3rd New Zealand and German CELL WALL SYMPOSIUM

SCION Next generation biomaterials

New Zealand Journal of Forestry Science

39 (2009) 243-250

http://nzjfs.scionresearch.com

Density, microfibril angle and modulus of elasticity as indicators of intra-ring checking in *Pinus radiata* wood†

Hema Nair*, Sandra Jackson (deceased) and Brian Butterfield

School of Biological Sciences, University of Canterbury, Private Bag 4800, Christchurch, New Zealand.

(Received for publication 23 December 2008; accepted in revised form 16 November 2009)

*corresponding author: hema.nair@canterbury.ac. nz

Abstract

Intra-ring checking (checking) is a wood quality defect that can develop sometimes in *Pinus radiata* D.Don (radiata pine) wood during the drying process. The aim of this small study was to examine various mechanical properties of intra-ring-checked wood to determine how they differed from non-checked wood. This would help understand which of these mechanical properties (if any) might make wood susceptible to checking. Three mechanical properties of wood (density, microfibril angle (MFA) and modulus of elasticity (MOE)) were examined in oven-dried radiata pine disks that displayed different degrees of checking. These measurements were made using SilviScan-2 equipment. Microfibril angle was also examined using X-ray diffraction (XRD) while MOE data was also obtained using a modified version of a Fullam micro-test stage.

None of the data obtained for any of the mechanical properties showed significant differences relative to checking. However, some trends could be observed that helped draw some conclusions about the mechanical properties observed in checked and non-checked wood. The checked wood samples had a lower density than the non-checked wood samples by SilviScan-2 analysis. When measured by XRD, the MFA of checked wood was higher than that of non-checked wood. However, when measured by SilviScan-2, the MFA of checked wood and non-checked wood did not show such clear differences. There was also no significant difference in the MOE of checked and non-checked wood obtained by either SilviScan-2 or by Fullam micro-test stage equipment. In summary, the three properties measured were insufficient predictors of checking in this small set of radiata pine samples.

Keywords: *Pinus radiata;* intra-ring checking; wood quality; wood properties; density; MFA; MOE

†Based on a paper presented at the 3rd Joint New Zealand – German Symposium on Plant Cell Walls, 13-15 February 2008, Auckland, New Zealand

Introduction

Radiata pine is the dominant plantation tree in New Zealand and is an important source of export revenue (Jayawickrama et al., 1997; Withers & Keena, 2001; Ministry of Agriculture and Forestry, 2005). Due to its economic importance, there is keen interest in trying to improve the quality of radiata pine wood in New Zealand. Radiata pine wood can develop a wood quality flaw referred to as 'intra-ring checking' (checking) usually during drying (Booker, 1995), although sometimes it has also been observed in green timber (Ball et al., 2005). The checks are usually confined to earlywood (Chafe, 1995) and extend radially through it (Booker et al., 2000) (Figure 1). The occurrence of checks lowers the value of appearance grade timber leading to huge economic losses for the forest industry (Putoczki et al., 2007). The aim of this study was to determine whether certain mechanical properties differed between intra-ring-

 ISSN 0048 - 0134

checked (checked) wood and non-checked wood. If so, these properties could be used as indicators for the tendency of wood to develop checks during drying. Density, MFA and MOE are all reputedly determinants of wood quality. Knowledge of these wood properties can help in efficient utilisation of wood. Hence, these mechanical properties were chosen for this study between checked and non-checked wood.

Density is considered generally a good predictor for wood strength and stiffness (Panshin & de Zeeuw, 1980). However, MFA of the secondary cell wall layer (mainly the S_2 cell wall layer in a tracheid) is also important. The secondary cell wall consists of an outer layer closest to the primary wall (S_1), a middle thick layer (S_2), and the innermost layer adjacent to the lumen (S_3) (Core et al., 1976; Brandstrom, 2001). The S_2 layer tends to dominate the physical and chemical properties of the cell wall (Donaldson & Burdon, 1995; Donaldson & Frankland, 2004). According to Cave and Walker (1994), MFA is the principal predictor of timber quality capable of affecting large changes in stiffness of wood, whereas density is an auxiliary variable. MOE is another important mechanical property of wood and can be influenced by both MFA (Cave & Walker, 1994; Hirakawa et al., 1998; Yang & Evans, 2003) and density (Panshin & de Zeeuw, 1980; Cown et al., 1999). MFA and density jointly accounted for 92% of MOE variations (Evans & Ilic, 2001; Yang & Evans, 2003). As all three of these properties are relevant for grading the quality of wood, they were all examined in this study.

