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CRC for Sheep Industry Innovation

Subprogram 2.3.1 Fabric Handle

Milestone / Task	Description	Due Date
R4.3.2.3	Report on further studies to determine the variation due to genetic and environmental factors affecting fibre properties such as fibre moduli and scale height.	30/5/09

Summary

This report is in two parts. The first part examines the variation in Young's modulus of wools that have extreme values of resistance to compression (RtC) for their mean fibre diameter and fibre curvature. The results indicate that Young's modulus varies considerably both between fibres from the same sheep (≈2000 to 9000 MPa) and also from sheep to sheep (≈5000 to 6500 MPa). The overall trend was an increase in modulus for wools with lower RtC. This is opposite of what one would expect, viz. less stiff fibres would be expected to be softer and have lower RtC. Further work is needed in this area: specifically isolating the role of diameter, diameter uniformity, ellipticity and fibre surface properties. No correlation was found between the decrimping stress and RtC. Young's modulus was negatively correlated with fibre diameter at the break and positively correlated with stress at 15% strain and the breaking stress.

The second part of this report examines methods of measuring ellipticity of fibres. Problems with fibre alignment have been identified with Sifan 3. A laser diffraction method for

determining fibre diameter coupled with a fibre rotator can be used to measure the diameter at several orientations enabling fibre ellipticity to be estimated.

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PART 1

Evaluation of the Young's modulus of wools with different resistance to compression properties

1 INTRODUCTION

Resistance to compression (RtC) is the force per unit area (in kilopascals, kPa) required to compress a fixed mass of wool to a fixed volume. The testing methodology is described in the Australian Standard AS 3535 – 2004 'Wool – Method for the measurement of resistance to compression. RtC was regarded as a characteristic of only secondary importance in wool textile processing (Whiteley et al., 1978, Teasdale, 1986), however it is now a small but significant factor in the prediction of the processing performance of a lot of wool and of the softness properties of the resulting fabric (Australian Standard AS-3535, 2004).

RtC has high heritability and is closely correlated to follicle curvature at the genetic level (Watson et al., 1977, Brown, 2005, Madeley et al., 1998b). Studies have shown that RtC is correlated to staple crimp frequency (Madeley et al., 1998b, Stevens, 1994, Madeley and Postle, 1994) and single fibre crimp frequency (Chaudri and Whiteley, 1968, Slinger, 1965, Whiteley and Balasubramaniam, 1965). Staple crimp frequency is also related to the fibre curvature (Swan, 1994) (Brown, 2005) which is defined as the arc measured in degrees across a one millimetre length of wool (Swan, 1993). Brown (2005) and Madeley (1995) observed that staple crimp frequency and curvature were negatively but not significantly related with fibre diameter. Other researchers have demonstrated that RtC increases with both increasing crimp frequency and fibre diameter, and the product of the two variables is correlated with RtC (Chaudri and Whiteley, 1968, Slinger, 1965, Madeley and Postle, 1994, Madeley, 1994a, Teasdale, 1986, Swan, 1993, Shah and Whiteley, 1971). Swan (1994) developed a prediction for RtC based on fibre diameter and the radius of fibre curvature that explained 92% of the variation in RtC. Brown (2005) could only explain 42% of the variation of RtC in his wools using Swan's prediction formula. Madeley (Madeley and Postle, 1994, Madeley, 1994a) found a weak positive relationship between RtC and fibre diameter (R=0.32) and attributed the greater spreading of data points for diameters greater than 18μm to variation in crimp, and for non-crimp cashmere and mutant Lustre wool (crimpless wool), RtC was independent of fibre diameter. Liu (2004) found a negative relationship between RtC and fibre diameter and suggested that RtC is a poor indicator of fibre softness, particularly for wool fibres of varying diameter. The increase in resistance to compression with increasing crimp frequency but decreasing fibre diameter has also been reported (Whiteley et al., 1986) (Madeley et al., 1998a).

Recent studies have confirmed that wools with low RtC and low crimp frequency have superior topmaking and spinning performance (Lamb et al., 1996, Lamb et al., 2000, Whiteley et al., 1986). This contrasts earlier work by Menkart and Detenbeck (1957) that reported that worsted processing performance is improved with increasing fibre crimp frequency. RtC has been linked to fibre softness (Madeley et al., 1998a, Ali et al., 1971, Stevens, 1994, Whiteley et al., 1986) and it has been shown that wools of low RtC have a softer handle than wools of the same average fibre diameter that exhibit a high RtC. A wool with high RtC is harder to squeeze and has a lower felting potential than a wool with low RtC and this makes RtC values useful in assessing the suitability of wool for specific end uses. For example, wools used for carpets and upholstery fabrics need to have a high RtC, whilst the preference for knitted apparel wools would be low RtC.

Madeley (1998b) plotted some of the bending moduli and crimp frequency data collected by Shah and Whiteley (1971) and obtained a positive relationship between the two parameters with an $R^2 = 0.56$. From this Madeley concluded that bending rigidity increased with fibre crimp independent of fibre diameter, and suggested that an increase in crimp may be accompanied by increased stiffness in fibres of constant diameter. Madeley did not conduct any experiments to prove his theory and had he have used all of Shah and Whiteley's bending data and crimp frequency rather than only selected points an $R^2 = 0.008$ is obtained. Shah and Whiteley (1971) found the bending modulus was not significantly related to handle scores. They found considerable variation in the bending modulus and suggested that the variation in modulus may be due to variability of fibre cross-sectional area along the length of the fibre segments. Similarly, Roberts (1956) considered the dependence of fibre softness on Young's modulus, using the rationale that Young's modulus is related to bending and torsional moduli involved in handle appraisal and that Young's modulus is the easiest to measure. Roberts studied 65 samples of wool and measured only 8 fibres for each sample. The average coefficient of variation of Young's modulus in his samples was 13.5% and his study failed to show a significant relationship between Young's modulus and handle.

