

## Mechanism of Strength Loss in Heat Treated Softwoods

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### ABSTRACT

Heat treatment conditions, appropriate for improvement of stability and fungal resistance, are known to reduce the breaking strength of treated timber relative to untreated controls. In the case of fast grown Sitka spruce and lodgepole pine, tested in three point bending, reductions in modulus of rupture of up to 50% were observed following application of established heat treatment processes, whilst moduli of elasticity remained substantially unchanged. Stress-strain curves for the heat treated materials are linear up to the point of abrupt fracture, which occurs at small deformation relative to controls. Untreated control materials show a more progressive fracture involving much greater work, judged by the area under the curve. Electron micrographs of fracture surfaces showed that in the case of control materials, cell pullout had occurred with cell walls remaining largely intact. In comparison heat treated materials clearly show cell wall cleavage and significant delamination of the cell wall layers. In particular the micrographs show separation of the middle lamella. It is proposed that lignin degradation within the middle lamella and within the cell wall is removing one of the essential conditions for composite material behaviour, in that stress transfer and stress distribution are seriously compromised by matrix degradation. Dynamic mechanical analysis shows that there is a small change in the relationships between timber stiffness and temperature, as assessed by the temperature coefficient of modulus. Timber stiffness under small strain is substantially determined by such factors as microfibril angle and this will be largely unchanged by heat treatment, whereas timber toughness under large strain is a function of the ability to transfer and distribute stress between fibrils, and this is dependent upon matrix integrity.

### INTRODUCTION

The general effects of heat treatment on wood mechanical properties are well known with reductions in breaking strength correlating approximately with the thermal energy input to the wood. The results presented here come from a study of the effects of heat treatment on fast grown softwoods from Irish forests, and the topic of particular interest is the mechanism of strength and stiffness loss, and the magnitude of these effects. The species evaluated were Sitka spruce, (*Picea sitchensis*), lodgepole pine (*Pinus contorta*) and Japanese larch (*Larix leptolepis*), chosen simply on abundance within the forest system, and heat treatment was carried out using two commercially available processes. Process A uses dry heat with constraint of the timber between platens, whilst Process B using a hygrothermal technology. Process A presents a greater thermal challenge to the timber than process B. No attempt is being made in this paper to compare the two processes in terms of their efficacy, rather the attempt is to use them as two degrees of severity in commercial processes. Both processes were effective in their primary aim of improving dimensional stability against moisture change and fungal durability.

## EXPERIMENTAL

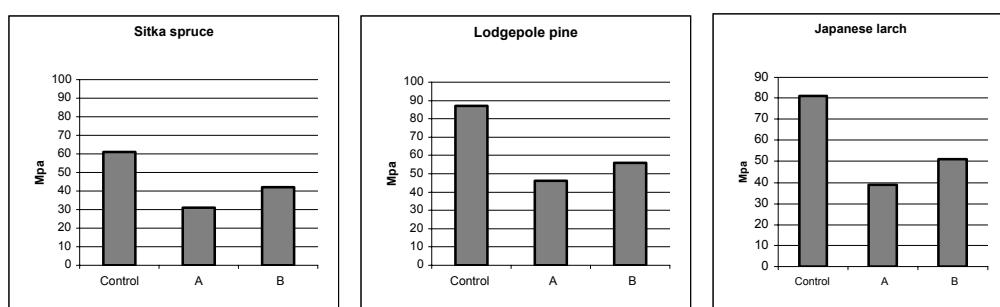
Samples for mechanical testing, of dimensions 300 x 20 x 20 mm were machined from boards of cross-section 150 mm by 45 mm and twenty five specimens were tested for each result. Samples were tested, using the single central load method, on an Instron 4301 series test frame in accordance with B.S. 373 and MOE and MOR were calculated in the usual way. Fracture surfaces were examined by scanning electron microscopy using a Jeol instrument.

Dynamic mechanical analysis was carried out using a Polymer Labs driven frequency instrument operating at 0.1, 1.0 and 5.0 Hz. Samples 45 x 10 x 5 mm were cut in the longitudinal direction and were oven dried before testing. A heating rate of 2 °C per minute was used between –100 °C and 150 °C and the parameters measured were the torsional shear modulus and tan δ. The torsional shear modulus at 1 Hz was then used to calculate a temperature coefficient of modulus in the manner previously used to examine fungal decay effects (McCarthy *et al.* 1999, Kollman and Côté 1984).