Materials and Methods

Plant material

Thirteen half-disks cut from 60 mm oven-dried disks of radiata pine were used for the study. Each disk represented a separate tree approximately 15 years old cut about 5 m above the stump. The disks were sawn from logs of trees grown on different North Island sites in New Zealand. These trees were selected as representative samples of varying degrees of checking susceptibility by a commercial laboratory carrying out routine resource screening for a large forestry company. The disks that showed no checks in the growth rings were categorised as **non-checked**. The disks with checks were categorised as either **severe** or **moderate**, depending on both the number of checks seen in each growth ring and the number of growth rings that showed checks. Severely checked disks (Figure 1A) displayed more growth rings with checks and more checks in each growth ring compared to moderately checked disks (Figure 1B). The number of disks examined in each sample group were: severe (3), moderate (6) and non-checked (4).

Samples of wood were excised from oven-dried disks of radiata pine and assessed, utilising SilviScan-2, XRD and the modified Fullam micro-test stage. Samples were prepared from the same growth ring (growth ring 7), in order to minimise the variations between the growth rings.

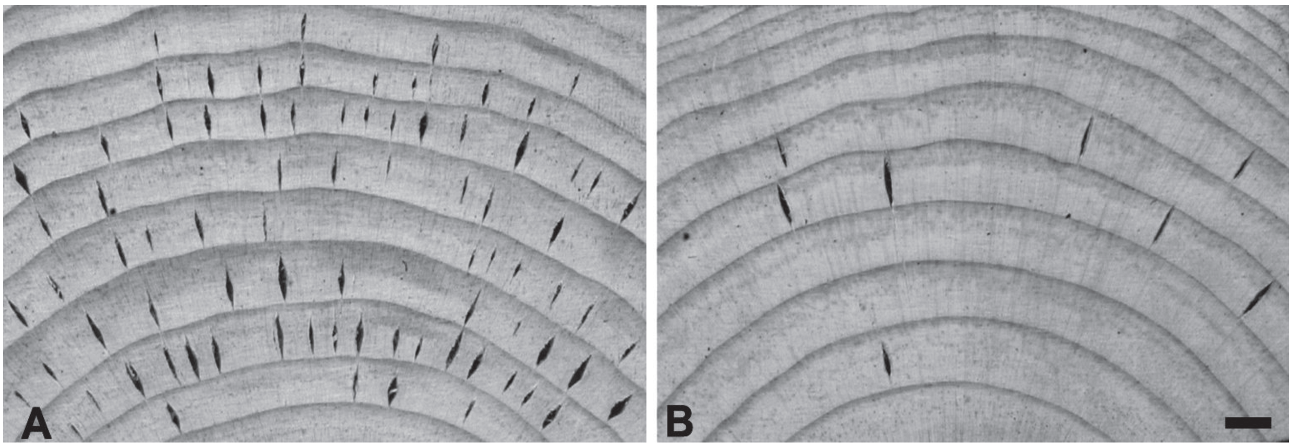

FIGURE 1: (A) Severely checked disk displaying a large of number of growth rings with checks. More checks were seen in each new growth ring affected;
(B) Moderately checked disk with few growth rings affected;
Scale bar = 1mm.

 ISSN 0048 - 0134

SilviScan-2 analysis of density, MFA and MOE

The samples for the SilviScan-2 measurements were sawn from the original disks and data collected for density, MFA and MOE. Observations were made from two samples taken from two different locations of the growth ring 7 of each disk. Tracheids of the growth ring that were in the region of checks were not considered for analyses of SilviScan-2 data. Observations from sample 1 were not taken into consideration for analyses for SilviScan-2 data due to the presence of the false growth ring observed in the ring 7 of the sample disk which could have influenced the measurements.

