Moisture Control Heat Treatment (Normalization)

The small furnace in the MATE furnace laboratory displayed good accuracy for heating the samples. They could reach the high 100 °C range required for this experiment. A thermocouple was placed internally near the samples to ensure consistent temperatures throughout the furnace.

In our pilot testing the addition of the normalization had no significant difference between the heat treated samples and the normalized. However, the normalized samples had lower variance compared to the heat treated samples. For this reason all of the heat treated samples were given the normalization heat treatment.

All of the samples were placed in the oven at 103 +/- 2 °C, and heated for 1 hour. The samples were removed from the furnace and kept in an airtight sealed container to eliminate moisture from re-entering the samples. The samples were then heat treated according to the procedure in the section that follows.

3.VI Formal Heat Treatment

For each heat treatment and time, 8 samples were tested in order to have statistically significant data, due to the inconsistent nature that is inherent with testing natural materials. For these reasons, 11 samples were tested for the non-heat treatment baseline, in order to have consistent data to compare the results of the heat treatment to. In our pilot testing the addition of the normalization had no significant difference on the average recorded strength between the heat treated samples and the normalized samples, but the normalized samples displayed lower variance. It was determined that all samples would be normalized at 103 +/- 2 C prior to heat treatment in order to eliminate the variable of different moisture contents. All fabricated samples were randomly scattered across heat treatment group to decrease variance related to culm position. Table IV shows the heat treatments schematically with a total of 131 samples.

Table IV: Schematic representation of heat treatment temperatures and times for formal testing

	All heat treated samples normalized at 103 +/- 2 °C for 1 hour prior to HT					
No Heat Treatment		130 +/- 2 °C	140 +/- 2 °C	150 +/- 2 °C	160 +/- 2 °C	170 +/- 2 °C
11 Samples	**Time**					
	0.5 hour	8 samples	8 samples	8 samples	8 samples	8 samples
	1.5 hour	8 samples	8 samples	8 samples	8 samples	8 samples
	3.0 hour	8 samples	8 samples	8 samples	8 samples	8 samples

4. Results and Discussion

4.I Pilot 1

The summary of the results and analysis from Pilot Test 1 are displayed in Figures 10 and 11 in the form of boxplots. The raw stress versus strain plots for all samples are displayed in the Appendix. Pilot 1 tested the effect of sample locations from different culm positions along the bamboo stalk as well as different fiber orientation.

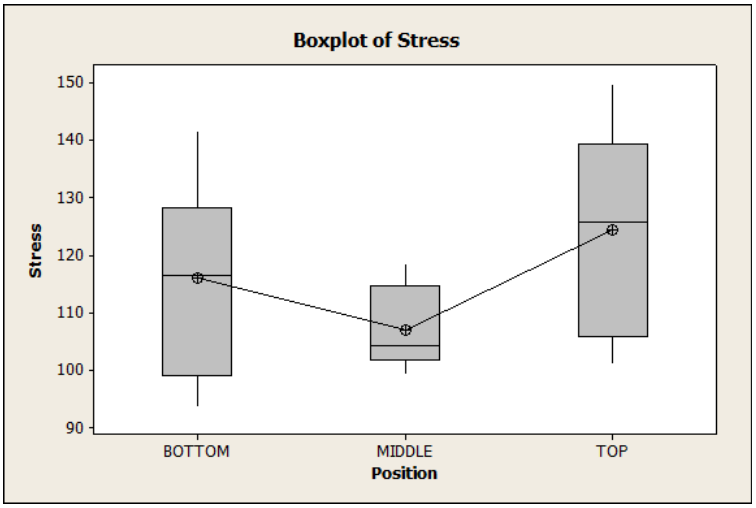


Figure 10. The boxplot of stress vs culm position from Pilot Test 1.

There was no statistically significant trend determined by comparing the stresses of the samples based on culm position. There was a statistical scheme of A, AB, and B, signifying overlapping groupings and no distinct difference. To be sure that this did not play a large effect on recorded values, it was determined that samples should be randomized throughout heat treatment groupings by culm position in order to decrease variance.

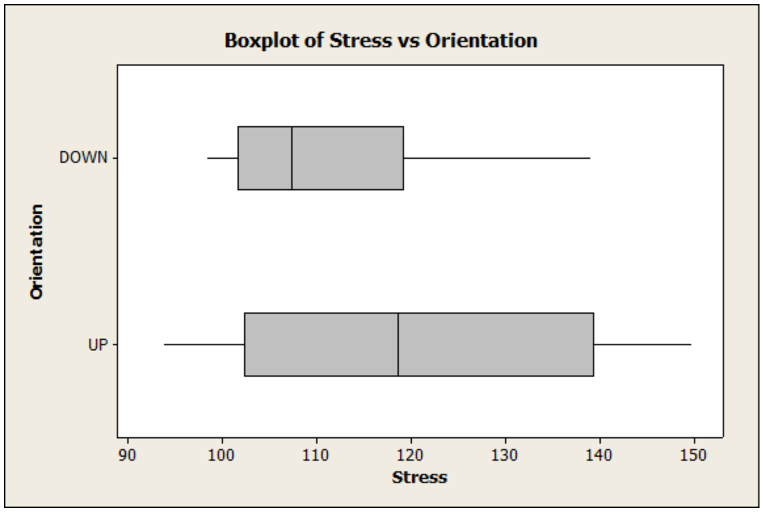


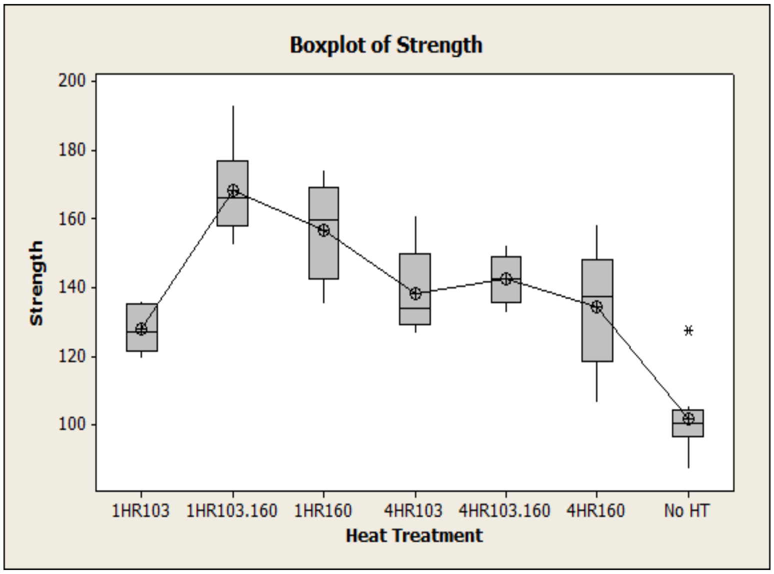
Figure 11. The boxplot of stress vs fiber orientation from Pilot Test 1.

There was a statistically significant difference determined by comparing the stresses of the samples based on the orientation of the fiber dense side during three-point bend testing. It was determined that the fiber-down orientation samples had less variance, so this was the method used for the remainder of testing in this experiment. This was likely due to more fibers being under tension during high stresses, causing a more consistent failure strength for all the samples.

Pilot Test 1 displayed some significant experimental effects. First, it was shown that the culm position did not have a significant effect on flexural strength. Second, it was determined that the fiber content down had the least variance. Observing this, we could continue testing by randomizing the culm position throughout all heat treatments and testing all samples with the fiber content heavy side down.

4.II Pilot 2

The summarized results from Pilot Test 2 are displayed in Figure 12. Pilot 2 was a proof of concept test to ensure that heat treatment did have an effect on the flexural strength and stiffness of bamboo. The raw stress-strain plots for all samples are displayed in the Appendix.



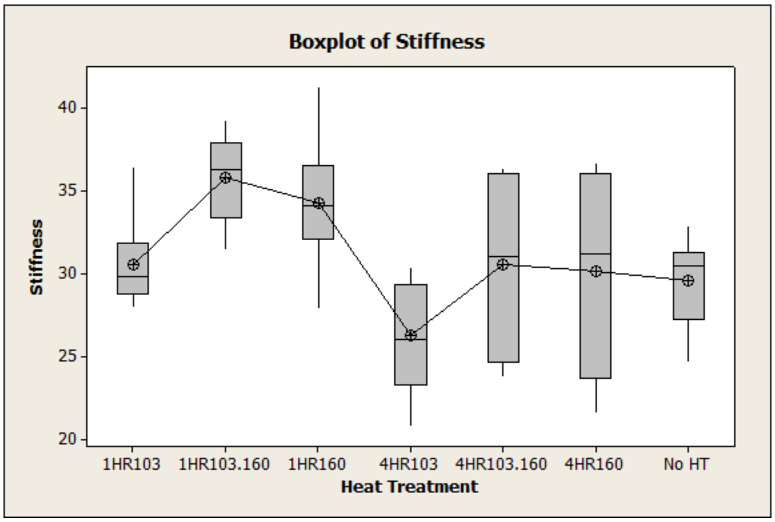


Figure 12. Correlation of stress and stiffness with heat treatment groupings.

This portion of the test displayed the differences between one and two step heat treatments, as well as short and long times of 1 hour and 4 hours and low and high temperatures of 103°C and 160°C. The maximum average strength was displayed by the sample grouping heat treated at 1 hour 103°C followed by 160°C. Comparing this to the non-heat treated samples, this is the equivalent to an average 70% strength increase. These samples displayed an average 20% increase in stiffness. This displays that heat treatments have the ability to increase the strength and stiffness of bamboo samples.