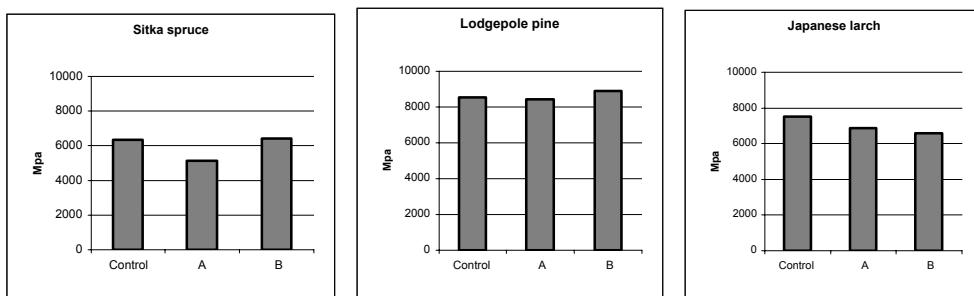
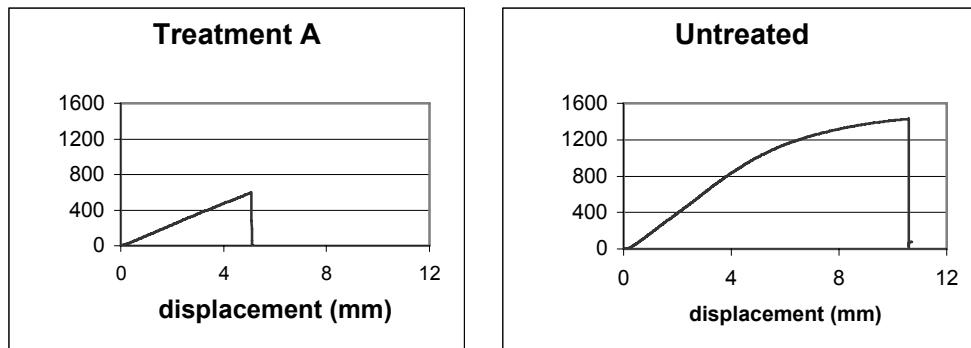
## RESULTS AND DISCUSSION

Modulus of rupture results are shown in Figure 1. There is consistency across the three species with significantly reduced modulus values for the modified wood when compared with the untreated control samples. There was also a larger scatter in both sets of treated samples than in the control samples, as shown by the standard deviation, and a large number of treated samples demonstrated sudden brittle failure.

Modulus of elasticity results are plotted in Figure 2 and in comparison with the MOR results, property changes are slight. The most significant change occurred in spruce modified by Treatment A, where MOE reduced by about 20% of the control value. Both spruce and pine showed a small increase under Treatment B conditions. Larch decreased by approximately 10% for each treatment. Examples of the stress-strain plots for Sitka spruce are shown in Figure 3, and very substantial reductions in work to fracture are evident following treatment.



*Figure 1: Modulus of rupture results.*

**Figure 2: Modulus of elasticity results.****Figure 3: Examples of three-point bend results for treated and untreated Sitka spruce samples.**

In general these results are consistent with those obtained using slower grown northern European softwoods where loss of strength has been reported by many previous workers. Early work by Seborg *et al.* (1953) specified losses of 50% and Davis and Thomson (1964) and Giebelser (1980) also reported significant strength reduction. Vernois (2001) reported strength losses up to 40% whilst Boonstra *et al.* (1998) and Militz (2002) report losses ranging from 5-18% in process conditions similar to Treatment B in this work. Similarly only moderate change in stiffness is to be expected, for example Rapp and Sailer (2000) and Militz (2002) report only small changes in MOE for a number of species modified by different heat treatment methods.

Figure 4 - 7 shows micrographs of typical Sitka spruce fracture surfaces for control, for Treatment A and for Treatment B. In the lower magnification images there is evidence of cell pullout in the control material and in the larger magnification images the cell walls are clearly defined and seem largely intact. In these samples fracture was gradual and progressive requiring considerable amounts of energy, as demonstrated in the work to fracture curves in Figure 3. In comparison the heat treated material fracture surfaces are consistent with abrupt failure, particularly with Treatment A. At high magnification considerable fragmentation of the cell wall is shown with separation of the middle lamella.

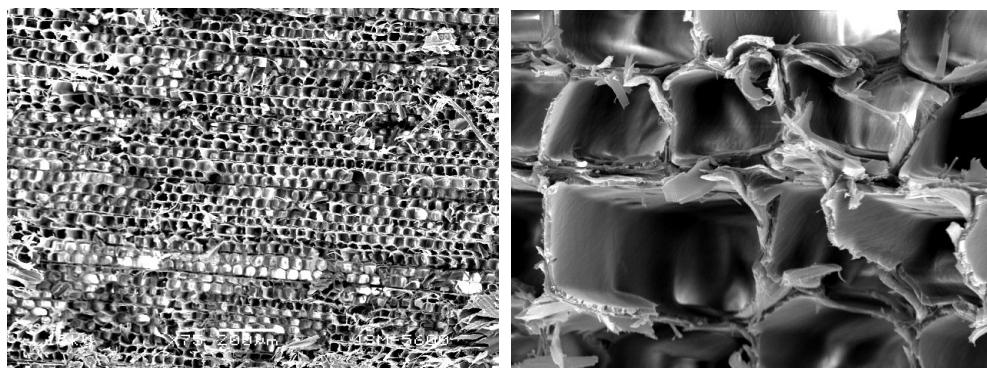


Figure 4: Fracture surfaces of Sitka spruce following Treatment A.