The method used was based on that described by Evans (1998). Briefly, the pieces of wood were cut into radial strips of dimensions 2 mm tangentially by 7 mm longitudinally for image analysis and microdensitometry. All the measurements were taken in a conditioned atmosphere maintained at 40% RH and 20 °C. The samples were re-conditioned from zero moisture content, having been dried with ethanol and then extracted with acetone. Samples were examined with the x-ray beam in the tangential direction. The growth ring orientation was measured by automated image analysis and the information used by the control software to maintain the growth rings parallel to the beam. The sample or the growth rings can be held at nominated fixed angle to the x-ray beam.

Image analysis and physical strength requirements dictated a sample thickness (tangential direction) between 1 mm and 2 mm. The absorption contrast for microdensitometry was done with copper Kα, on an average for wood samples approximately 2 mm thick. The analysis rate depends on the chosen spatial resolution, which was limited to the 0.2 mm diameter for the x-ray beam. Radial profiles with a 0.2 mm step size were obtained at the rate of 30 mm/h. To avoid problems associated with variation in fibre axis orientation, the integration span was usually limited to 10 mm, on the assumption that fibre orientation was constant within the chosen span.

A copper rotating-anode in point-focus mode was used in conjunction with a nickel filter and a capillary focussing system to produce a beam cross-section of diameter *ca* 0.2 mm at the sample. The diffraction patterns recorded with a charge coupled device (CCD) area detector. The strong (002) equatorial reflection was used for MFA analysis. Useful images of the equatorial reflections obtained in as little as 10 s, although 30 s was commonly used to improve the signal to noise ratio.

Modified Fullam micro-test stage analysis of MOE

The method used was based on that described by Butterfield & Pal (1998). Briefly, the top and bottom transverse cut surfaces of wood were smoothened using a sledge microtome (MSE microtome) to form a wood block of 4 mm. These blocks were then cut further into 1 mm wide strips, and then again at right angles into 1 mm pieces using a specially designed engineers press with rotating stage (Olympus, Japan model no. 219722). All the sticks were then examined under a microscope and those with any cutting defects were rejected. A minimum of 20 sticks per sample group were selected for testing. The sticks were first weighed then measured for length, height and width at 40% humidity. They were then compression load tested in a modified micro-test stage (E F Fullam Inc) at 40% humidity environment. The load cell and the linear transducer (for measuring linear compression) were interfaced to a personal computer (PC) fitted with data acquisition board and running locally written software. The PC automatically records maximum crushing strength and stiffness. The data for the stiffness were taken from the central part of the load/displacement graph to avoid any distortion caused by end collapse of cut cells during initial compression loading.

X-ray diffraction analysis of MFA

Wood pieces were cut from the original disks with a chisel and hammer. Smaller sticks 4 mm x 1 mm x 1 mm were further cut from the wood pieces using a specially designed engineers press with rotating stage (Olympus, Japan model no. 219722). The sticks (five from each sample group) were then used to measure MFA using the XRD method of Cave and Robinson (1998). Angular distribution of the longitudinal microfibril axes of cellulose in the cell wall is represented by the distribution of the crystal plane poles. These poles are parallel to the microfibril axis and XRD is capable of measuring these pole distributions. Modern computer-controlled diffractometers using area detectors directly acquire digital data from the area displayed then automatically produce pole figures. The area detector from the wood displays three sets of broad arcs on concentric circles. Two of the crystal planes are so close together that they almost merge, and the third plane is perched on the outer shoulder of the other two. All are planes that include the longitudinal microfibril axis, and they are much the same along their respective diffraction circles. It means that almost every microfibril orientation contributes to the diffraction arc and so the profile contains a measure of MFA orientation.

Statistical Analyses

All statistical analyses were carried out using S Plus statistical package (S-Plus 8, TIBCO Software Inc, Palo Alto, California). Main effects between the mechanical property and the type of wood were examined using analysis of variance (nested ANOVA). Tukey's least significant difference test was used where applicable, to distinguish among individual mean values with a confidence level of $p \leq 0.05$.

 ISSN 0048 - 0134

TABLE 1: Density and MFA data for severe, moderate and non-checked wood obtained using SilviScan-2 at 40% RH and 20 °C. Data are ± standard deviation of the two samples taken from two different locations of growth ring 7 of each of the severe, moderate and non-checked sample groups. Samples size represents the number of tracheids measured in growth ring number 7 of each of the samples.

