An improved fibril angle measurement method for wood fibres

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Abstract A rapid, reliable technique for the observation and measurement of the fibril angle in wood cell walls has been developed. Sonication in the presence of solutions of certain cobalt and copper salts (5%, wt/vol) was found to be most effective in facilitating fibril angle visualisation. Latewood fibre fibril angle, which previously had been difficult to measure, was also visible, though less frequently. This method has been successfully applied to a number of softwood species including coastal Douglas-fir whose prominent spiral thickenings make it difficult to determine the fibril angle by other methods. The method was also used to determine the fibril angle of some hardwood species as well as a non-woody material, flax straw. It can also be used to determine the microfibril angle of pulp fibres although this procedure is less convenient than with wood sections.

Introduction
The orientation of microfibrils in wood fibres is closely related to the physical and mechanical properties of both solid wood and single wood fibres. It has been reported (Wimmer 1992) that fibril angle and tangential diameter of latewood tracheids are important determinants of clear wood strength. The fibril angle affects the basic strength and behaviour of wood fibres (Prud’homme and Noah 1975) which in turn influence the final characteristics of paper products (Spark et al. 1958; Guha 1961; Watson and Dadswell 1964; Cowdrey and Preston 1966; Tamolang et al. 1967; Page et al. 1972). The fibril angle shows a strong positive correlation with stretch and rupture energy of paper (Watson and Dadswell 1964; Horn 1972). A curvilinear relationship was observed (Harris and Meylan 1965) between fibril angle and shrinkage, with minimum longitudinal shrinkage occurring at a fibril angle of about 25° in Pinus radiata. A small change in fibril

Received: 24 June 1999

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The authors would like to acknowledge the late George Williams for his helpful discussions on the effect of fibril angle on wood and pulp strength and for use of the SEM photomicrograph in Fig. 1 and Dr. R. S. Seth for his critical review of the manuscript.
angle, for angles less than about 15°, results in significant changes in the axial fibre modulus (Mark and Gillis 1973).

Microfibrils in the secondary wall of wood fibres are closely packed and mostly parallel. In the middle secondary wall (S₂) layer, the fibrils spiral around the axis at an angle that is not uniform throughout a given length of a single fibre and which varies from fibre to fibre within a tree (Panshin and Dezeeuw 1980). The S₂ layer typically contains 30–40 lamellae, but in certain cases over 150 lamellae (Sjöström 1993) with a Z-helix averaging 10° to 30° to the longitudinal axis have been observed (Wangaard 1979). It has been suggested that the variation of this angle might be genetically controlled (Hirakawa and Fujisawa 1995; Boyd 1985). The S₂ layer fibril angle has long been of interest to wood anatomists, because the S₂ layer forms the main portion (80–90%) of the cell wall (Pleasants and Parker 1995) and its properties will therefore exert the most influence over fibre behaviour. The fibril angle is known to be greater in juvenile wood (37° to 55°) than in mature wood (7° to 20°) for coniferous species (Haygreen and Bowyer 1982). Thus, not only is fibril angle an important parameter in determining wood and fibre quality but may also be used to define the zone of wood juvenility. Fast grown species, with a large proportion of juvenile wood, are generally associated with inferior wood strength and excessive longitudinal shrinkage (Ying et al. 1994).

There are three basic methods for measuring microfibril angle in wood cell walls: X-ray diffraction (Cave 1966; Boyd 1977; Stuart and Evans 1994), polarized light microscopy (Preston 1934; Manwiller 1966; Page 1969; Leney 1981) and direct or indirect observation (Bailey and Vestal 1937; Cockrell 1974; Senft and Bendtson 1985). X-ray diffraction cannot be applied to pulp fibres because of the difficulty in properly aligning the sample, and because it can only provide accurate results on cylindrical fibres. Accurate estimation of the fibril angle by X-ray diffraction involves interpretation of many variables (Stuart and Evans 1995). Polarized light methods are generally tedious, time consuming or cannot be applied to industrial pulps. Both Page and Leney’s techniques are subject to error from the S₁ and S₃ layers of the secondary wall (El-Hosseiny and Page 1973; Page and El-Hosseiny 1974). A fracture SEM (scanning electron microscopy) method has also been developed (Armstrong et al. 1977) but tends to selectively exclude high angle fibres. In addition SEM studies on fibril angle are limited since the number of fibres with visible S₂ fibril orientations is very small and they are hard to find (Fig. 1). Most recently a confocal microscopy technique which detects the intensity of polarized laser light reflected from the fibre has been reported (Batchelor et al. 1997).

Though an important fibre property, the fibril angle has been largely neglected because of the difficulty and excessive amount of time involved in its measurement. Hence, the true role that fibril angle plays in macro-wood properties requires further in-depth investigation. It would be advantageous to be able to determine fibril angles directly on whole wood samples with a minimum of effort. If the fibril angle determined on the whole wood sample could be positively related to any of the important pulp properties, species evaluation for pulping purposes could be quicker and eliminate the more expensive pulping studies now required.

It is desirable to have a direct, accurate and cost effective method for fibril angle determination. A common procedure for fibril angle measurement using light microscopy observes and measures the checks and splits along the direction of the microfibrils caused by rapid shrinkage in the S₂ layer, or measures the angle of elongated pit apertures. These methods have serious disadvantages because cracks and pit apertures cannot always be found in sufficient quantity