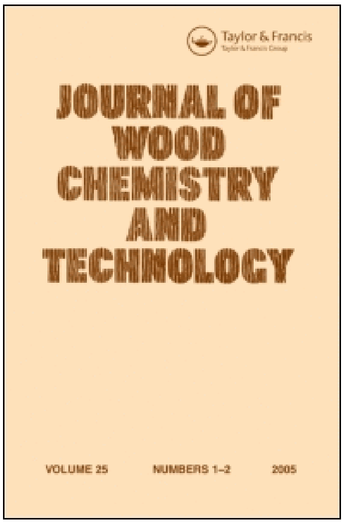

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Dynamic Mechanical Analysis of Mountain Pine Beetle–Infested Lodgepole Pine: Effect of Time-Since-Death

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Abstract: The chemical and thermo-mechanical properties of the constituent polymers mountain pine beetle (MPB)–infested Lodgepole pine were investigated by dynamic mechanical (DMA), chemical, and X-ray diffraction (XRD) analysis to validate durability as influenced by time-since-death. Chemical and XRD analysis did not show any significant differences between stages of MPB attack or location within the tree (top to bottom). However, DMA analysis revealed significant differences between the 1st and 2nd transition temperatures in the tan δ curves of the specimens.

Keywords: Chemical and mechanical properties, degradation, dynamic mechanical analysis, mountain pine beetle, X-ray diffraction

INTRODUCTION

The extent of the mountain pine beetle (MPB) infestation in British Columbia, Canada is of catastrophic proportions. Recent mild winters, fire suppression, and the remoteness of many attacked areas have allowed the beetle to thrive. Approximately 4.2 million hectares of the total 8 million hectares of mature lodgepole pine in the central interior of the province is considered to be 1–2 years dead (red attack). Assuming 50% of mature pine (more than 80 years old) will be killed, and estimating that the shelf life of the mountain pine beetle killed wood will be $\sim$15 years, the BC Ministry of Forests and Range plans to increase the allowable cut (AAC) over the next 15 years. The MPB epidemic is causing BC forest products companies to redirect planned timber development

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to absorb the immediate availability of MPB infested wood and the anticipated long-term losses in AAC.

Studies indicate that tree death is primarily caused by fungal-induced occlusions of the sapwood by sapstaining fungi. The fungal infection spreads from the bark through the sapwood until it reaches the heartwood, where the fungi are unable to grow due to high concentrations of diterpenoid resin acids.[1–3] Once the zone of occlusion encircles the tree, the tree suffers from limitations in water transport and ultimately dies.[1,4–6]

The progression of the beetle attack has been defined by three stages, based on color of tree foliation.[7] Green attack, the change from bright to dull green, occurs in the fall of the year following initial attack. This stage marks the beginning of interrupted water transport. Green attack is followed by red attack, which reflects the loss of chlorophyll in the foliage due to cell death. Trees that have been dead for more than a year and have lost most of their foliage are characterized as grey attack. According to Kim et al.[8] red and grey attack-stage trees are also vulnerable to infestation by other insects that harbor, among other things, decay fungi.

A recent study has reported that infested wood has lower lignin and hemicelluloses contents, as compared to sound wood.[9] Structurally, lignin and to a lesser extent hemicelluloses serve as an amorphous polymer matrix that consolidates the highly ordered fibrous network providing adhesion between cellulose microfibrils. To-date, there has been little or no evidence of the degradation of lignin or hemicelluloses by sapstaining fungi; loss of these components indicates co-infection by other fungi. Limited short-term dry strength tests show no change or a slight increase in strength, and available data imply the long-term behavior, especially the durability of wood products in service, could be an issue. However, the sapstaining fungi may have an adverse effect on the mechanical properties of the sapwood because they grow through the ray parenchyma and invade the wood cell lumen.[10,11] The durability issue is especially important to ensure the performance of existing wood products and the development of new wood products from MPB wood. Information on the physical, mechanical, and chemical properties of the wood will aid in establishing the shelf life and durability of the wood.