In a survey of the Australian Merino flock, it is apparent that RtC and fibre diameter are not significantly related, however wool fibres of similar diameter and curvature do exhibit large variations in RtC behaviour (Brown, 2005). The variation in RtC suggests that there may be a contribution from some other source such as fibre shape, surface or other material or substance specific property of the fibre as suggested by Madeley (1994b). The purpose of this work is to revisit the work of Roberts (1956) and using the same rationale establish if Young's Modulus, a material specific property, contributes to RtC of wool when diameter and curvature are constant.

The slope of the pre-yield or Hookean region of a stress-strain curve is called the Young's modulus, *E*. It is also known as initial modulus or modulus of elasticity because in this region complete recovery from deformation does occur. The Young's modulus is an intrinsic property and provides information on the molecular arrangement as well as the chemical structure of the fibre. The Young's modulus of wool has contributions from the oriented α-helices and the matrix, coupled in parallel and increased molecular orientation along the fibre axis increases the Young's modulus. As relative humidity increases the matrix becomes more highly compliant, and in the wet state, only the α-helices contribute to the Young's modulus (Rao and Gupta, 1991, Warner, 1995). A number of workers have shown that an inverse relationship exists between Young's modulus and crimp, and Young's modulus decreases with increasing levels of crimp (Evans, 1954, Dusenbury and Wakelin, 1958, Brand and Backer, 1962, Menkart and Detenbeck, 1957, Bendit, 1980, Dillon, 1952, Barach and Rainard, 1950). Evans suggested that the stress across the diameter of a crimped fibre is not uniform during extension as shown in Figure 1, with the inside of the crimp (point B) experiencing greater tension than the outside of the crimp (point A) were the material is probably in compression when the initial small tension is applied.

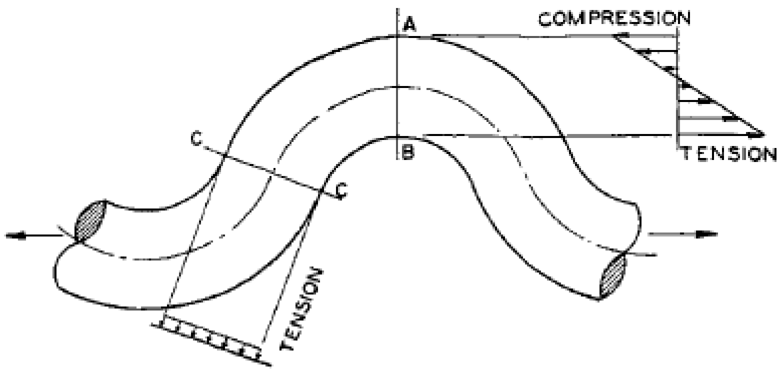

Figure 1 Stress in a crimped wool fibre under small tension (Evans, 1954)

This paper reports on the relationship between Young's modulus and RtC for wools of constant diameter and curvature. There is also a suggestion that Young's modulus for wool is related to other tensile properties such as stress at break and stress at 15% strain (Huson and Turner, 2001, Thompson, 1998). This paper also reports on the relationship between these other tensile parameters and Young's modulus.

2 EXPERIMENTAL PROCEDURE

A brief summary of the experiment details is provided as full details of the fibre selection, scouring, conditioning, handling, Araldite mounting and techniques used for the measurement of tensile properties, fibre diameter and the calculation of Young's modulus are provided in Task report 4.3.1.2. The wools used in this study were selected from the greasy, mid-side keeper samples, retained from the Novel Merino Wool Quality Traits – Sheep CRC Project 1.2.6 - Improved sheep wool and meat production and were selected purely on their extremes of RtC at constant diameter and curvature. Greasy staples were cleaned by gentle scouring in Lissapol TN450 (ICI) detergent. After drying and conditioning the diameter of single fibres were measured using a vibroscope and each fibre was mounted on to individual plastic tabs with a gauge length of 20mm. Before anchoring the fibres with Araldite glue, the crimp frequency of each fibre was determined by counting the number of wave crests over the 20mm gauge length when the fibre was free of tension and when tensioned with the appropriate vibroscope weight (typically a 200mg weight, however occasionally the 100mg and 300mg weights were also used). After ageing for seven to ten days in a standard atmosphere, fibres were subjected to tensile testing in the standard atmosphere air at 20°C and 65 % relative humidity. The force-extension curves were recorded for a minimum of 50 fibres per sample and tests were carried out on an Instron Tensile Tester (model 4500) at a gauge length of 20mm and an extension rate of 5mm/min. Fractured ends were relaxed in water at 20°C for 30 minutes, then allowed to air dry and condition for at least 24 hours in a standard atmosphere of 20°C and 65 % relative humidity. An optical microscope and image analysis software was used to measure the diameter of the side view of the fractured ends. Cross-sectional area at the break was calculated for each fibre from the average diameter of the relaxed, fractured ends. The fibre cross-sectional area is used to normalise the slope and force data so that material specific properties of Young's modulus and intrinsic strength can be determined.

3 RESULTS AND DISCUSSION

The wools originated from client flocks of Merino Genetics Services located in Victoria, New South Wales and Queensland as shown in Figure 2. Wools from this library were selected for constant diameter and curvature (as measured by Laserscan), and extremes in resistance to compression. Details of these wools are given in Table 1. Selection was not based on geographic location (environment), however some information on the influence of geographic location may be derived from the results given the samples selected for this trial originated from various regions across eastern Australia.