Comparing the 4 hour 103°C & 160°C HT to the 4 hour 160°C HT, we observe a significant decrease in variance with the sample that was treated with the 103°C treatment prior to elevated temperature treatment. A similar effect was seen in the 1 hour samples. From these results, it can be determined that the 103°C normalization had a significant effect by decreasing variance in the samples.

Pilot Test 2 displayed a few significant experimental effects. First, it was shown that there strength and stiffness can be improved by heat treatment. Second, it was determined that normalizing the samples at 103°C prior to high temperature HT reduced variance among the samples. Third, it was observed that too much heat could decrease the mechanical properties displayed by the samples performing below their maximum values. Observing this, we confirm bamboo can be over-aged by too much heat input into the microconstituent decomposition regime as seen by previous TGA plots.

4.III Formal Testing

Two raw stress-strain plots from the formal test displaying the most significant increase in strength are displayed in Figures 13 and 14. Figure 13 displays the curves from the non-HT sample group and Figure 14 displays the raw stress-strain plots from the sample group that was normalized and heat treated at 140°C. The raw stress-strain plots for all samples are displayed in the Appendix.

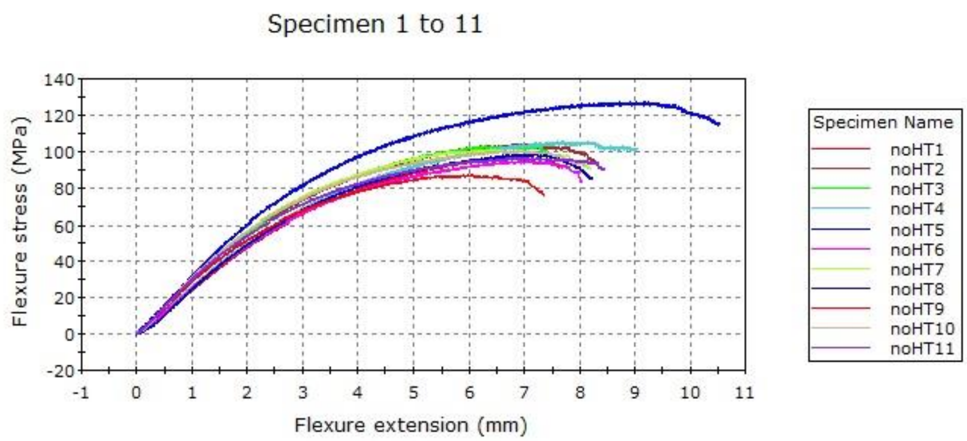


Figure 13. Raw stress-strain plot of 11 bamboo samples with no heat treatment (control samples).

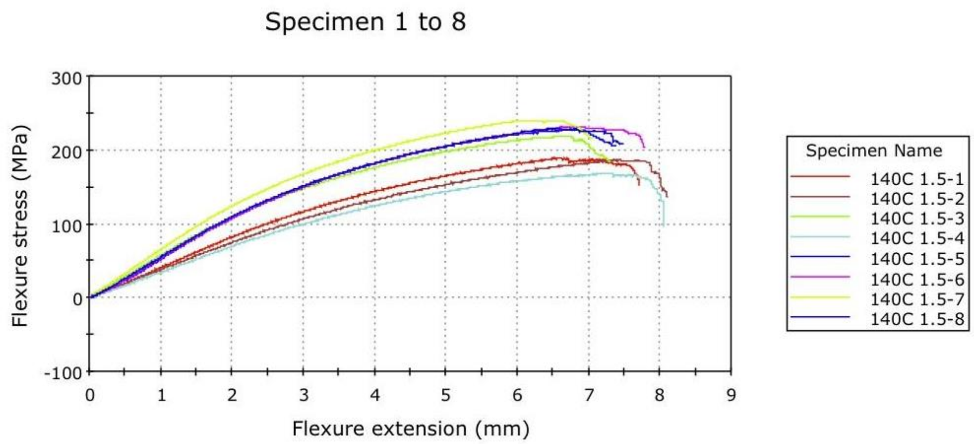


Figure 14. Raw stress-strain plot of 8 samples given a 1 hour 103°C normalization followed by 1.5 hour at 140°C heat treatment.

Comparing these two test groups, there is clearly a significant effect of heat treatment on the mechanical properties of bamboo, through the increase in maximum strength for the heat treated samples. These samples displayed an average strength of 200 MPa compared to the maximum values of the non heat treated samples with an average strength of 100 MPa. The flexure extension did not decrease dramatically for the heat treated samples, so it can be said that the toughness (area under the curve) increased with the heat treatment as well. The results from the full formal tests are displayed as boxplots in Figures 15 and 16.

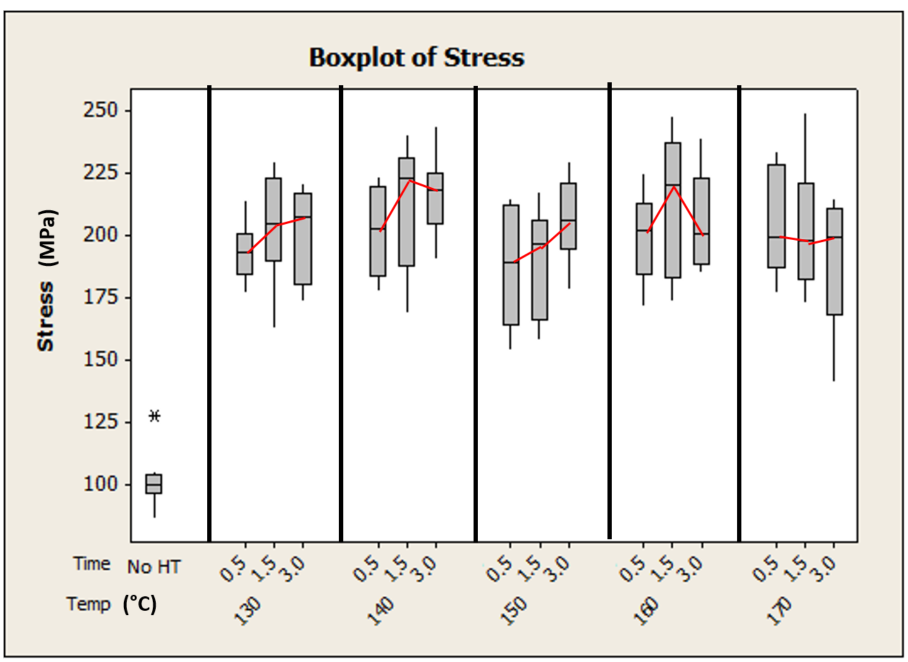


Figure 15. Stress values and means of heat treated samples as a function of heat treatment time and temperature.

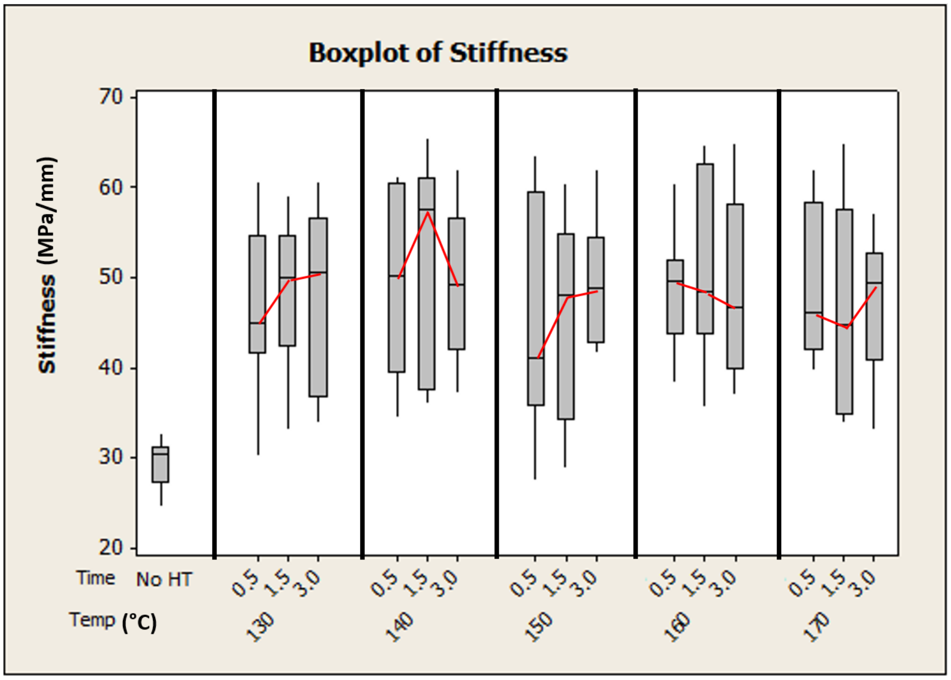


Figure 16. Stiffness values and means of heat treated samples as a function of heat treatment time and temperature.

The boxplots of stress and stiffness display a significant difference in grouping between the non-heat treated samples compared to the heat treated samples. In both the stress and stiffness groupings, a larger variance in sample properties was displayed for all heat treatments than for the non-HT samples, although nearly every value of the HT samples was higher by multiple standard deviations. The maximum strength displayed by any of the samples was a stress of 248 MPa by a sample from the 160°C/1.5 hour heat treatment group. The maximum stiffness displayed by any of the samples was a relative stiffness of 65 MPa displayed by a sample from the 140°C/1.5 hour heat treatment group. The average values displayed by the non-HT samples were flexural stresses of 100 MPa and flexural stiffnesses of 31 MPa. The average values displayed by the entire group of heat treated samples was a flexural stress of 200 MPa and a flexural stiffness of 48 MPa. This relates to an average 100% increase in strength and a 50% increase in stiffness.