Type of wood sample group	Average density (kg/m³)	Average MFA (degrees)	Sub-sample Size
Severely checked	370 ± 30.9	20.2 ± 3.9	490
Moderately checked	357 ± 36.4	18.4 ± 4.9	1369
Non-checked	397 ± 25.9	17.5 ± 4.1	860

Results and Discussion

The samples analysed in this study were prepared from the earlywood region of the wood, as checking occurs usually in this region and is rarely seen in the latewood region.

Density

Surprisingly, the lowest density was recorded for moderately checked wood rather than severely checked wood. Overall, however, the SilviScan-2 measurements showed that checked wood had a lower density compared to non-checked wood (Table 1) and that this difference was significant ($p < 0.05$). Some previous studies have shown similar results (Ball et al., 2005; Simpson et al., 2002; Ilic, 1999a, b; Chafe, 1994) and suggest that low density can increase checking (Ball et al., 2005; Simpson et al., 2002; Ilic, 1999a).

Density is an important property of wood. It is quality trait that is a function of xylem anatomy and can be influenced by cell wall thickness, cell diameter, and the chemical content of wood (Cave & Walker, 1994). Higher wood density and thicker cell walls tend to lower the tendency of the wood to check (Simpson et al., 2002). Thick cell walls ensure that only small compressive stresses occur in the cell walls. Such cells can resist collapse and prevent development of checks. Collapse of the wood fibres can lead to checking (Simpson et al., 2002). Earlier studies (Booker, 1995; Putoczki et al., 2007; Nair et al., 2009) have reported associations between checked wood and collapsed fibres. In the current study, SilviScan-2 samples observed using a dissecting microscope showed the presence of collapsed cells in checked wood, whereas collapsed cells were rarely seen in non-checked wood (Figure 2). This microscopy data of the samples indicated that lower density wood was more inclined to collapse and check compared to high density wood.

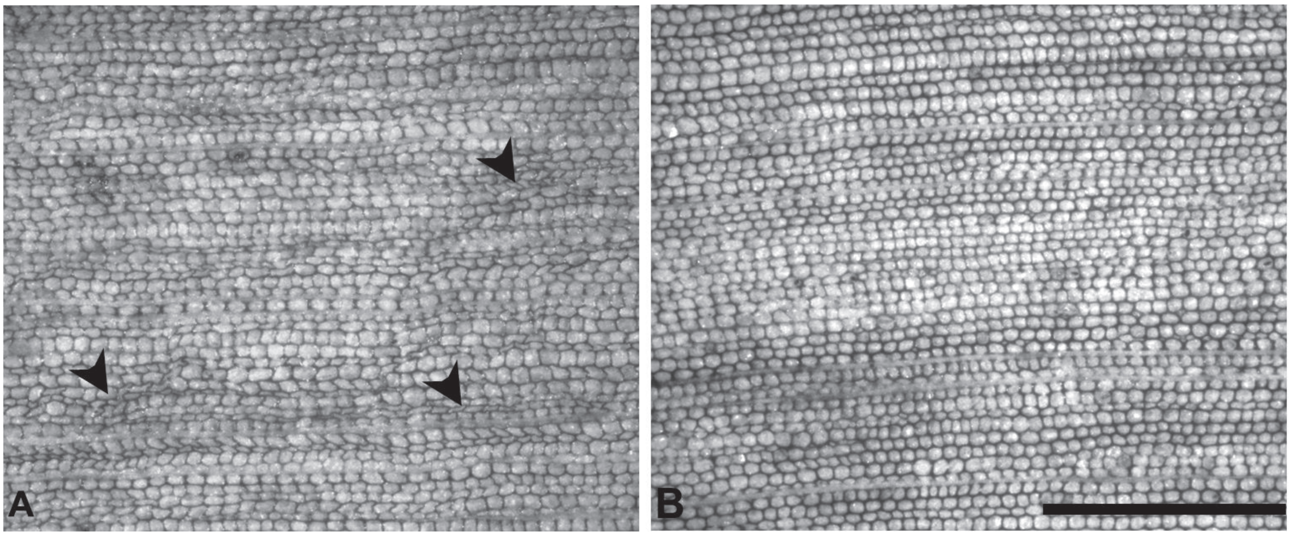

FIGURE 2: (A) Severely checked wood sample. The arrowheads indicate the presence of collapsed cell files;
(B) Non-checked sample;
Scale bar = 5cm.