Durability of wood is directly related to its mechanical and thermal properties, which at the macro level are related to the viscoelastic properties of the wood components (polymers) at the micro level.[12] Understanding the viscoelastic properties of beetle attacked wood polymers, may enable a better understanding of the impacts of MPB infestation on wood durability and potential wood product development.

Dynamic mechanical analysis (DMA) is a powerful technique to investigate thermal and mechanical properties of polymers. DMA permits determination of the viscoelastic behavior of wood polymers and provides valuable insights into the relationship among structure, morphology, and properties of wood polymers and their composites.[13] This article explores the effect of MPB infested lodgepole pine at different stages of attack and location within the tree using dynamic mechanical analyses (DMA) in conjunction with X-ray diffraction (crystallinity) and chemical analyses.

EXPERIMENTAL

Materials

MPB attacked wood at <6 months ("green"), 1 year ("red"), and 3 years ("grey") were collected from the interior of British Columbia. Ten trees of each stage of attack were processed through a sawmill wherein a complete mill cleanout was performed between each condition to ensure no cross contamination. Blocks were cut from 1 × 4 sapwood samples from boards representing three positions up the tree: bottom (B), middle (M), and top (T). The wood specimens were machined to the appropriate size for dynamic mechanical analysis (DMA): 35 mm (length, longitudinal) × 12 mm (width, radial) × 2.5 mm (thickness, tangential). Samples were taken from blocks having the most discoloration to minimize errors according to non-uniform degradation. The samples were conditioned at 20°C and 65% relative humidity (RH). All wood samples were then categorized according to Table 1, mixed and randomly selected for analysis. DMA, chemical, and X-ray (XRD) analysis were conducted on all samples.

Chemical Analysis

For each stage of attack, three samples from each position within the tree were combined and subjected to lignin and carbohydrate analyses. Wood specimens were milled in a Wiley mill using a 40-mesh screen. The milled wood (wood meal) was oven-dried at 105°C overnight and stored in a 40°C vacuum oven for at least 1 week prior to analysis. The wood meal was equilibrated to ambient temperature and extracted with acetone for 48 h using a Soxhlet apparatus according to TAPPI Method T264cm-97. Klason and acid-soluble lignin (ASL) were determined on the extracted wood meal according to TAPPI Method T222

Table 1. MPB attack sample designation

	Green	Red	Grey
Top	TN	TR	TG
Middle	MN	MR	MG
Bottom	BN	BR	BG

om-98 and TAPPI Useful Method UM250, respectively. Carbohydrate content was determined by sugar analysis of the Klason lignin analysis filtrate using High Performance Anion Exchange Liquid Chromatography. The HPLC system (Dionex DX-500, Dionex, CA, USA) was equipped with an ion-exchange PA1 (Dionex) column, a pulsed amperometric detector with a gold electrode, and a Spectra AS3500 auto-injector (Spectra-Physics, CA, USA). Prior to injection, samples were filtered through 0.45 μm HV filters (Millipore, MA, USA) and a 20 μL volume of sample was loaded, containing fucose as an internal standard. The column was equilibrated with 250 mM NaOH (Fisher Scientific, Nepean Ont.) and eluted with de-ionized water at a flow rate of 1.0 mL·min^{-1}.

Sample Conditioning

Four different pretreatment conditions were used in this study. The wood samples were: (1) conditioned at 60% relative humidity and 20°C for 1 week (referred to as Cond); (2) soxhlet extracted with acetone (24 h) followed by conditioning at 60% relative humidity and 20 °C for 1 week (Ex-Cond); (3) soaked in water for 7 days followed by conditioning at 60% relative humidity and 20°C for 1 week (S-Cond); and soaked in water for 7 days and then oven–dried overnight at 105°C followed by conditioning at 60% relative humidity and 20°C for 1 week (SD-Cond). The moisture contents of among conditioned samples were measured prior DMA.

Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis was performed with a TA Instruments Q800 DMA using a dry-nitrogen purge of the sample chamber and dual-cantilever bending geometry. Samples were secured with a clamping torque of ~110 N·cm. All experiments were replicated 3–5 times. Prior to dynamic thermal testing, the linear viscoelastic region (LVR) of the samples was determined using a strain sweep,[13] where the sample was sinusoidally deformed with increasing amplitude/strain at a constant frequency (1 Hz). The LVR tests were conducted at six different temperatures covering the temperature range of –145 to 150°C. All dynamic scans (from –145 to 150°C) were performed at a heating rate of 3°C·min^{-1} at constant frequency (1 Hz) with constant stain (0.1%). Samples were rapidly cooled to –145°C using liquid nitrogen, and equilibrated for 10 min prior to scanning.

X-Ray Diffraction (XRD)

X-ray diffraction analysis of the wood specimens was performed immediately before and after DMA using a Bruker AXS D8 Discover X-ray diffractometer.

Samples were mounted with the grain perpendicular to the detector at a distance of 10 cm. The goniometer was tilted at an angle of 17° and data were collected over the range $4^\circ < 2\theta < 40^\circ$. The radiation source was Cu and the generator was set to 40 kV and 20 mA. Collection time was 100s. Data were collected from two different points for each sample in order to obtain representative diffraction patterns. After subtraction of the incoherent scattering, the diffraction patterns were normalized to the height of the 200 reflection, which occurs at $2\theta \sim 22^\circ$.

Statistical Analysis

Analysis of variances (ANOVA) was conducted between groups of data. The analysis followed single factor ANOVA for groups having different Standard Deviations. An assumption was made, based on having independent variables (stages of attack) when comparing various locations in a tree; and independent variables (location) when comparing different stages of attack. Namely, we assumed each type of attack is caused, based on its own environmental and growth conditions, and does not reflect any other part or stage of attack property. Analysis included chemical analysis data and DMA data for the 1st and 2nd transition temperatures.

RESULTS AND DISCUSSION

Chemical Analysis

The results of Klason lignin, acid-soluble lignin (ASL) and sugar analysis are shown in Table 2. Standard deviations are reported in parentheses. ANOVA

Table 2. Lignin and carbohydrate composition of wood samples

MPB	Arabinose	Galactose	Xylose	Mannose	Glucose	Klason lignin	ASL
TG	1.29(0.13)	1.86(0.07)	6.07(023)	12.06(031)	45.98(0.77)	30 (0.38)	0.54(0.0)
MG	1.28(0.03)	1.6(0.01)	5.39(0.04)	11.64(0.09)	46.7(0.30)	30.1(1.13)	0.53(0.0)
BG	1.08(0.02)	1.85(0.06)	5.74(0.08)	11.34(0.21)	46.61(0.15)	30.1(0.95)	0.53(0.0)
TR	1.22(0.08)	1.61(0.07)	5.99(0.25)	10.97(0.45)	44.42(0.02)	31.1(0.26)	0.54(0.0)
MR	1.24(0.02)	1.69(0.01)	6.11(034)	11.3(0.66)	46.17(0.65)	29.1(0.38)	0.57(0.0)
BR	1.17(0.05)	1.59(0.05)	5.38(0.13)	11.78(0.30)	46.75(1.29)	28.9(1.07)	0.58(0.0)
TN	1.3(0.00)	1.55(0.09)	5.96(0.10)	11.72(042)	47.03(1.24)	29.2(0.38)	0.59(0.0)
MN	1.25(0.05)	1.38(0.02)	5.98(0.39)	11.85(0.64)	47.36(0.98)	28.3(0.38)	0.53(0.0)
BN	1.24(0.05)	1.66(0.08)	5.9(0.20)	11.17(0.27)	47.17(1.74)	29.4(0.50)	0.54(0.0)

analysis did not show any significant differences between stages of attack and tree height of attack for any of the carbohydrate monomers, or lignin (data not shown). Although mannose and xylose show some differences between stages of attack, they do not exhibit any trend. This may be because of the high standard deviations. Variation in chemical analysis among stages of attack can be discussed based on non-uniform degradation. According to Blanchette and Abad[14] chemical analysis is a misleading method to analyze decayed wood because degradation is not uniformly distributed. In this study, we tried to prepare samples from attacked areas (highly discolored areas) of wood blocks, but it seems that degradation is highly non-uniform and occurs at the molecular level, which cannot be distinguished based on staining or changes in color. Ballard et al.[11]reported that MPB attacked lodgepole pine trees retain the ability to maintain water in the needles for a long period of time, in fact dying prior to any needle browning. Our findings, wherein no compositional changes occurred among the three "stages" of attack, is in agreement with this and supports the notion that the MPB attacked trees may have been dead at the green stage of attack, and the transition from green to red, and grey is based on the resistance of the needles to browning.