Sample Code	Sheep Location & Rainfall Intensity Colour Code	Laserscan Diameter (μm)	Laserscan Curvature (°/mm)	RtC (kPa)
A	Location not provided	16.9	51	7.0
B	Location not provided	16.9	51	8.0
C	Victoria - grey	16.9	59	4.2
D	Location not provided	16.9	59	6.5
E	Victoria - grey	16.9	59	7.7
F	Victoria - grey	16.9	59	7.8
G	Victoria - grey	16.9	59	9.0
H	Location not provided	16.9	59	8.0
I	NSW - green	16	73	4.1
J	NSW - yellow	16.2	73	10.1
K	NSW - green	16	73	7.0
L	QLD - yellow	16	53	4.7
M	QLD - yellow	16	53	8.1

Table 1: Geographic location of sheep and fibre diameter, curvature and resistance to compression data for these wools

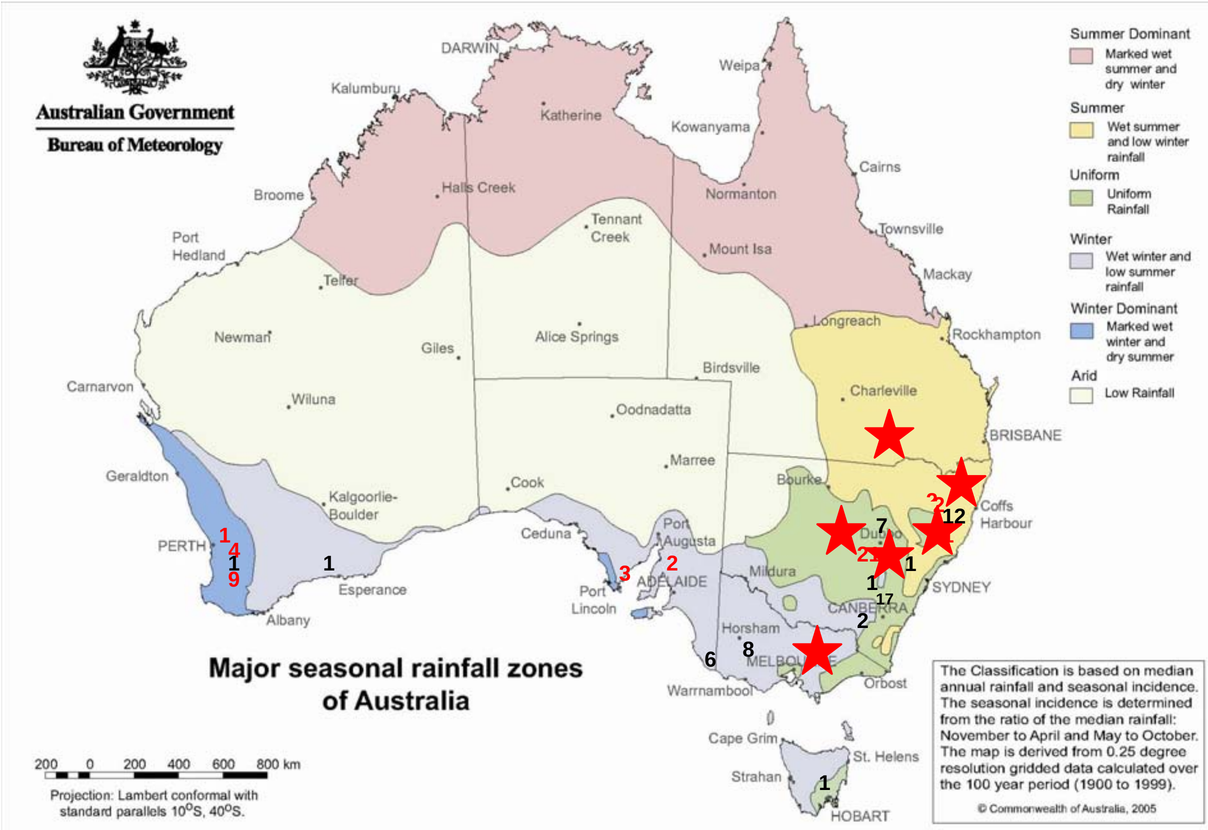

Figure 2: Geographic locations of wool samples used in this trial are marked (Map supplied by Dr Ken Geenty).

The stress-strain properties of 680 single wool fibres from 13 sheep with extremes in RtC behaviour have been measured. The average diameter of these fibres as measured at the point of break along with the average stress-strain properties are given in Table 2a and 2b.

<u>Measurement of diameter</u>

Across all the sheep, the average coefficient of variation in cross-sectional diameter (CV_D) at the point of break between fibres from the same sheep varied from 8% to 33%. The sheep "B" from an unknown location had the lowest average CV_D. Sheep "J,L and M" from the yellow rainfall regions in New South Wales and Queensland also had low, average CV_D. Sheep "C" from the "grey" rainfall region in Victoria and sheep "H" from an unknown location had high, average CV_D. The CV_D in the diameters measured by the vibroscope ranged from 7% to 18%. Again sheep "B" had very uniform fibre diameter and sheep "C and H" were the least uniform. A positive relationship exists between the vibroscope diameter measurement and the diameter measured at the break (Figure 3). Fibres have a tendency to break at their weak spot which is generally the thinnest spot along the length of the fibre (Kwak et al., 2007) and consequently the diameter of the broken fibres were on average 17% thinner than the original fibres (range 12% to 24%). For some coarse fibres above 25 micron, the diameter measured at the point of break was greater than the original diameter as measured on the vibroscope. Apart from these coarse fibres requiring a larger tensioning weight (300mg instead of 200mg) for vibroscope diameter measurement, the only other possible explanations for this observation is that fibre shape may be interfering with diameter measurements or alternately the larger diameters at the break may be due to a flaw on the fibre. Since accurate diameter measurements are required for normalising tensile data, it is recommended that diameters at the break are measured using the optical method rather than estimated from the relationship between initial vibroscope diameter and diameter at the break as this relationship does not appear to be linear.