 ISSN 0048 - 0134

TABLE 2: MFA data from XRD analysis. Data are ± standard deviation of the samples taken from each of the severe, moderate and non-checked sample groups.

Type of wood sample group	Average MFA (degrees)	Sub-sample size
Severely checked	41.2 ± 2.6	5
Moderately checked	34.0 ± 2.0	5
Non-checked	27.8 ± 4.5	5

One has to be careful in making predictions, whether a particular wood is prone to checking or not, based on density alone, however. High density displayed by a wood could also possibly be due to the presence of compression wood or a false growth ring.

MFA

Differences in MFA data were not statistically significant in the present study. This finding is consistent with a previous study on radiata pine (Ball et al., 2005). However, average MFA values from either XRD or SilviScan-2 equipment showed the same trend - that checked wood had higher MFA compared to non-checked wood. XRD data (Table 2) showed higher values for MFA measurements compared to the SilviScan-2 data (Table 1). Three of the reasons for such a variable result could be: (i) the presence of checks in some of the samples that were used for SilviScan-2 measurements that might have affected the data; (ii) the samples analysed by XRD were not from exactly the same point of growth ring 7 as the SilviScan-2 samples; and (iii) the plane along which the measurements are taken is tangential with SilviScan-2 and radial with XRD.

Researchers have shown MFA to be linked to dimensional changes in wood that occur changes in moisture content (Harris & Meylan, 1965; Meylan, 1968). During drying, moisture is lost from the wood surface more easily than compared from the centre which leads to the development of a moisture content gradient that, in turn, results in uneven shrinkage (Pang, 2002). As they dry, the outer layers shrink before the inner core and, when the stress between the layers exceeds the yield stress of the wood, checking takes place (McCurdy & Keey, 1999; Huang et al., 2003; Barnett & Bonham, 2004). An increase in MFA can increase longitudinal shrinkage (Huang et al., 2003; Barnett & Bonham 2004, Lundgren 2004) that could affect checking. The minimum longitudinal shrinkage in radiata pine occurs at an MFA of 25° (Wang et al., 2001). XRD data in the present study showed that the non-checked wood had an average MFA of 27.8° compared with an average MFA of 34° for moderately checked wood and 41.2° for severely checked wood, Table 2. It is possible that wood with a high MFA angle could undergo greater differential shrinkage stresses during drying and develop checks.

MFA can also influence the fracturing properties of wood. Small MFAs favour trans-wall fracture as opposed to intra-wall fracturing under transverse shear (Donaldson, 1998). In a previous study, Putoczki et al. (2007) found that the checks usually occurred along the compound middle lamella and S_1 cell wall layer. The predominant form of fracturing observed was intra-wall fracturing at the site of the check. It seems that the high MFAs observed in checked wood could make the wood vulnerable to fracture between the walls and lead to checking.

However, as with density, we cannot use MFA alone to predict shrinkage in wood. Factors other than MFA can also influence shrinkage, such as the extent of accumulation of internal growth stresses in the tree and their release when a tree is cut (Barnett & Bonham, 2004).

MOE

The MOE data obtained using modified Fullam micro-test stage were lower than the corresponding SilviScan-2 measurements. One of the possible reasons for this discrepancy could be that the samples used in the modified Fullam micro-test stage were cut into sticks of very small dimension and presence of broken tracheids exposed along the edges of the samples could affect the measurements by the machine.

There was much variability observed in the MOE data using either SilviScan-2 or modified Fullam micro-test stage. However, when mean values of each wood sample were calculated using the Fullam micro-test stage data, it was seen that most checked wood samples had lower MOEs compared with non-checked wood (Figure 3).