Unfortunately, there was no statistical distinction between the different heat treatment groupings. But the different groupings did display observable trends. Similar to Pilot Test 2, it was observed that high time and temperature combinations led to decreased strength. This was due to embrittlement of the samples that led to an increased number of failure modes, such as longitudinal fracture along the fiber lengths. The occurrence of visible warping in these samples increased significantly as well, which implies that these high temperature heat treatment would not likely be suitable for most applications.

5. Conclusion

Bamboo is a woody grass that is one of the fastest growing plant species on earth. It is used worldwide because of it's highly renewable nature and good mechanical strengths and properties. Bamboo grows in hollow culm units separated by nodes that form cross-sectional rings throughout its length. Its microstructural components are primarily composed of cellulose, hemicellulose, and lignin. Bamboo's strength is derived from fibers composed of highly crystalline cellulose polymer chains, which are held together by shorter amorphous hemicellulose chains and bulk lignin polymers. The fiber content in a bamboo culm increases from inside to outside and from bottom to top, giving it unique properties not seen in other woody plants. Thermal analysis of other woody plants shows a correlation between strength and stiffness properties and the thermal heat treatment of the different microstructural constituents, primarily lignin and hemicellulose. A thermal analysis through differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) showed two absorption peaks that occurred at 140°C and 225°C (from DSC) before a rapid loss in mass at 235°C (from TGA). This corresponds with the rapid theoretical degradation of hemicellulose from 225-250°C.

Our results indicated a significant increase in flexural strength and stiffness of bamboo samples that were heat treated between 130-170°C. During testing, it was determined that a normalization heat treatment of the samples at 103°C prior to elevated heat treatment procedures reduced variance in the mechanical properties displayed. Although the range of heat treatment times and temperatures did not show any statistically significant difference, the samples displayed an average 100% strength increase and 50% stiffness increase compared to the control non-heat treated samples. Maximum values of strength and stiffness were seen in the 160°C 1.5 hour and 140°C 1.5 hour heat treatments, with a maximum flexural strength of 248 MPa and maximum flexural stiffness of 65 MPa.

The theorized mechanism of increasing strength is based on analyses from a combination of the DSC, TGA, and mechanical testing. It is seen there is a small mass loss of in the TGA curve of bamboo at 100°C which is mainly due to a reduction in moisture content (Figure 7). This is seen as well in the degradation curves of the microconstituents (Figure 4) with the largest mass loss observed in the hemicellulose and lignin curves, signifying their higher absorbed water content and a higher connectivity with water through hydrogen bonding. With heat treatment these bonds with water are broken, which increases the free bonding sites and enables a higher degree of bonding between microconstituents, increasing the connectivity in the structure as a whole. With increased connectivity the number of bonds required to break for the structure to fail increases, leading to a higher overall strength and stiffness in the bamboo. From the DSC curve (Figure 7), there is a maximum heat flow into the bamboo structure at 140°C, meaning the number of bonds being broken and reformed is a maximum at this temperature, which could be the reason for the maximum values in strength and stiffness being seen in the 140°C 1.5 hour heat treatments.

6. References

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Appendices

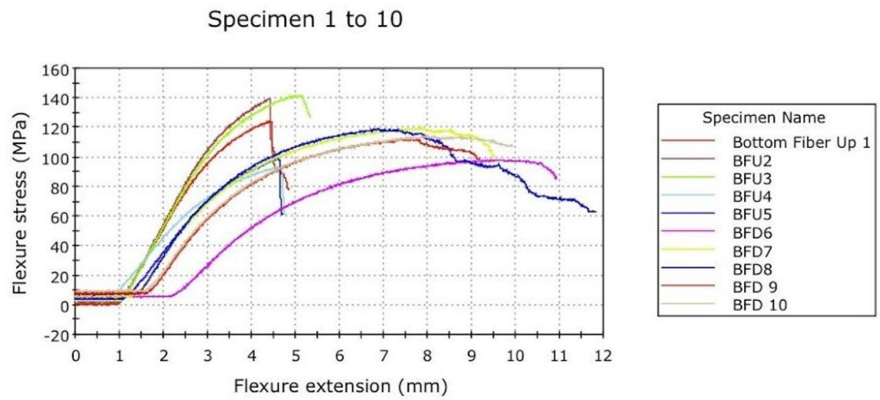

Figure 17. Pilot 1: Raw stress-strain plot of 10 bamboo samples at the bottom culm location, with 5 samples tested in the fiber up and 5 samples tested in the fiber down positions.

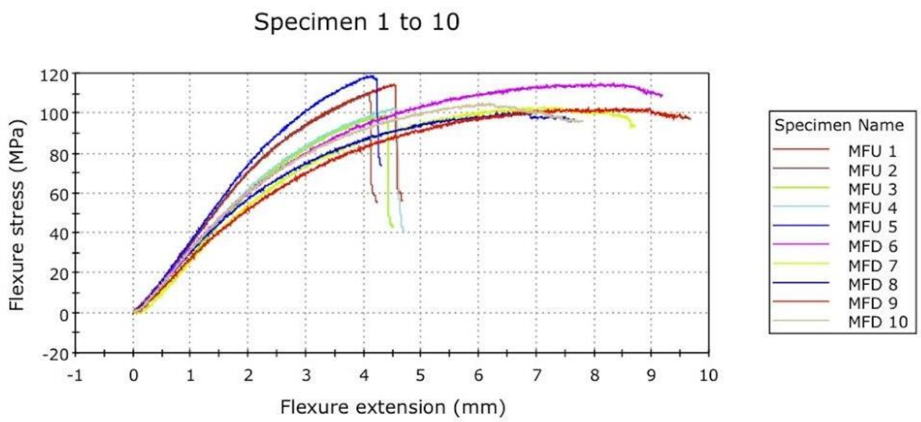

Figure 18. Pilot 1: Raw stress-strain plot of 10 bamboo samples at the middle culm location, with 5 samples tested in the fiber up and 5 samples tested in the fiber down positions.

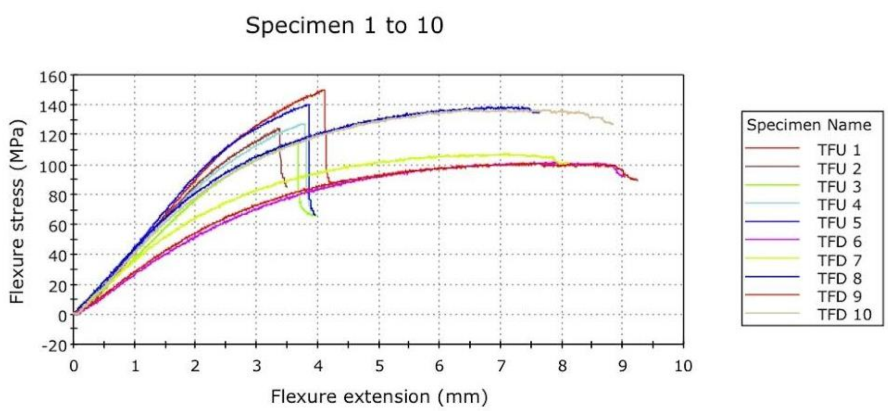

Figure 19. Pilot 1: Raw stress-strain plot of 10 bamboo samples at the top culm location, with 5 samples tested in the fiber up and 5 samples tested in the fiber down positions.

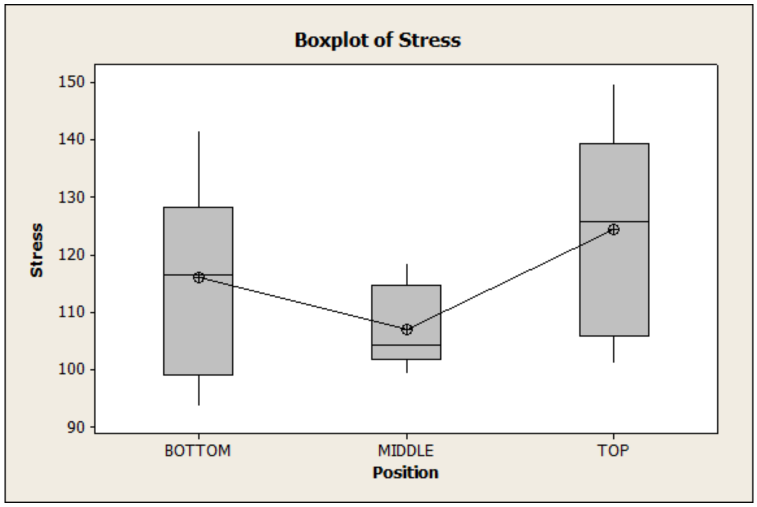

Figure 10. The boxplot of stress vs culm position from Pilot Test 1.