Dynamic Mechanical Analysis

LVR Analysis

Prior to DMA the LVR had to be determined for the MPB samples. Figure 1 shows the result of the LVR analysis of samples at different temperatures. The LVR limit was the same range for all of the tested temperatures (0.1 to 0.25%). As a result a strain of 0.1% was chosen for the DMA tests.[13]

DMA of Conditioned Samples

In DMA, a dynamic force/strain is applied and the material response is captured in a viscoelastic manner. When internal molecular motion of segments within the polymer chains occurs, the resulting strain/stress lags behind the applied stress/strain. The tangent of this lag (tan δ) is analyzed as a dynamic response; or transition when ramping temperature. Usually, amorphous polymers exhibit more than one transition in the linear viscoelastic region. A high-temperature transition (α or 1st transition) which is highly dependent on the moisture content, and secondary transitions (2nd or β and γ). Studies show that the interval of a transition depends on the degree of crystallinity of the polymer,[15] with peak sharpness increasing with increasing crystallinity.

In wood, the lower temperature 2nd transition, has been reported to arise from methylol groups in lignin and xylans.[16] As our samples are softwood, wherein the xylan composition is only 5–10% of the total hemicellulose content, this transition primarily represents methylol groups in lignin. Thus, any

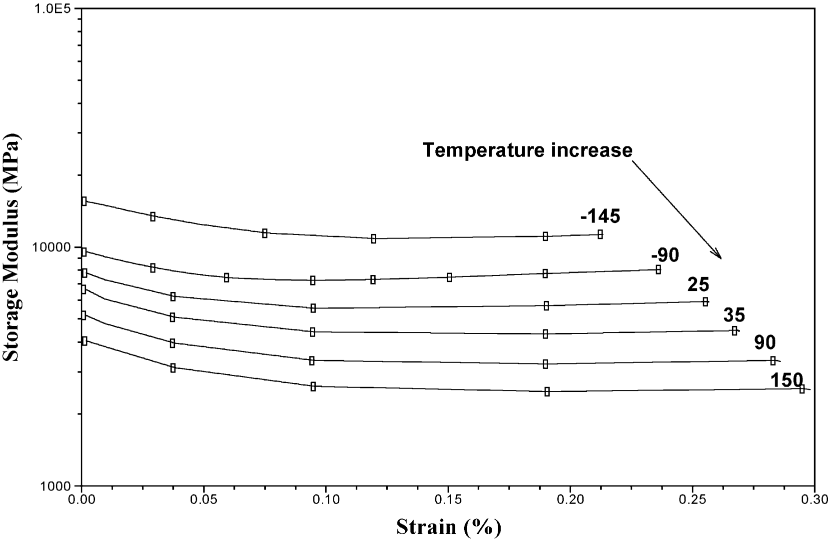

Figure 1. Linear viscoelastic region (LVR) determination of MPB wood samples (temperatures in °C).

difference observed in the 2nd transition would imply a probable difference in the lignin structure or macromolecular superstructure. The higher 1st transition has been reported as representing the glass transition temperature of lignin in the presence of moisture.[17,18] In fact, Sun et al.[19] concluded that there is no transition for completely dry wood between room temperature and 150°C, and suggest that any transition in this temperature range likely arises from the presence of water molecules, which act as a plasticizer.[20] Plasticization causes a reduction in the energy required to initiate chain mobility for the amorphous components of the wood. Therefore, any differences in the 1st transition temperature (in the presence of water/moisture), may further suggest changes in lignin structure/macromolecular superstructure. However, the precise temperature at which the transitions are found can be expected to vary with moisture content.[17]