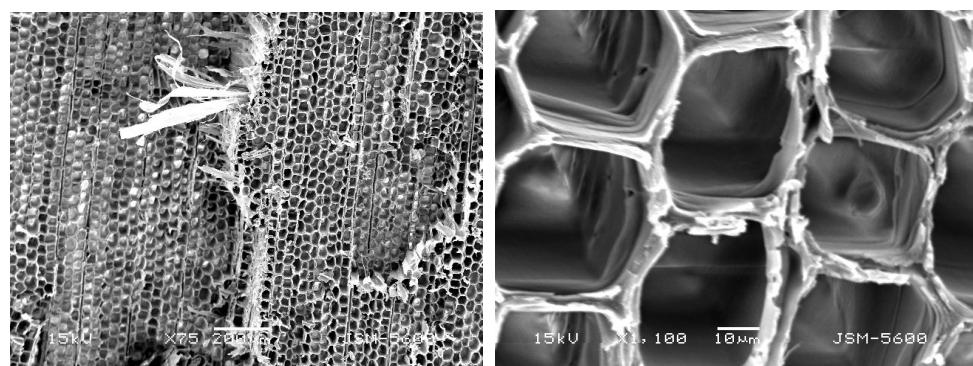


Figure 5: Fracture surfaces of Sitka spruce following Treatment B.

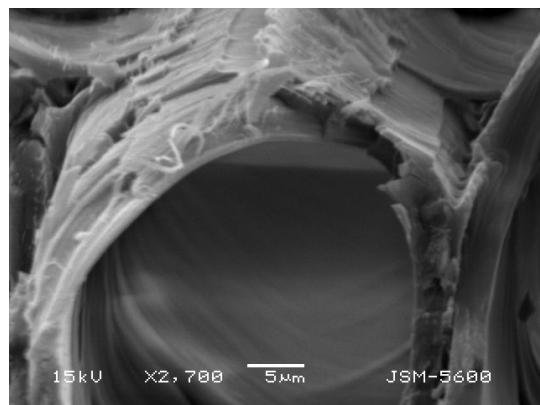
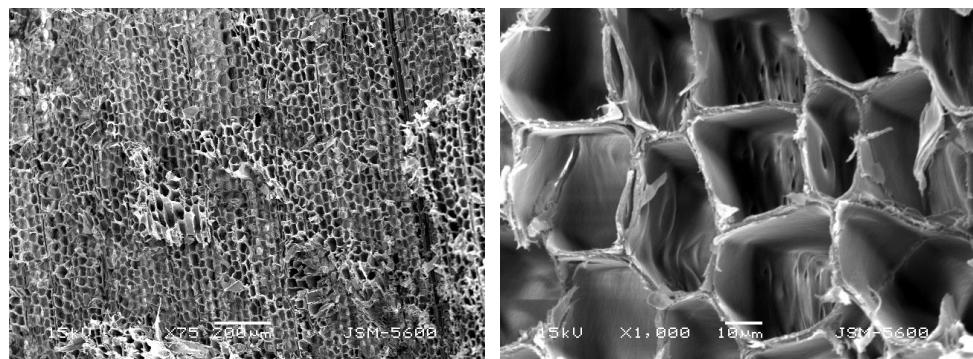


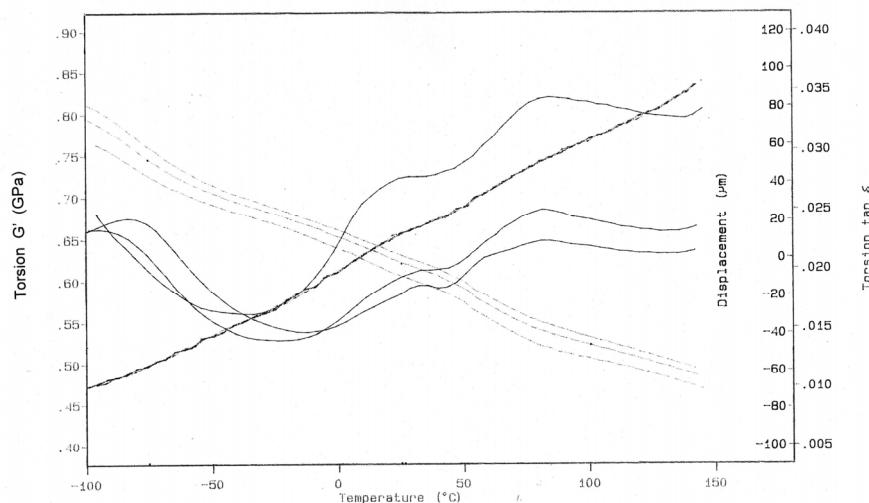
Figure 6: Detail of a Treatment A fracture surface showing individual microfibrils which have broken free from the matrix.