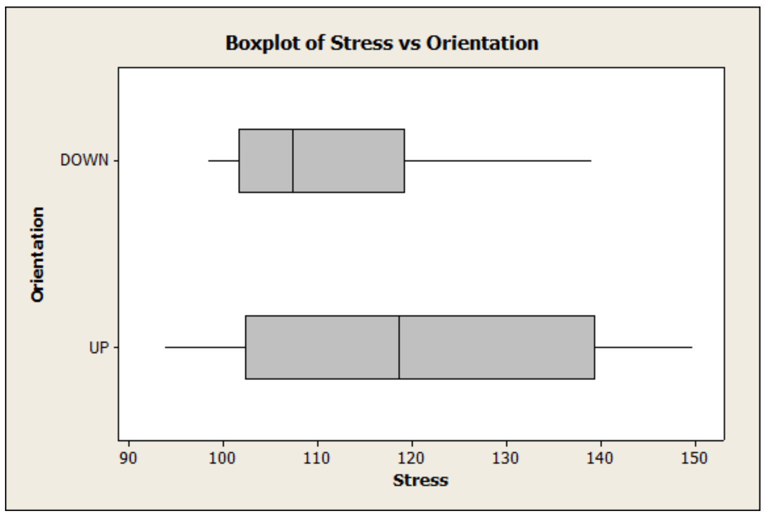

Figure 11. The boxplot of stress vs fiber orientation from Pilot Test 1.

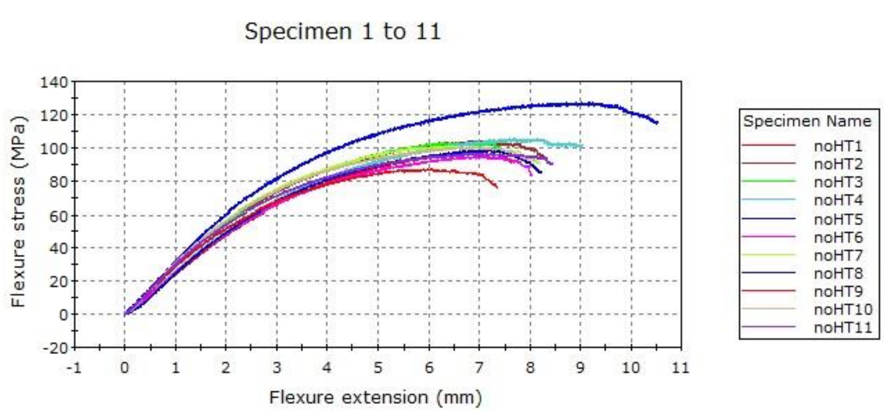

Figure 13. Raw stress-strain plot of 11 bamboo samples with no heat treatment (baseline samples).

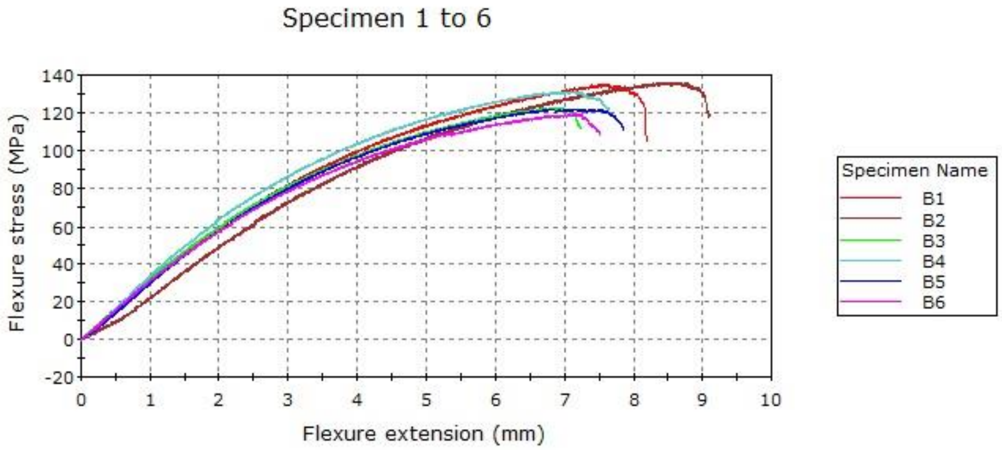

Figure 20. Raw stress-strain plot of 6 bamboo samples given a 1 hour 103°C normalization heat treatment.

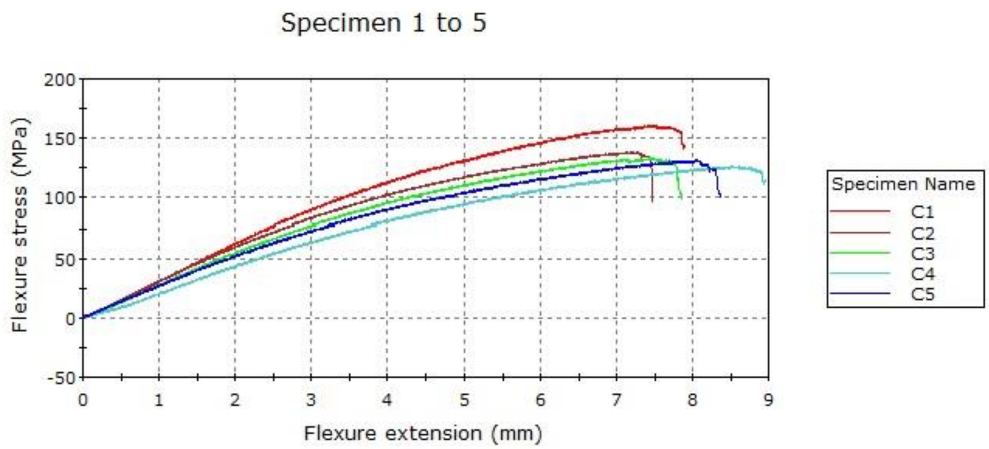

Figure 21. Raw stress-strain plot of 5 bamboo samples given a 1 hour 160°C heat treatment.

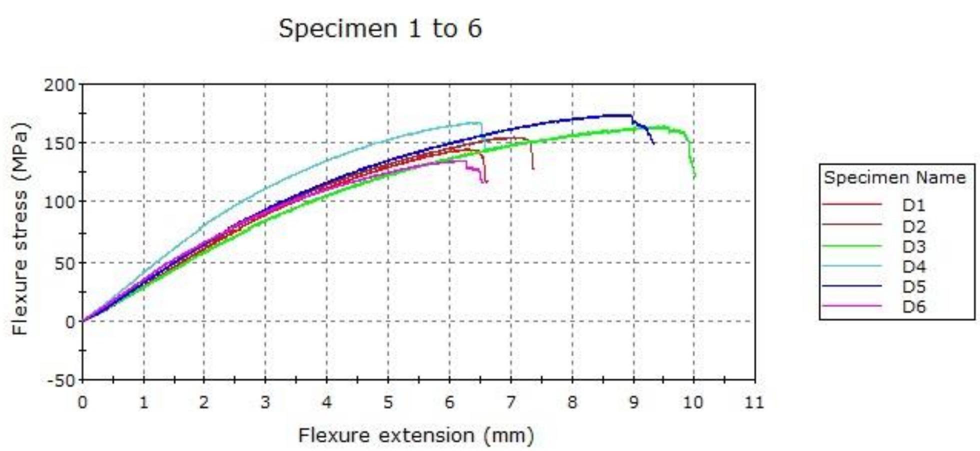

Figure 22. Raw stress-strain plot of 6 bamboo samples given a 1 hour 103°C normalization followed by 1 hour at 160°C heat treatment.

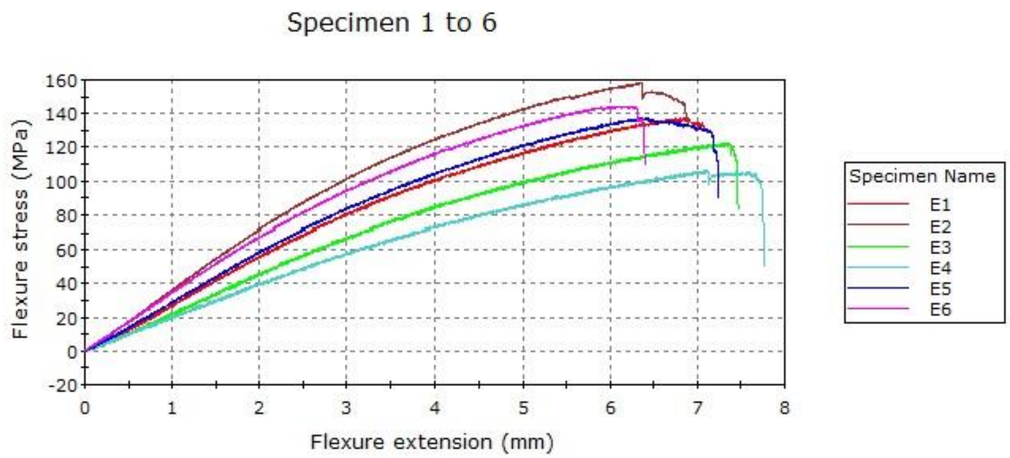

Figure 23. Raw stress-strain plot of 6 bamboo samples given a 4 hour 103°C normalization heat treatment.