Figure 2 illustrates tan δ results for conditioned samples at all 9 categories (3 stages of attack and 3 positions within the tree). All other data, a total of 144 tests, exhibit similar profiles (data not shown). Two distinctive broad transitions were observed and the tan δ peaks of both transitions showed variations between categories, based on stage of attack or location within the tree. As all of the samples were conditioned at 60% relative humidity and 20°C, all had an initial moisture content of $\sim$11 wt%, which likely changed as a result of the temperature sweep experiment. To observe the hygrothermal response of the wood samples, bottom grey (BG) samples were treated isothermally

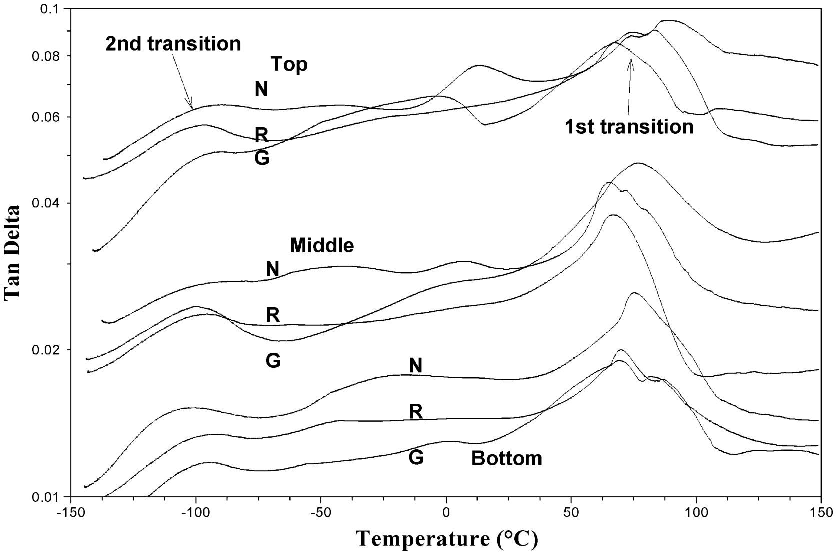

Figure 2. DMA tan δ curves of MPB attacked wood; three stages of attack (N, R, G) and three positions within the tree (Top, Middle, Bottom).

at 40, 60, 80, and 100°C for 30 min before scanning. Figure 3 shows the DMA results of the hygrothermal analysis. The results showed similar tan δ profiles at all temperatures except 100°C, indicating the moisture content remains almost constant during heating to 100°C. Heat treatment at 100°C caused complete desiccation and a change in the 1st transition. Consequently, the 1st transitions can be assigned to the glass transition of water plasticized lignin.

ANOVA analysis of the DMA data showed some differences between the conditioned samples. Tables 3 and 4 show the result of the ANOVA analysis. The analysis reveals that only some of the transition temperatures have significant differences (95% levels of confidence). Table 3 exhibits significant differences for the 1st transition of the top of tree samples and the 2nd transition of the middle part of the tree samples. Table 4 shows differences for the 1st transition between red and grey stages of attack, as well as with the 2nd transition of the green attack stage.

It seems that for all of the data the 1st transition is more affected than the 2nd transition. Also the transition is more related to the position within the tree than stage of attack, which agrees with the result of chemical analysis regarding to non-uniform degradation. The observation of different transition temperatures might be related to variation and heterogeneity of the amorphous polymers in the MPB wood specimens. The variation in the tan δ transition can be rationalized on the basis of differences in both density and