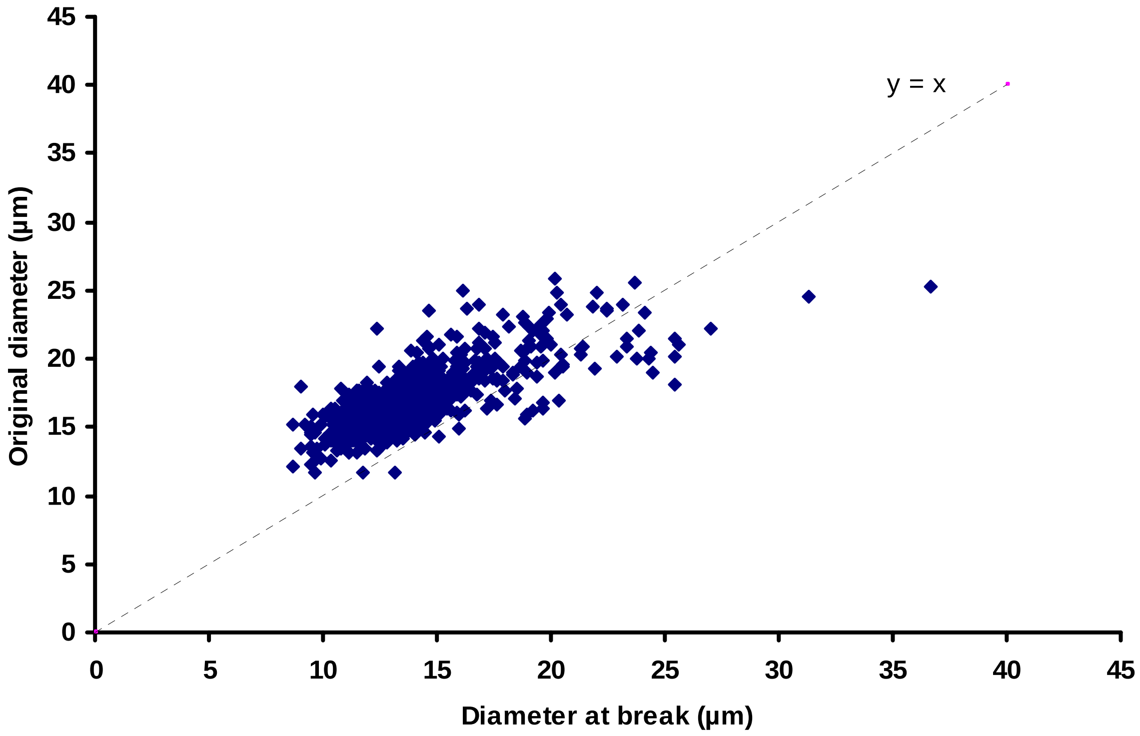

Figure 3: Relationship between original fibre diameter (vibroscope) and diameter at the break (optical microscope). Pooled data from all fibres from all sheep (n_{fibres} = 678)

Young's Modulus

As a first analysis, Young's modulus was plotted against fibre diameter at the break for all fibres from all sheep (Figure 4). Data from sheep "I" and "J" were overlaid to demonstrate the variation in diameter and modulus from fibres selected from sheep that have the same mean fibre diameter (16.9 µm) and same fibre curvature (73°/mm) but differ in RtC behaviour. The results indicate that for all fibres, Young's modulus is larger for finer fibres, with the modulus of individual fibres ranging from approximately 2000 to 9000 MPa, consistent with the work of Thompson (1998) who showed variation from 2000 to 7000 MPa. The low RtC wool had more fibres with high Young's modulus than the high RtC wool (Figures 4 and 6). Some researchers have used the $\log_{10}$ transformation to analyse their data (Evans, 1954, Thompson, 1998). When the $\log_{10}$ transformation was applied to this current data (Figure 5), Young's modulus was negatively correlated with diameter at the break (R^2 = 0.69). The low RtC and high RtC wool had correlation coefficients of 0.76 and 0.59 respectively.

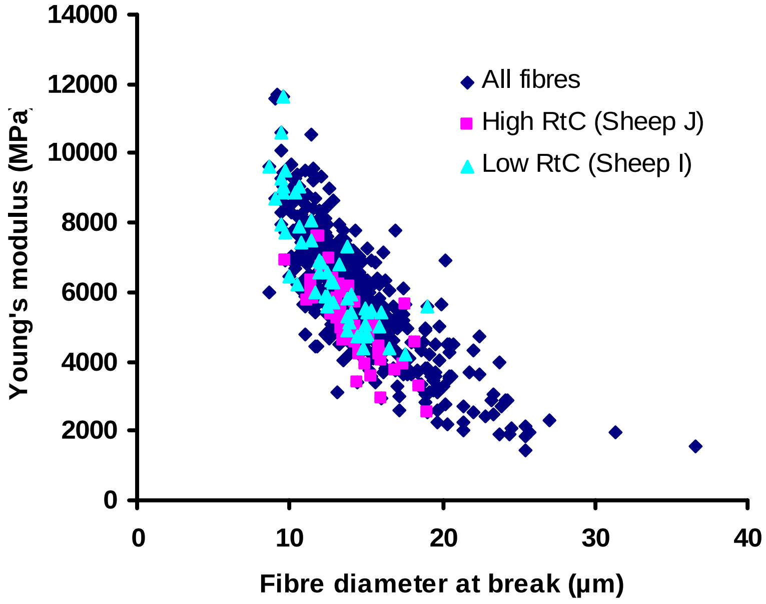

Figure 4 The effect of RtC on the diameter dependency of Young's Modulus

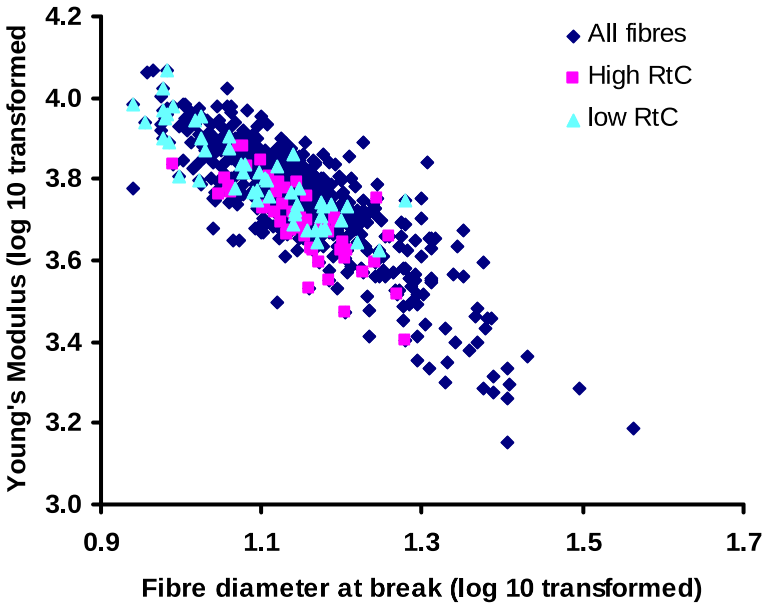

Figure 5 The effect of RtC on the diameter dependency of Young's Modulus where all data is log_{10} transformed.