Neither the SilviScan-2 nor the Fullam micro-test stage MOE data showed any significant relationship

 ISSN 0048 - 0134

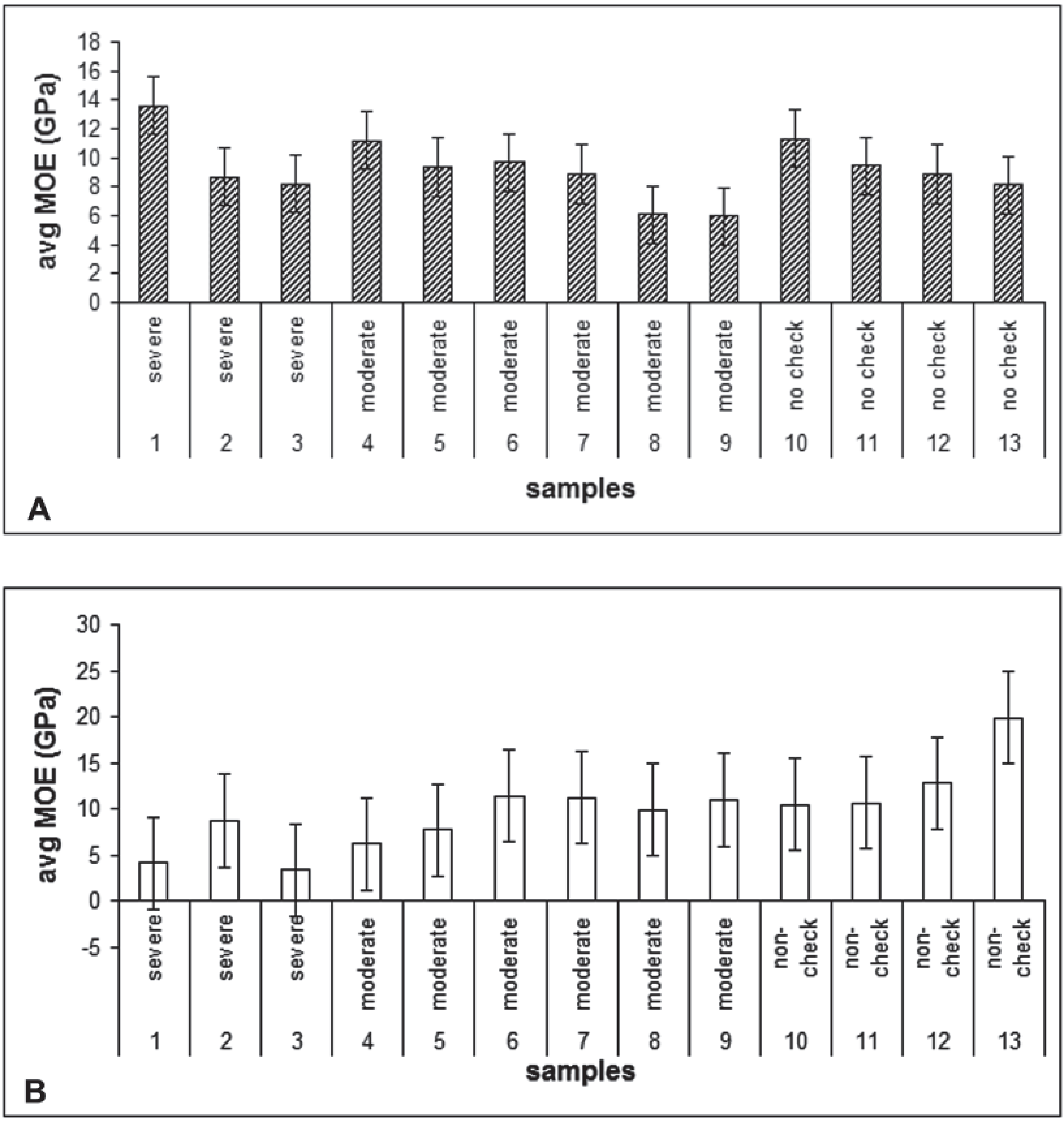

FIGURE 3: MOE data analysis of the checked and non-checked wood samples.
(A) SilviScan-2 data;
(B) modified Fullam micro-test stage data.

 ISSN 0048 - 0134

(p <0.05) between MOE and checking. This observation is similar to the one made in a previous radiata pine study (Ball et al., 2005). A combination of high MFA and low density can result in low strength wood (Lundgren, 2004). The lower the strength of wood cells the more readily they will collapse under the drying stresses that could lead to formation of checks in the wood.