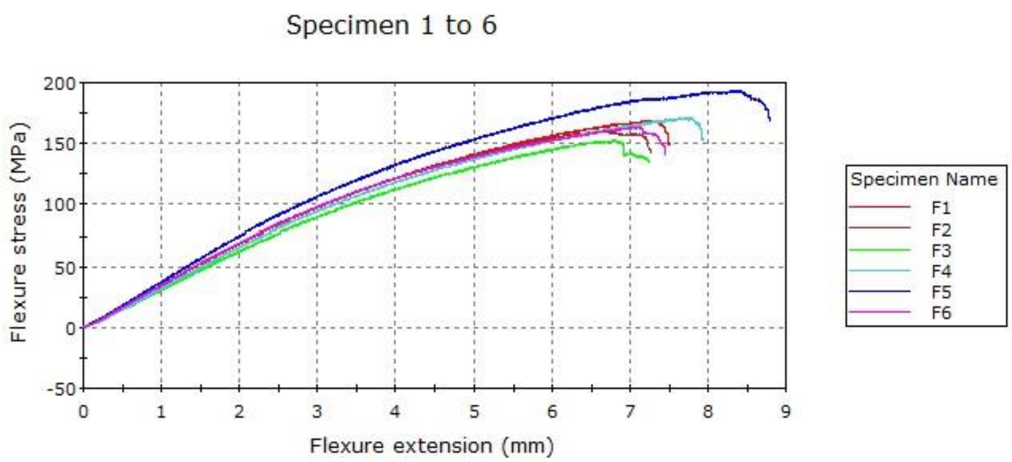

Figure 24. Raw stress-strain plot of 6 bamboo samples given a 4 hour 160°C heat treatment.

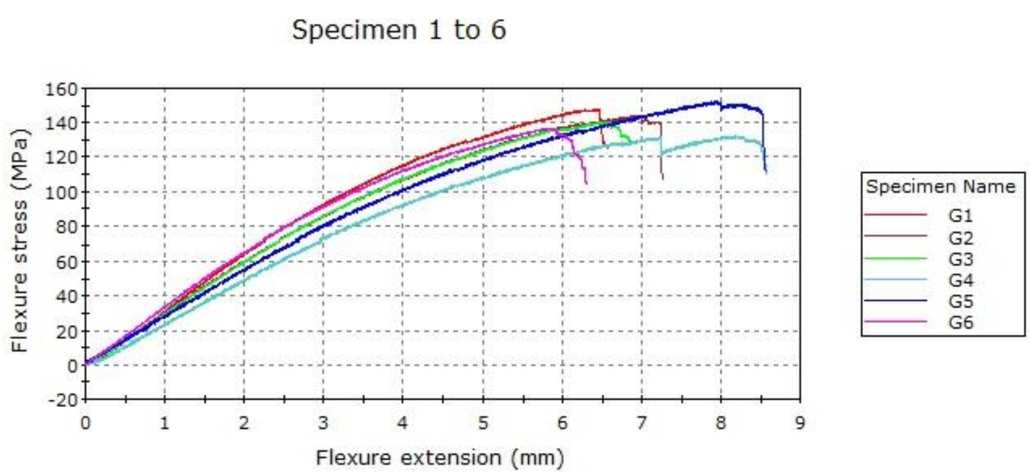

Figure 25. Raw stress-strain plot of 6 bamboo samples given a 4 hour 103°C normalization followed by a 4 hour 160°C heat treatment.

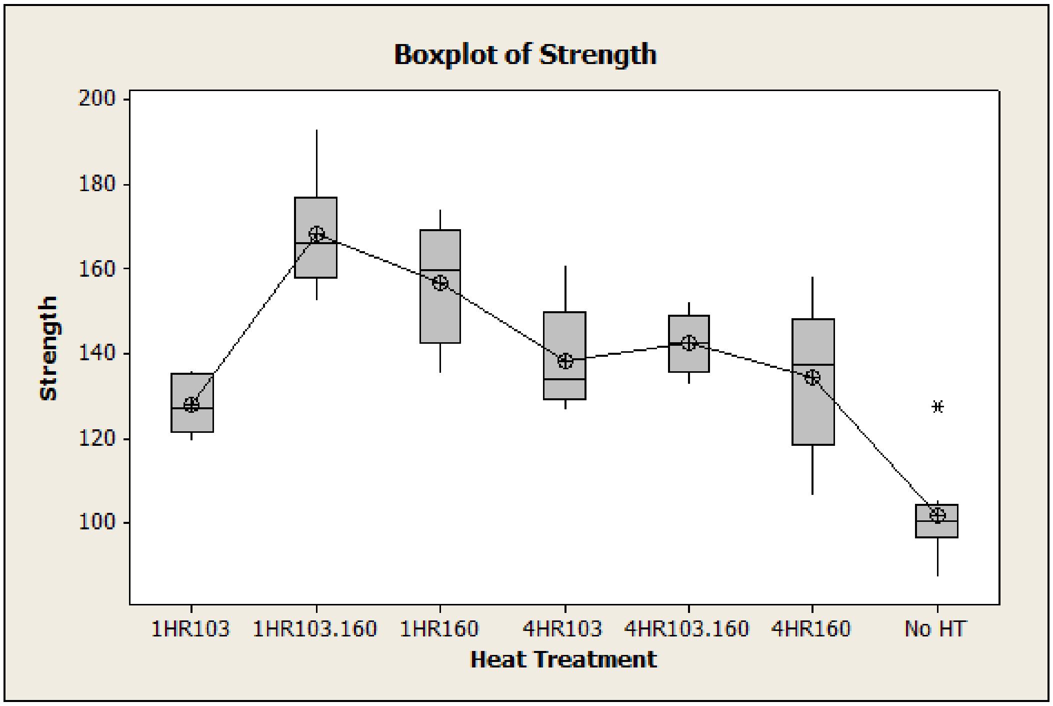

Figure 12. Pilot 2: Stress values and means of heat treated samples in Pilot 2 as a function of heat treatment time and temperature.

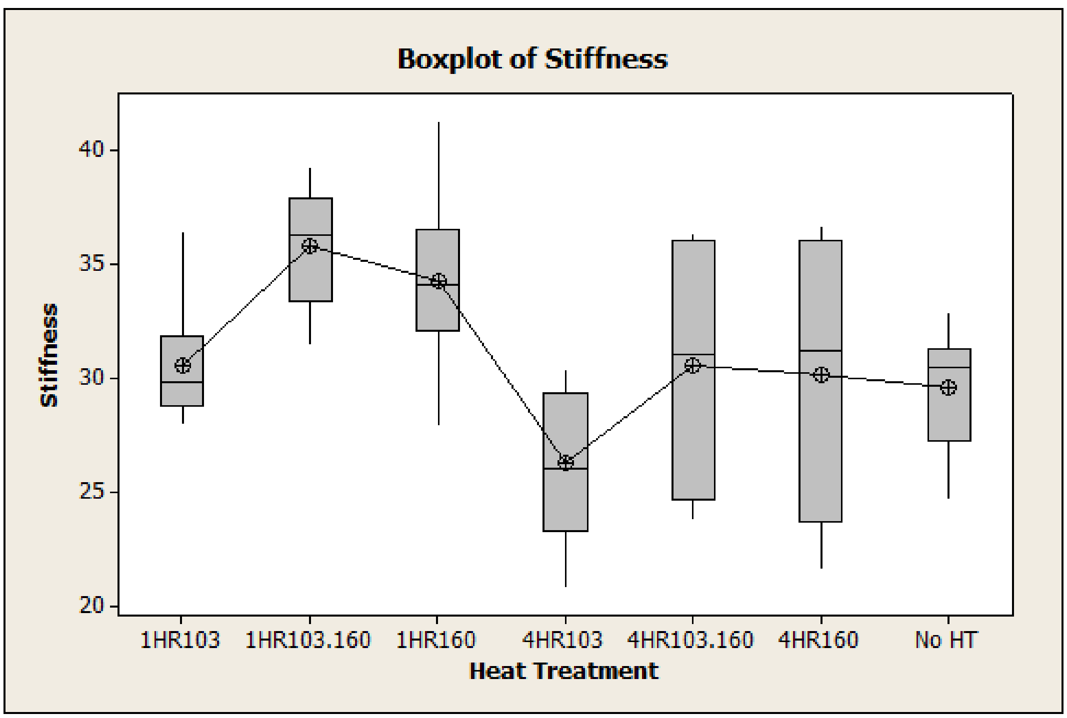

Figure 12. Pilot 2: Stiffness values and means of heat treated samples in Pilot 2 as a function of heat treatment time and temperature.

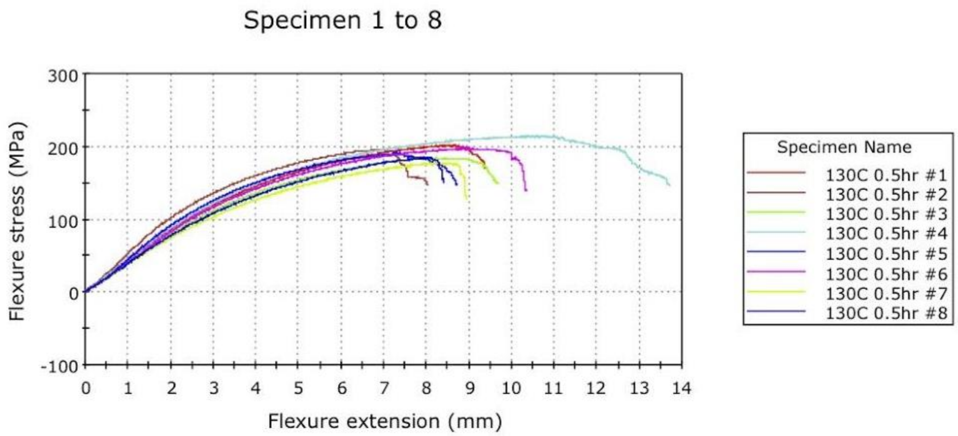

Figure 26. Raw stress-strain plot of 8 samples given a 1 hour 103°C normalization followed by 0.5 hour at 130°C heat treatment.