Figure 6 illustrates the relationship between Young's modulus and RtC for the four categories of bulk fibre properties and also highlights the variation in Young's modulus from sheep to sheep One-way analysis of variance (ANOVA) was conducted using Minitab and the full analysis is provided in Appendix 1. The results indicate that there are difference in the average Young's modulus within each of the four groups, suggesting that there is an effect due to RtC at the 95% confidence level. In some cases the effect was only just significant at $\alpha = 0.05$. The overall trend was an increase in modulus for wools with lower RtC. This is opposite of what one would expect, viz. less stiff fibres would be expected to be softer and have lower RtC. Further work is needed in this area: specifically isolating the role of diameter, diameter uniformity, ellipticity and fibre surface properties.

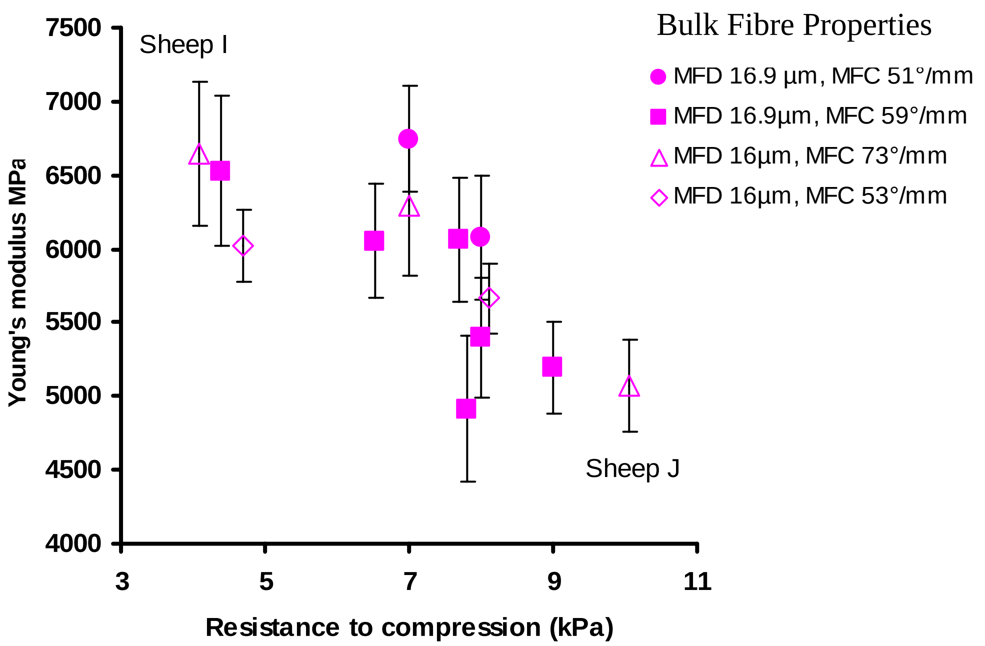

Figure 6: The relationship between Young's modulus and resistance to compression (RtC) showing the 95% confidence interval

Fibre Crimp Frequency

As can be seen from Figure 4, Young's modulus is highly dependent on fibre diameter and the literature suggests that fibre crimp also has an influence on Young' modulus (Evans, 1954, Dillon, 1952), hence the crimp frequency of individual fibres used in this study were also measured (Table 2a) and interestingly were found to be uncorrelated to the Laserscan measured curvature values (Table 1). Across all the sheep, the average reduction in crimp as a result of applying tension was 51% (range 12% to 86%). The reason for this large range in crimp reduction can not be explained in terms of fibre diameter or RtC alone, since the fibres that retained most of their original crimp were of average diameter and did exhibit variation in RtC.

Researchers have extrapolated the Hookean slope to the abscissa, and the corresponding force (F_0) at this point has been defined as the decrimping force (stress) or crimp parameter and the point F_{20} was used to characterise the region of the force-extension curve where there is a slow increase in force with extension as shown in Figure 7 (Balasubramaniam and Whiteley, 1974, Whiteley and Balasubramaniam, 1974, Evans, 1954). At F_0, the fibre is essentially straight . Evans (1954) studied only 12 fibres per sheep and found that F_0 increases more rapidly than the first power of the cross sectional area. Whiteley and Balasubramian (1974) found that soft handling fibres possessed low F_0 and low decrimping energy.

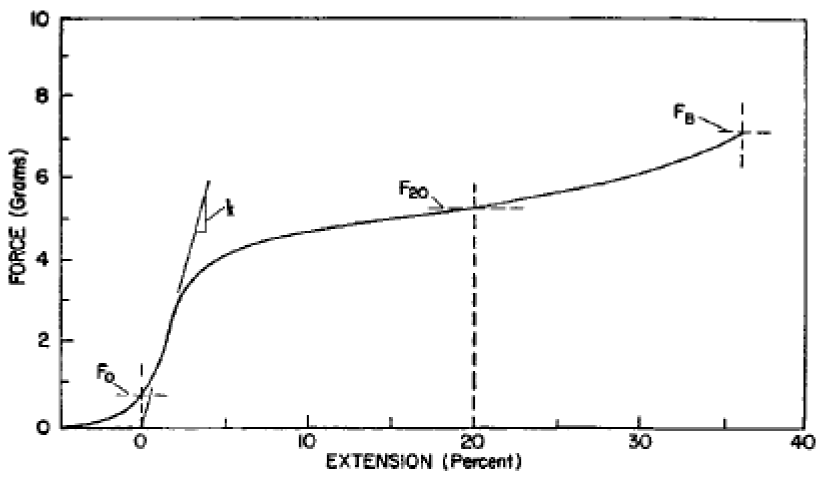

Figure 7: Typical force extension curve for a wool fibre (Evans, 1954)

Figures 8 to 12 show the relationships between specific fibre parameters and decrimping stress (Fo). The fibre parameters examined include the individual fibre properties of diameter and crimp frequency (tensioned and under no tension) and the measured bulk fibre properties of fibre curvature and RtC. The results indicate that decrimping stress is lower for larger diameter fibres and no correlation between decrimping stress and the other fibre or bulk fibre properties was found.