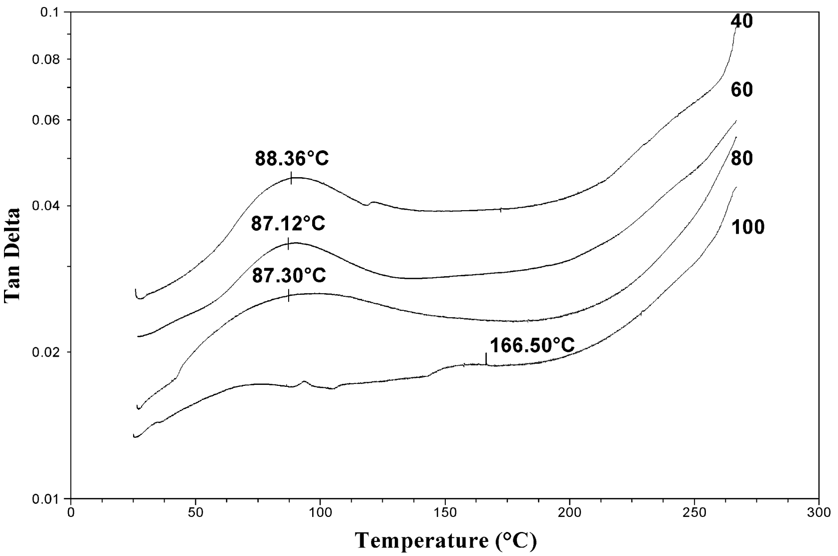

Figure 3. Hygrothermal effect on the 1st transition in the tan δ curves of BG specimens. DMA scans of BG specimens after isothermal desiccation at 40, 60, 80, and 100°C. (3°C/min heating rate, 1 Hz frequency, and 0.1% strain.)

frequency of crosslinking within the wood's amorphous components, lignin and hemicellulose.[17] The height of the main tan δ peak is a relative measurement of the amount of material taking part in the transition. Variation in the height of the tan δ peaks may reflect the change in content of amorphous wood components in the different samples.[21]

Table 3. ANOVA analysis of MPB DMA data based on tree position; analysis of 1st and 2nd transition temperature differences

Degree of freedom: 2 (between groups), 6 (within groups),
8 (total) F critical = 9.55

Source	1st transition temperature			2nd transition temperature		
Analysis factors	Top	Middle	Bottom	Top	Middle	Bottom
F calculated	112.7	19.22	1.62	0.47	551.66	0.60
Probability	0.002	0.190	0.270	0.580	0.0001	0.670

Table 4. ANOVA analysis of MPB DMA data based on stage of attack; analysis for 1st and 2nd transition temperature differences

Degree of freedom: 2 (between groups), 6 (within groups),
8 (total) F critical = 9.55

Source	1st transition temperature			2nd transition temperature		
Analysis factors	Green	Red	Gray	Green	Red	Gray
F calculated	1.29	51.64	41.85	123.69	0.88	10.47
Probability	0.390	0.005	0.006	0.001	0.500	0.044

DMA at Different Preconditioned Samples

The result of ANOVA analysis did not show any significant differences for tan δ between stage and height of attack when wood samples were soaked, soak-dried, and extracted before conditioning at 60% RH and 20°C (data not shown). The results, however, were different from one treatment to the other. Figure 4 illustrates the results obtained for the 1st transition for the different pretreatments. It can be seen that the conditioned sample has the highest tan δ value of all samples regardless of location within the tree or stage of attack, followed by the soaked-dried-conditioned (SD-cond), extracted-conditioned (Ex-cond), and soaked-conditioned (S-cond) samples. Exceptions are top-normal (TN), which exhibits soaked-conditioned as the highest tan δ; and top-red (TR) in which

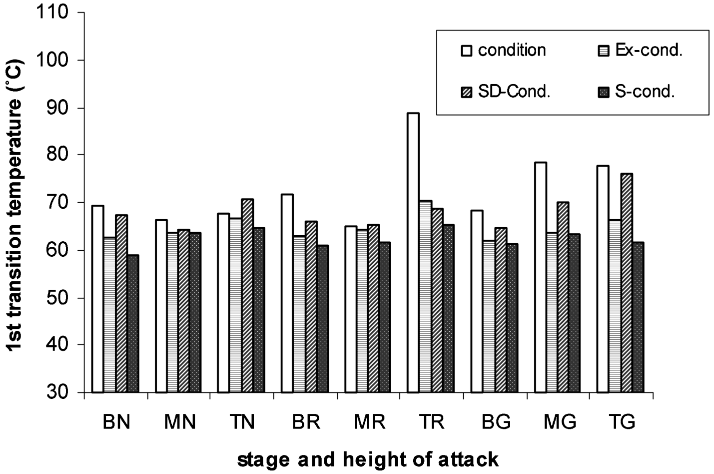

Figure 4. Analysis of tan δ results for the 1st transition at different pretreatment conditions.