Figures 8 and 9 shows typical dynamic mechanical analysis plot for Sitka spruce control and for treatment B, and broad similarity in behaviour is apparent. The damping maxima around 50 °C arise from relaxation processes in the lignin and hemicelluloses with the lower temperature event probably arising from the hemicellulose (Kelley *et al* 1987). The procedure for calculating a temperature coefficient of modulus is to compare the moduli at -70 °C and at 100 °C, that is above and below these transitions, and this gives the dimensionless values in Table 1 which represent the average of several runs. In general there is small reduction in the temperature coefficient of dynamic stiffness following heat treatment and this increases with the severity of the treatment. In one sense this parameter is a measure of the thermoplasticity of the wood

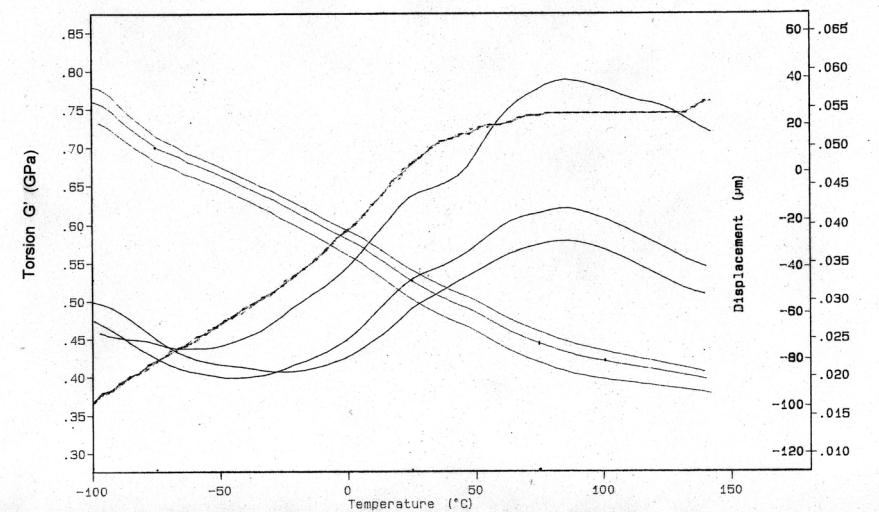
and a reduction indicates reduced thermoplasticity which is consistent with hemicellulose degradation.



**Figure 7:** Fracture surface of untreated Sitka spruce.



**Figure 8:** Dynamic mechanical response of Sitka spruce control.



**Figure 9:** Dynamic mechanical response of Sitka spruce subject to Treatment B.

It is generally considered that timber stiffness under small strain is substantially determined by such factors as microfibril angle, (Yang and Evans 2004, Barnett and Bonham 2004, Hofstetter *et al.* 2006) and although this will be largely unchanged by heat treatment stress transfer at the fibril level will be dependent upon the integrity of the composite matrix and the micrographs show that this has been compromised. At larger strain, timber toughness measured by work to fracture, is a function of the ability to transfer and distribute stress between fibrils and between cells, and this is much more critically dependent upon matrix integrity which is clearly being severely reduced by the heat treatment process.

**Table 1: Effect of heat treatment on the temperature coefficient of dynamic modulus.**

Sample	Sitka spruce	Lodgepole pine	Japanese larch
Control	-1.7 x 10 <sup>-3</sup>	-2.1 x 10 <sup>-3</sup>	-1.8 x 10 <sup>-3</sup>
Treatment A	-1.5 x 10 <sup>-3</sup>	-1.9 x 10 <sup>-3</sup>	-1.3 x 10 <sup>-3</sup>
Treatment B	-1.2 x 10 <sup>-3</sup>	-1.9 x 10 <sup>-3</sup>	-1.9 x 10 <sup>-3</sup>

## CONCLUSIONS

Considering the results overall, both the change in fracture behaviour and the change in the stiffness-temperature relationship are consistent with hemicellulose degradation. In a composite material, matrix integrity is essential for efficient stress transfer, and both of the trends observed here, under elastic deformation at low strain and under high strain fracture, are indicative of matrix change. The cell wall fragmentation evident in the micrographs, particularly under the more severe treatment condition suggests failure of the binding polymers and the dynamic mechanical results confirm some loss of stress transfer mechanisms is occurring at stress levels much lower than those required to cause catastrophic failure.

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