Conclusion

Compared to earlier work (Ball et al*.,* 2005), the number of samples analysed in the current study was small and none of mechanical property data obtained showed statistically significant differences between checked and non-check wood. However, the trends seen in the study are similar to those obtained in the previous work (Ball et al*.,* 2005). This study confirms that checking in wood is complex. A more reliable indicator for checking in wood might be a combination of the mechanical properties of wood with other wood properties, such as tracheid dimensions and chemical content.

Acknowledgements

We would like to thank Wood Quality Initiative Limited, New Zealand, the Tertiary Education Commission, New Zealand and FRST, New Zealand (Grant No. UOCX0801) for financial support for this work. Graeme Young (Tenon Limited, New Zealand) thanks for providing radiata pine disks for the study. Our thanks also go to Robert Evans (CSIRO, Australia), who kindly collected SilviScan-2 measurements and Dr. Jan Wikaira (Department of Chemistry, University of Canterbury), for helping out with XRD. We would also like to thank Manfred Ingerfeld for help with the manuscript.

References

Ball, R. D., McConchie, M. S., & Cown, D. (2005). Evidence for associations between SilviScan measured wood properties and intraring checking in a study of twenty-nine 6-year old *Pinus radiata*. *Canadian Journal of Forest Research*, *35*(5), 1156-1172.

Barnett, J. R., & Bonham, V. A. (2004). Cellulose MFA in the cell wall of wood fibres. *Biological Review*, *79,* 461-472.

Booker, R. E. (1995). Internal checking and collapse investigations at NZ FRI. In K. R. Klitscher, D. J. Cown & L. A. Donaldson (Eds.), *Wood quality workshop 95, Rotorua, New Zealand, November, 1995*. *FRI Bulletin No. 201* (pp. 26-28). Rotorua, NZ: New Zealand Forest Research Institute.

Brandstrom, J. (2001). Micro and ultrastructural aspects of Norway spruce tracheids. *IAWA Journal*, *22*, 333-353.

Butterfield, B. G., & Pal, V. (1998). Relating MFA to wood quality in clonal seedlings of radiata pine. In B. G. Butterfield (Ed.), *Proceedings of IAWA/ IUFRO International workshop on the significance of MFA to wood quality*, *Westport, New Zealand. Christchurch* (pp. 337-347). New Zealand: University of Canterbury.

Cave, I. D., & Robinson, W. T. (1998). Measuring microfibril angle distribution in the cell walls by means of X- ray diffraction. In B. G. Butterfield (Ed.), *Proceedings of IAWA/ IUFRO International workshop on the significance of MFA to wood quality*, *Westport, New Zealand. Christchurch* (pp. 94-107). Christchurch, NZ: University of Canterbury.

Cave, I. D., & Walker, J. C. F. (1994). Stiffness of wood in fast- grown plantation softwoods: the influence of MFA. *Forest Products Journal*, *44*(5), 43-48.

Chafe, S. C. (1994). Preheating green boards of mountain ash (*Eucalyptus regnans* F. Muell.). II. Relationship amongst properties. *Holzforschung*, *48*, 163-167.

Chafe, S. C. (1995). Preheating and continuous and intermittent drying in boards of *Eucalyptus regnans* F. Muell. *Holzforschung*, *49*, 227-233.

Cown, D. J., Hebert, J,. & Ball, R. (1999). Modelling *Pinus radiata* lumber characteristics. Part 1: Mechanical properties of small clears. *New Zealand Journal of Forestry Science*, *29*(2), 203-213.

Donaldson, L. A., & Burdon, R. D. (1995). Clonal variation and repeatability of microfibril angle in *Pinus radiata*. *New Zealand Journal of Forestry Science, 25*(2), 164-174.

Donaldson, L. A. (1998). Ultrastructure of transwall frature surfaces in Radiata pine wood using transmission electron microscopy and digital image processing. *Holzforschung*. *51*, 303-308.

Donaldson, L. A., & Frankland, A. (2004). Ultrastructure of iodine treated wood. *Holzforschung*, *58,* 219-225.