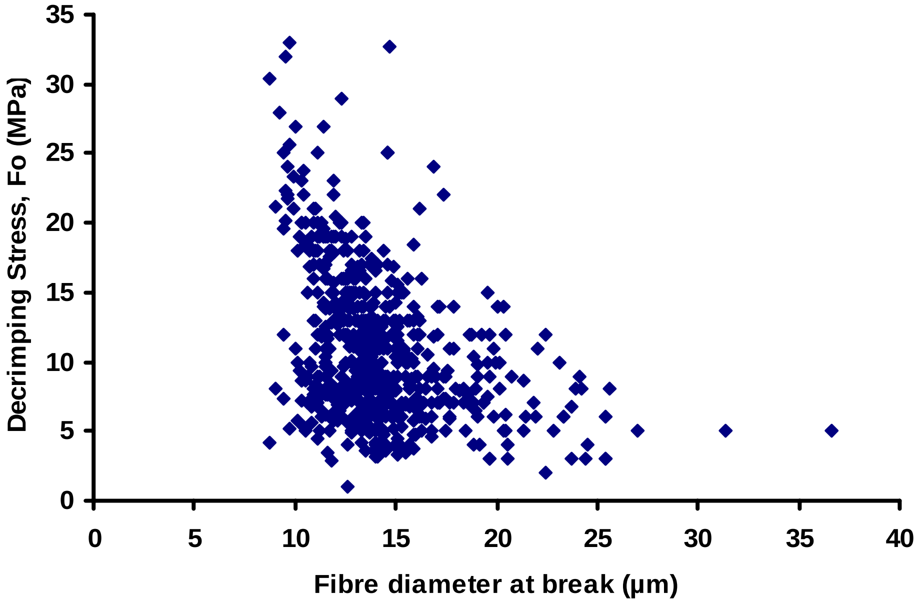

Figure 8: Relationship between fibre diameter at the break and decrimping force (F_0). Pooled data from all fibres from all sheep (n_{fibres} = 678)

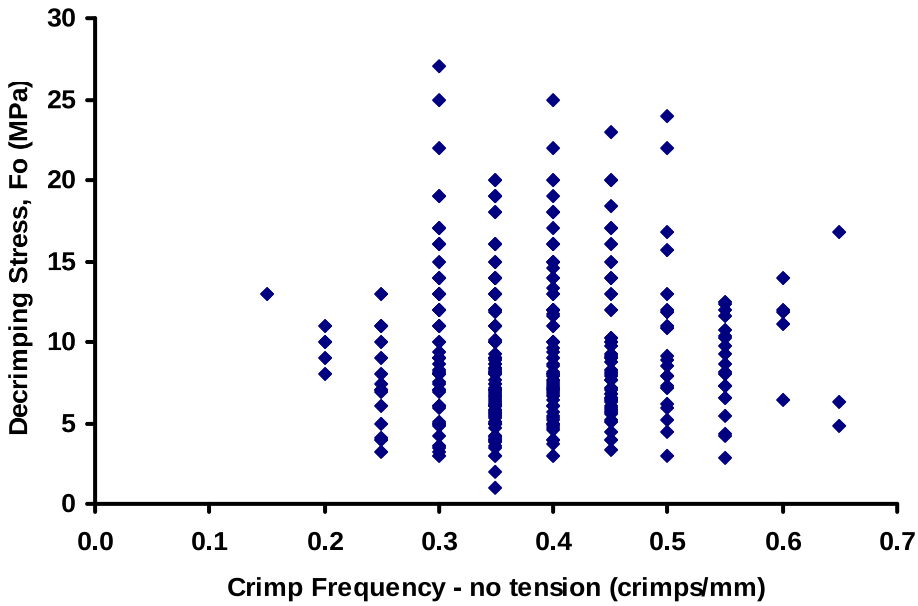

Figure 9: Relationship between crimp frequency (no tension) and decrimping force (F_0). Pooled data from all fibres from all sheep (n_{fibres} = 678)

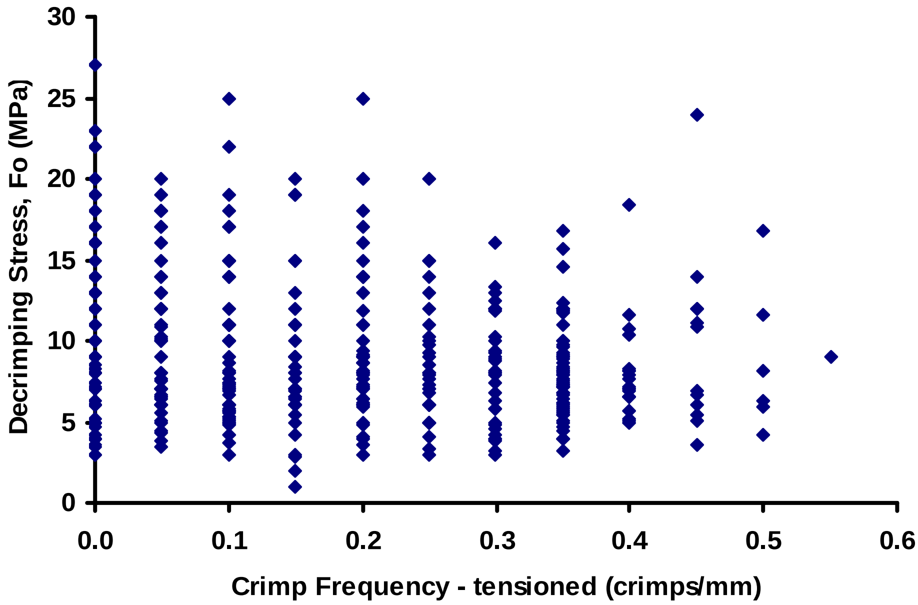

Figure 10: Relationship between crimp frequency (tensioned) and decrimping force (F_0). Pooled data from all fibres from all sheep (n_{fibres} = 678)