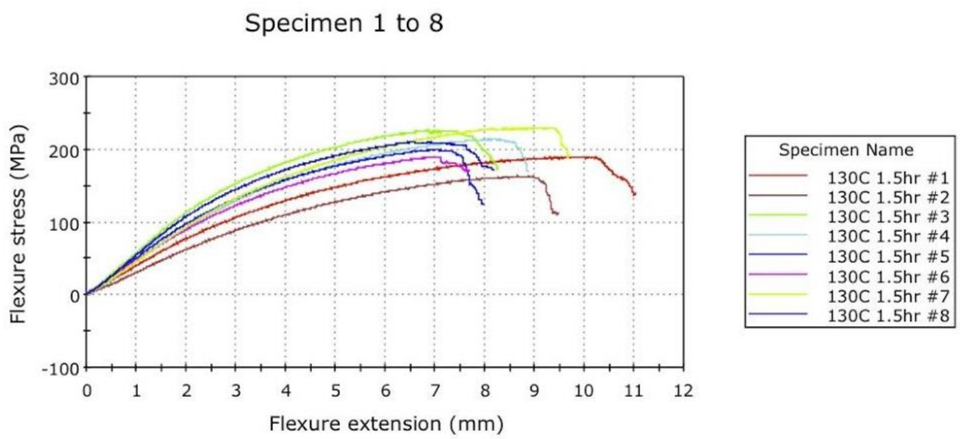

Figure 27. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 1.5 hour at 130°C heat treatment.

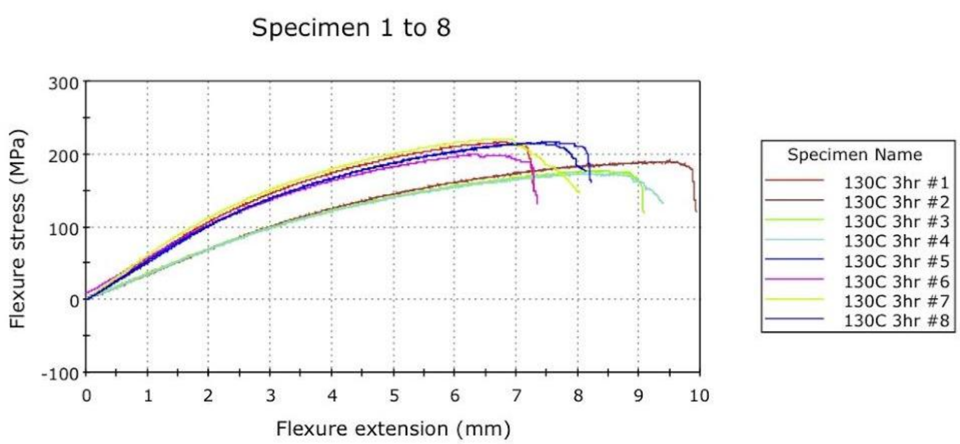

Figure 28. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 3.0 hour at 130°C heat treatment.

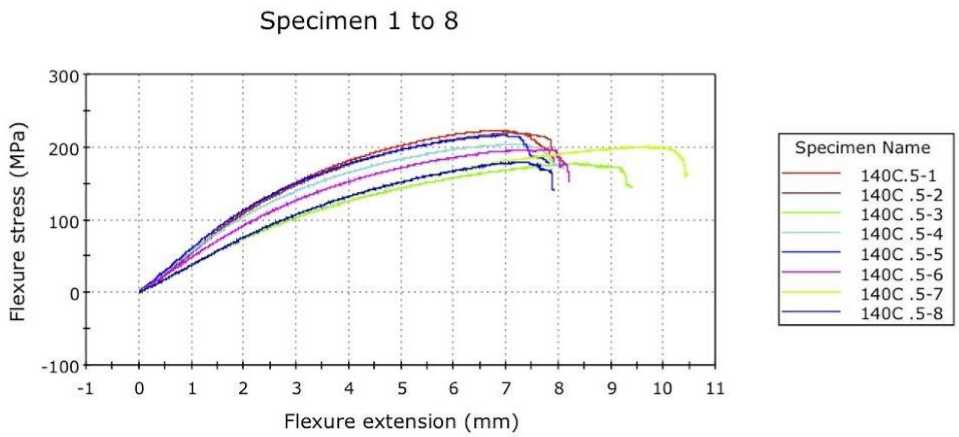

Figure 29. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 0.5 hour at 140°C heat treatment.

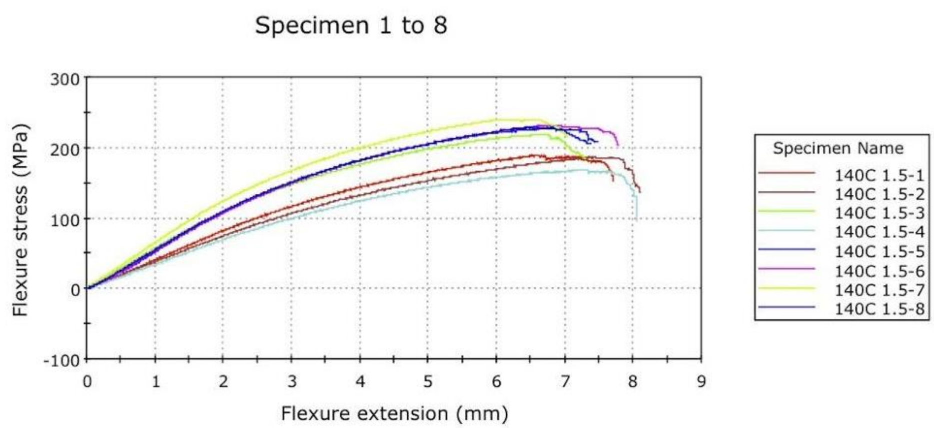

Figure 30. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 1.5 hour at 140°C heat treatment.

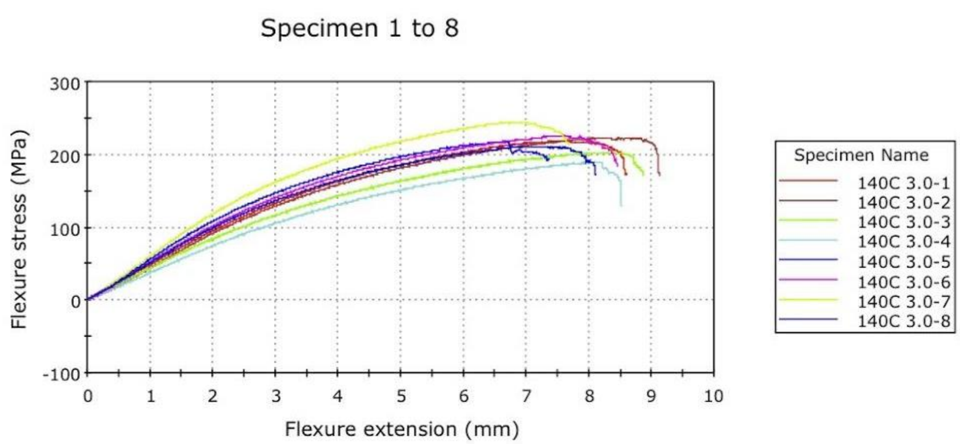

Figure 31. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 3.0 hour at 140°C heat treatment.

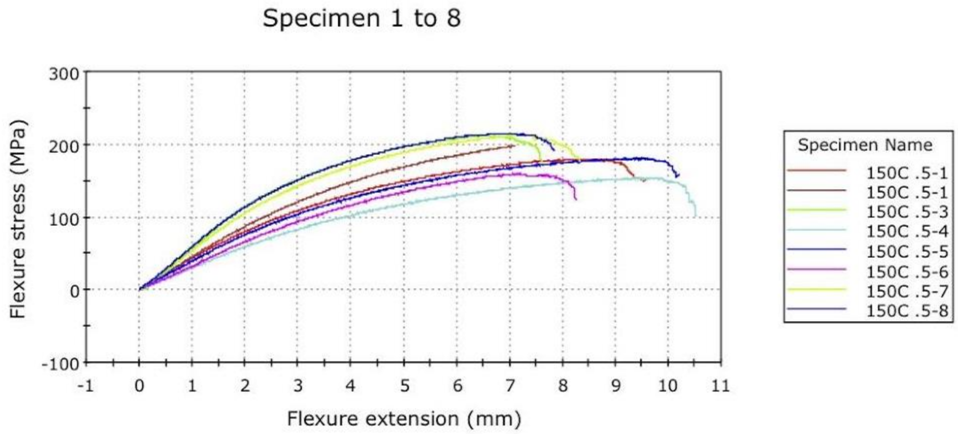

Figure 32. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 0.5 hour at 150°C heat treatment.

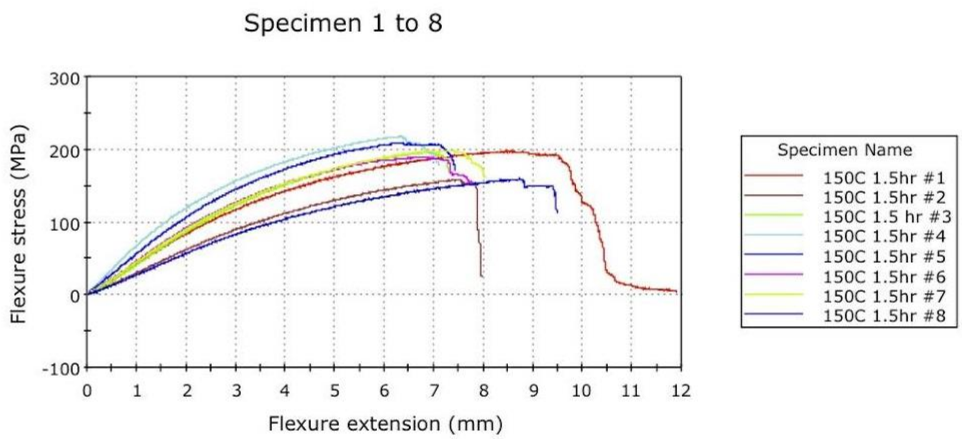

Figure 33. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 1.5 hour at 150°C heat treatment.