extracted-conditioned exceeds soaked-dried-conditioned. As mentioned, the 1st transitions are attributed to the lignin component. It is reported that the glass transition of amorphous polymers like lignin is influenced by the main-chain flexibility, the nature of the side groups, and the presence of plasticizers.[22] All samples had the same initial moisture content prior to DMA, and therefore the same plasticizer effect. However, depending on the pretreatment, the samples may have different supramolecular properties, which could contribute to a difference in thermal and physical history, both of which can affect transition temperatures.[15] Acetone extraction of samples will remove extractives as well as some polysaccharides.[23] Likewise, water soaking, which will remove water soluble materials, swells wood and saturates the amorphous regions. Then depending on how the water is removed, fast (oven-drying) or slow (conditioned) the physical environment may differ; kinetic versus thermodynamic arguments. Further conditioning of oven-dried samples might reinforce the absorption of water into the amorphous regions rather than natural hysteresis. Soaked-conditioned samples, however, have sufficient time to loose excess water and keep the already saturated amorphous regions without stress. It means that soaked-dried-conditioned samples might exhibit properties of less amorphous domains, which agrees with the tan δ results.

Figure 5 shows the tan (δ) results for the 2nd transition temperature at different preconditions. It seems that the different pretreatments have less effect on the 2nd transition than on the 1st transition (Figure 4). Here, the extracted-conditioned (Ex-Cond) sample shows the lowest 2nd transition temperature. As mentioned, methylol groups have been reported as being responsible for the 2nd transitions.[16] Methylol groups might be affected by acetone extraction and heat drying, which could shift the temperature.

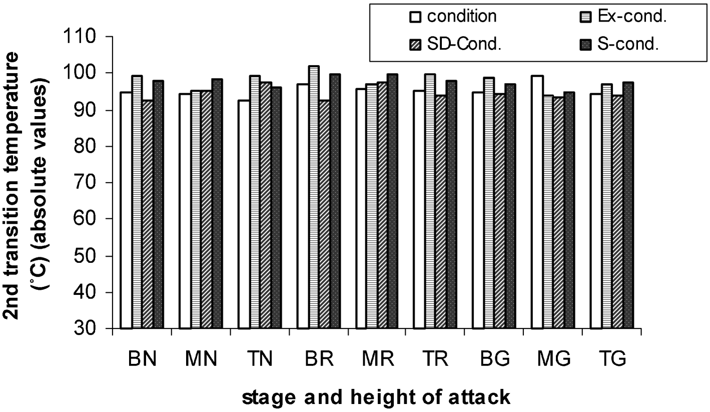

Figure 5. Analysis of tan δ results for the 2nd transition at different pretreatment conditions.

X-Ray Analysis

The degree of crystallinity (X_c) was the same for all samples (50 ± 1) before DMA. This implies there is no significant difference in the wood polymers in the different levels of attack, consistent with the chemical analysis and broadness of the tan (δ) profiles. X-ray analysis showed a drop in crystallinity to $X_c \sim 40$ when samples were analyzed immediately after being heated in the DMA chamber. Interestingly, this decrease in crystallinity was only temporary, as the samples revealed the same initial degree of crystallinity after three weeks of conditioning. It seems that heating the wood specimens up to 150°C and applying a controlled strain affects intermolecular interactions, perhaps introducing stress between microfibrils and matrix substances. Upon removal of the applied strain the wood constituents are able to slowly relax back to the initial chemical/physical environment.

CONCLUSIONS

Statistical analysis was conducted on the results obtained from both chemical and dynamic mechanical analysis of MPB attacked wood. The results of chemical analysis did not show any significant differences between stages of attack and location within the tree (top to bottom). Likewise, X-ray diffraction results showed similar crystallinity values (50 ± 1) for the different samples. DMA analysis revealed some significant differences between the 1st and 2nd transition temperatures in the tan δ curves of wood specimens conditioned at 60% relative humidity and 20°C; proposed to be the effect of variations in structure and composition between trees and within a tree rather than levels of attack. Based on these analyses, it seems that there is no significant chemical or structural difference between the MPB attacked Lodgepole pine investigated in this study as a function of time-since-attack.

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