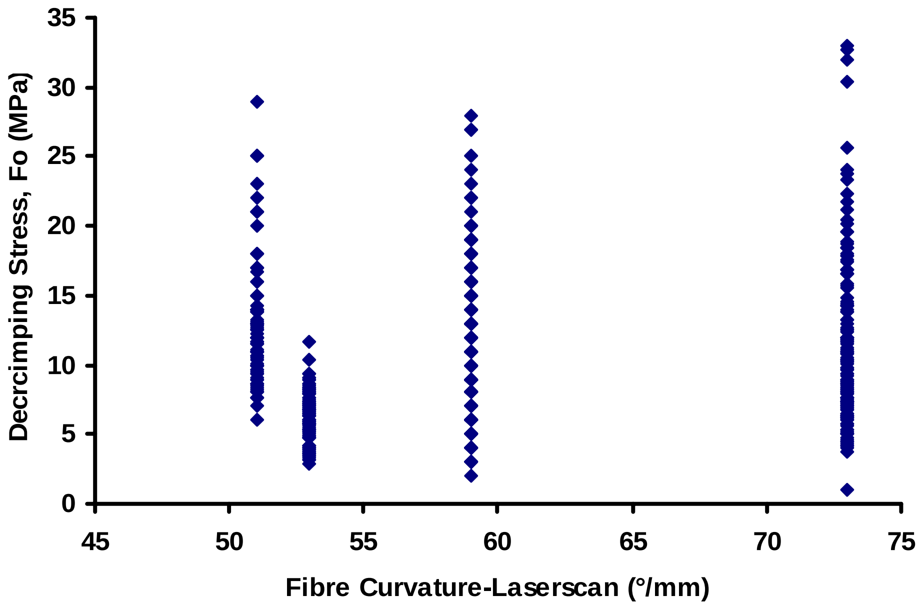

Figure 11: Relationship between Laserscan fibre curvature and decrimping force (F_0). Pooled data from all fibres from all sheep (n_{fibres} = 678)

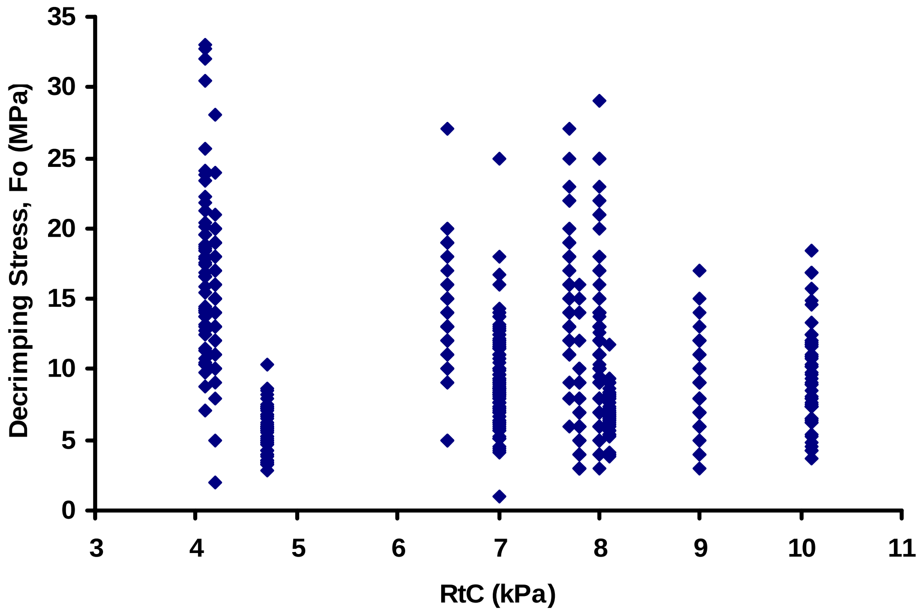

Figure 12: Relationship between RtC and decrimping force (F_0). Pooled data from all fibres from all sheep (n_{fibres} = 678)

Extension Behaviour

Approximately 99% of the all the fibres tested broke beyond the Hookean region at about 3% extension, with more than 86% breaking at extensions greater than 30%. Across all fibres the average breaking extension in air at 20 °C and 65% relative humidity was 35%. The average coefficient of variation in strain at break between individual fibres from the same sheep was 22.6% (range 9% to 26%).

Young's modulus in relation to stress at 15% strain (F_{15}) and stress at break

Evan's used the stress at 20% strain to characterise the region of the force-extension curve where there is a slow increase in force with extension as shown in Figure 4. More recently researchers, particularly those looking at tender wools, use the stress at 15% strain (F_{15}) to characterise the yield region (Thompson, 1998). In this study the both the stress at 15% strain and the stress at break were examined in relation to Young' modulus as shown in Figures 13 and 14 respectively. Both the stress at break and stress at 15% strain were positively correlated with Young' modulus.