Evans, R. (1998). Rapid scanning of microfibril angle in increment cores by X-ray diffractometry. In B. G. Butterfield (Ed.), *Proceedings of IAWA/ IUFRO International workshop on the significance of MFA to wood quality*, *Westport, New Zealand. Christchurch* (pp. 116-139). Christchurch, NZ: University of Canterbury.

Evans, R., & Ilic, J. (2001). Rapid prediction of wood stiffness from MFA and density. *Forest Products Journal*, *51*(3), 53-57.

Harris, J. M., & Meylan, B. A. (1965). The influence of MFA on longitudinal and tangential shrinkage in *Pinus radiata*. *Holzforschung*, *195,* 144-153.

Hirakawa, Y., Yamashita, K., Fujisawa, Y., Nakada, R., & Kijidani, Y. (1998). The effects of S2 microfibril angle and density on modulus of elasticity in sugi tree logs. In B. G. Butterfield (Ed.), *Proceedings of IAWA/ IUFRO International workshop on the significance of MFA to wood quality*, *Westport, New Zealand. Christchurch* (pp. 312-322). Christchurch, NZ: University of Canterbury.

Huang, C. L., Linstrom, H., Nakada, R., & Ralston, J. (2003). Cell wall structure and wood properties determined by acoustics - a selective review. *Holz als Roh- und Werkstoff*, *60,* 165- 174.

Ilic, J. (1999a). Shrinkage-related degrade and its association with same physical properties in *Eucalyptus regnans F. Muell*. *Wood Science and Technology*, *33,* 425-437.

Ilic, J. (1999 b). Influence of prefreezing on shrinkage-related degrade in *Eucalyptus regnans F. Muell*. *Holz als Roh und Werkstoff*, *57,* 241-245.

Jayawickrama, K. J. S., Shelbourne, C. J. A., & Carson, M. J. (1997). New Zealand long internode breed of *Pinus radiata*. *New Zealand. Journal of Forestry Science, 27,* 126-141.

Lundgren, C. (2004). MFA and density patterns of fertilized and irrigated Norway spruce. *Silva Fennica*. *38*(1), 107-117.

McCurdy, M., & Keey, R. B. (1999). Moisture saturation profiles in *Pinus radiata* during high- temperature drying. *IPENZ Transactions, 26,* 29-35.

Meylan, B. A. (1968). Cause of high longitudinal shrinkage in wood. *Forest Product Journal, 18*(4), 75-78.

Ministry of Agriculture and Forestry (MAF). (2005). Forestry Statistics. Retrieved on 11th October 2006. http://www.maf.govt.nz/statistics/primaryindustries/forestry/forestry-facts-and-figures-06.pdf

Nair, H., Jackson, S., & Butterfield, B.G. (2009). Are rays and resin canals causal sites for intra-ring checking in the wood of *Pinus radiata*. *IAWA, 30,* 189-198.

Pang, S. (2002). Predicting anisotropic shrinkage of softwood. Part 1. Theories. *Wood Science and Technology, 36,* 75-91.

Panshin, A. J., & de Zeeuw, C. (1980). *Textbook of Wood Technology* (p 112). 3rd ed. New York: McGraw-Hill.

Putoczki, T., Nair, H., Butterfield, B. G., & Jackson, S. (2007). Intra-ring checking in *Pinus radiata* D. Don: the occurrence of cell wall fracture, cell collapse, and lignin distribution. *Trees, 21,* 221-229.

Simpson, I., Booker, R., & Haslett, T. (2002). Within-ring internal checking. *Wood Processing Newsletter, 32,* 1-3.

Wang, H. H., Drummond, J. G., Reath, S. M., Hunt, K., & Watson, P. A. (2001). An improved fibril angle measurement method for wood fibres. *Wood Science and Technology, 34,* 493-503.

Withers, T. M., & Keena, M. A. (2001). *Lymantia monacha* (nun moth) and *L dispar* (gypsy moth) survival and development on improved *Pinus radiata*. *New Zealand Journal of Forestry Science, 31,* 66-77.

Yang, J. L., & Evans, R. (2003). Prediction of MOE of eucalyptus wood from microfibril angle and density. *Holz als Roh- und Werkstoff*, 61, 449-452.