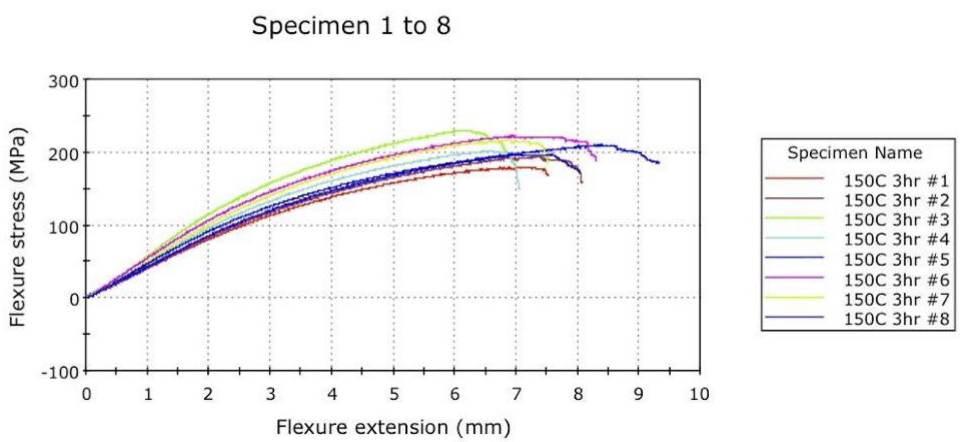

Figure 34. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 3.0 hour at 150°C heat treatment.

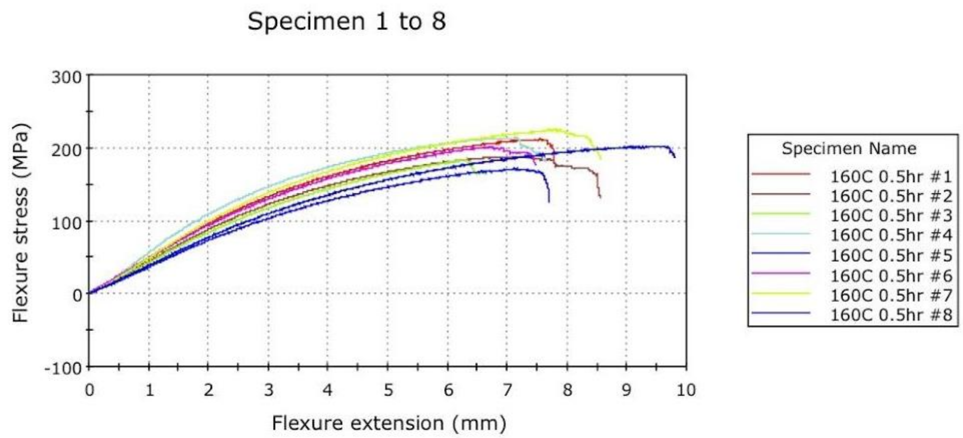

Figure 35. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 0.5 hour at 160°C heat treatment.

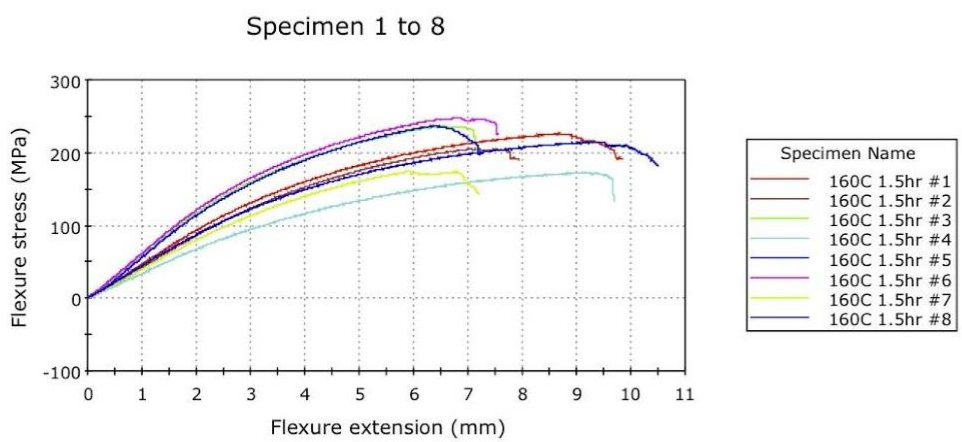

Figure 36. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 1.5 hour at 160°C heat treatment.

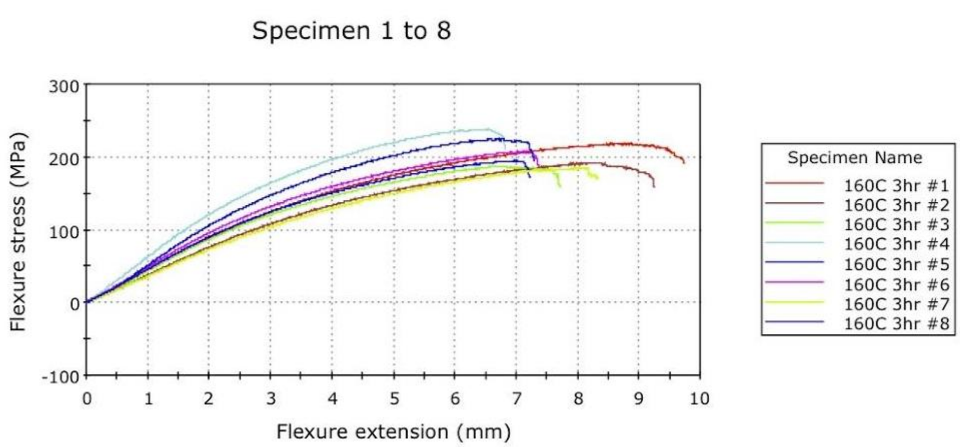

Figure 37. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 3.0 hour at 160°C heat treatment.

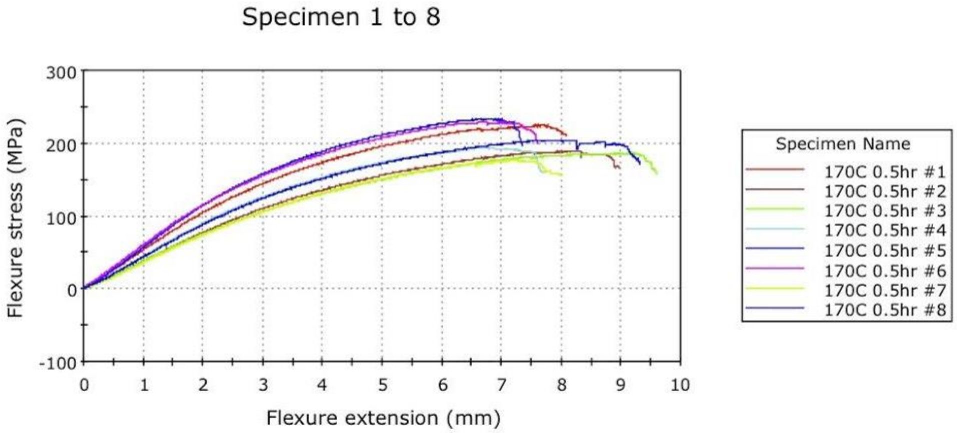

Figure 38. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 0.5 hour at 170°C heat treatment.

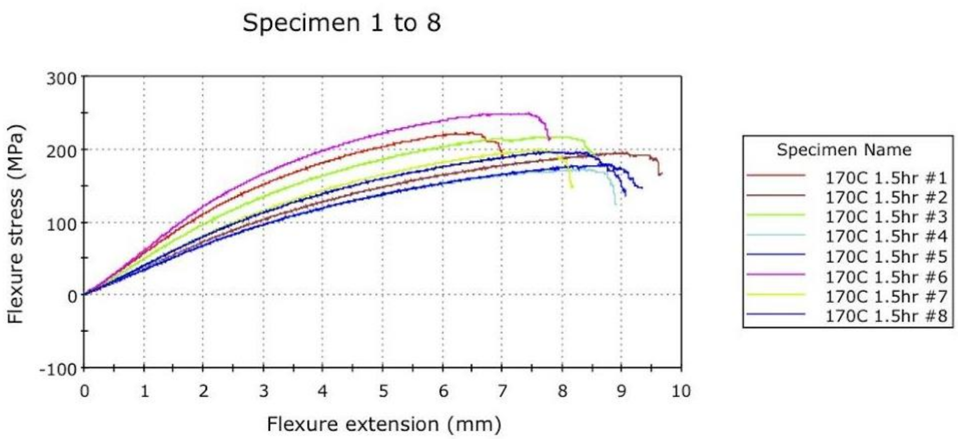

Figure 39. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 1.5 hour at 170°C heat treatment.

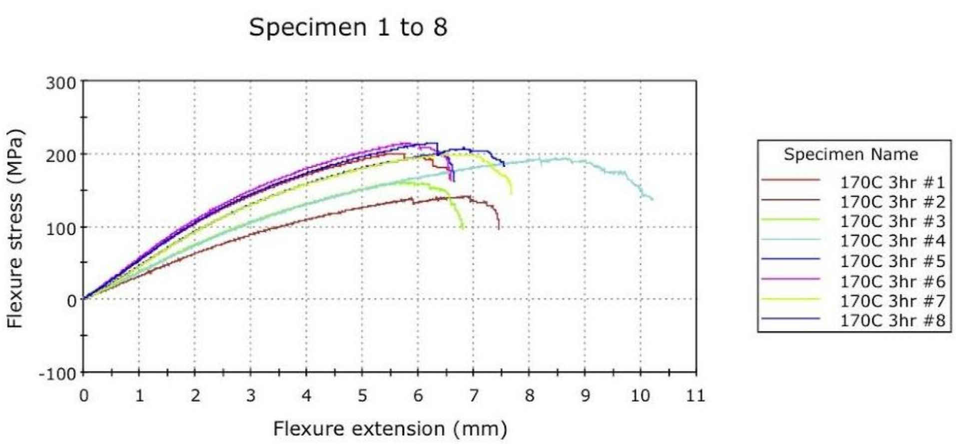

Figure 40. Raw stress-strain plot of 8 bamboo samples given a 1 hour 103°C normalization followed by 3.0 hour at 170°C heat treatment.

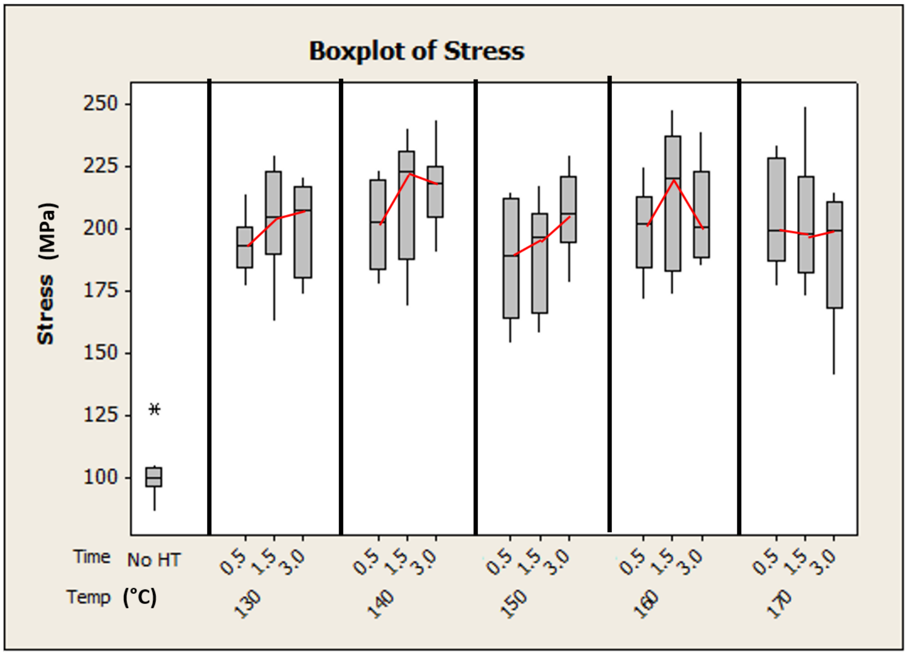

Figure 15. Stress values and means of heat treated samples from the formal testing as a function of heat treatment time and temperature.

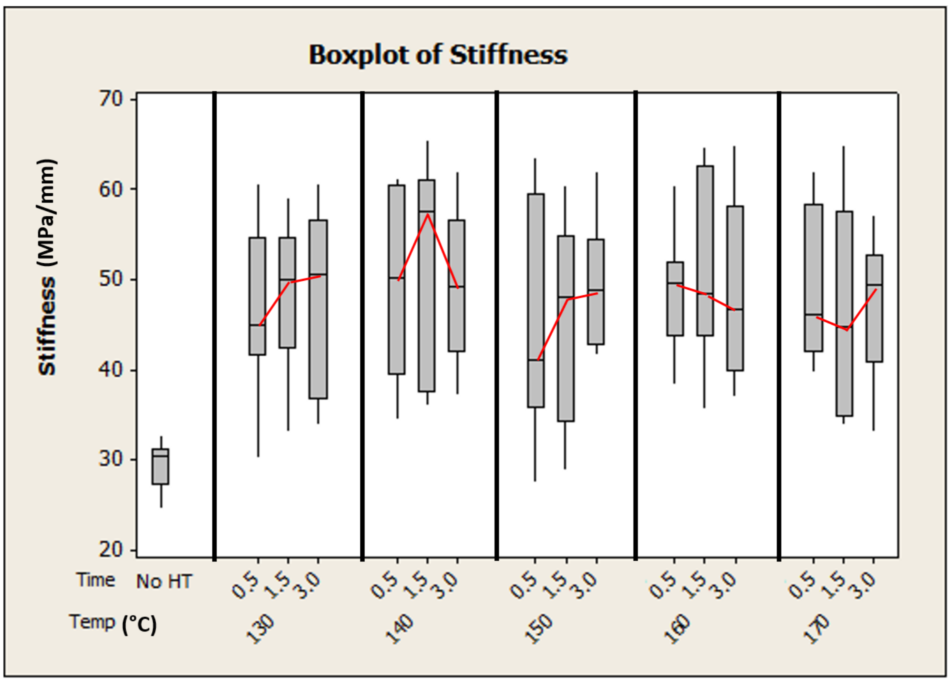

Figure 16. Stiffness values and means of heat treated samples from the formal testing as a function of heat treatment time and temperature.

```
Source   DF      SS    MS      F      P
Temp/t   15  110132  7342  17.38  0.000
Error   115   48580   422
Total   130  158712

S = 20.55   R-Sq = 69.39%   R-Sq(adj) = 65.40%


                          Individual 95% CIs For Mean Based on
                          Pooled StDev
Level   N    Mean  StDev  --------+---------+---------+---------+-
  25   11  101.64  10.03  (--*--)
1301    8  193.20  11.65                         (--*---)
1302    8  202.67  21.92                           (---*--)
1303    8  201.14  18.96                           (--*---)
1401    8  202.35  17.43                           (---*--)
1402    8  211.62  25.99                             (---*---)
1403    8  216.68  16.04                               (--*---)
1501    8  188.75  24.22                        (--*---)
1502    8  190.57  21.52                        (---*--)
1503    8  206.15  16.67                            (---*--)
1601    8  199.58  17.61                          (---*--)
1602    8  214.63  28.22                              (---*--)
1603    8  206.05  19.58                            (---*--)
1701    8  204.89  21.64                            (--*---)
1702    8  203.79  24.78                           (---*---)
1703    8  190.35  26.15                        (---*--)
                          --------+---------+---------+---------+-
                                120       160       200       240

Pooled StDev = 20.55


Grouping Information Using Tukey Method

Temp/t   N    Mean  Grouping
1403     8  216.68  A
1602     8  214.63  A
1402     8  211.62  A
1503     8  206.15  A
1603     8  206.05  A
1701     8  204.89  A
1702     8  203.79  A
1302     8  202.67  A
1401     8  202.35  A
1303     8  201.14  A
1601     8  199.58  A
1301     8  193.20  A
1502     8  190.57  A
1703     8  190.35  A
1501     8  188.75  A
  25    11  101.64    B
```

Figure 41. Tukey's Pairwise Comparison results of mean stress values generated from MiniTab statistical analysis software.

```
Temp/t   15   3992.6  266.2  2.98  0.000
Error   115  10269.0   89.3
Total   130  14261.6

S = 9.450   R-Sq = 28.00%   R-Sq(adj) = 18.60%


                              Individual 95% CIs For Mean Based on
                              Pooled StDev
Level   N    Mean   StDev  ------+---------+---------+---------+---
  25   11  29.536   2.710  (-----*----)
1301    8  46.530   9.377                 (------*-----)
1302    8  48.550   8.388                   (------*-----)
1303    8  47.569  10.282                  (------*-----)
1401    8  49.456  10.612                    (-----*------)
1402    8  52.031  12.078                      (------*------)
1403    8  49.806   8.432                    (------*-----)
1501    8  45.306  12.921                (-----*------)
1502    8  45.726  11.096                (------*-----)
1503    8  49.369   7.097                    (-----*------)
1601    8  48.744   6.595                   (------*-----)
1602    8  51.231  10.548                      (-----*------)
1603    8  48.491   9.979                   (-----*------)
1701    8  49.069   8.470                   (------*------)
1702    8  46.171  11.660                 (-----*------)
1703    8  47.350   7.773                  (-----*------)
                           ------+---------+---------+---------+---
                                30        40        50        60

Pooled StDev = 9.450


Grouping Information Using Tukey Method

Temp/t   N    Mean  Grouping
1402     8  52.031  A
1602     8  51.231  A
1403     8  49.806  A
1401     8  49.456  A
1503     8  49.369  A
1701     8  49.069  A
1601     8  48.744  A
1302     8  48.550  A
1603     8  48.491  A
1303     8  47.569  A
1703     8  47.350  A
1301     8  46.530  A
1702     8  46.171  A
1502     8  45.726  A
1501     8  45.306  A
  25    11  29.536    B
```

Figure 42. Tukey's Pairwise Comparison results of mean stiffness values generated from MiniTab statistical analysis software.