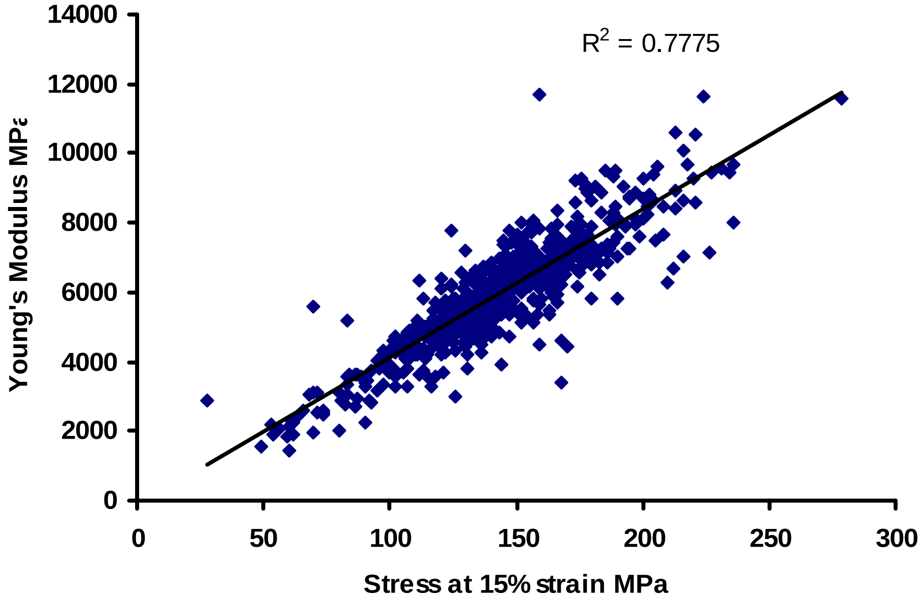

Figure 13: Young's modulus in relation to stress at 15% strain (F_{15}). Pooled data from all fibres from all sheep ($n_{fibres} = 678$)

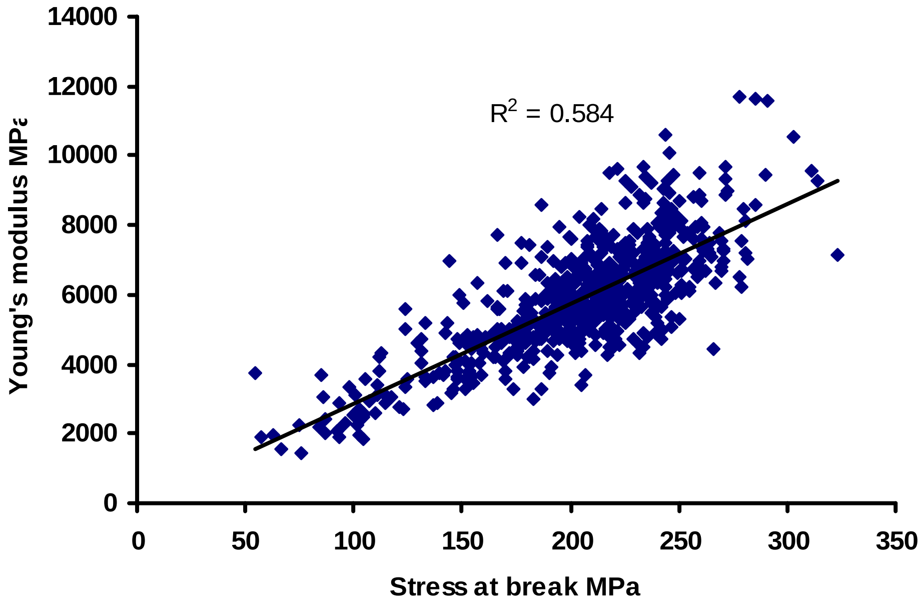

Figure 14: Young's modulus in relation to stress at break. Pooled data from all fibres from all sheep ($n_{fibres} = 678$)

Sheep Code	RtC (kPa)	Crimps (cm^{-1})		Fibre Diameter (μm) at		Young's Modulus (MPa)	Stress (MPa) at		Strain at point of break (%)
		Not Tensioned	Tensioned	Original - vibroscope	Break		Break	15% strain	
A	7.0	N/A	N/A	16.25 (7)	13.24 (8)	6746 (19)	220 (15)	147 (16)	41 (13)
B	8.0	N/A	N/A	17.40 (14)	13.20 (17)	6081 (26)	207 (19)	148 (22)	38 (19)
C	4.2	3.9 (13)	1.1 (100)	18.86 (19)	15.23 (33)	6527 (28)	215 (23)	158 (28)	31 (25)
D	6.5	N/A	N/A	16.25 (9)	13.05 (17)	6054 (23)	210 (19)	139 (21)	40 (17)
E	7.7	3.6 (18)	0.5 (131)	15.57 (10)	14.24 (22)	6067 (25)	203 (20)	152 (23)	37 (26)
F	7.8	3.6 (16)	1.2 (89)	18.44 (10)	16.25 (24)	4913 (39)	188 (28)	127 (31)	36 (22)
G	9.0	3.7 (19)	1.5 (94)	18.88 (11)	16.3 (16)	5194 (23)	190 (24)	130 (19)	36 (24)
H	8.0	3.1 (17)	1.1 (99)	16.41 (17)	14.66 (29)	5424 (29)	200 (26)	135 (24)	38 (18)
I	4.1	N/A	N/A	15.56 (13.2)	12.65 (19)	6644 (27)	206 (16)	145 (21)	36 (21)
J	10.5	5.0 (17)	2.5 (59)	15.85 (11)	14.26 (14)	5071 (22)	192 (21)	134 (20)	37 (22)
K	7.0	3.7 (16)	2.4 (52)	16.9 (9)	13.45 (22)	6287 (27)	202 (22)	155 (24)	33 (25)
L	4.7	3.8 (17)	3.0 (37)	15.97 (12)	13.89 (12)	5971 (15)	221 (12)	137 (14)	40 (10)
M	8.1	4.1 (14)	3.6 (22)	16.92 (12)	14.13 (15)	5665 (15)	212 (13)	134 (14)	41 (9)

Table 2a: Average fibre physical and tensile properties. Value in brackets are CV%.

Sheep Code	RtC (kPa)	Stress (MPa) at	
		0% strain	15% strain
A	7.0	11 (29)	147 (16)
B	8.0	15 (23)	148 (22)
C	4.2	15 (28)	158 (28)
D	6.5	14 (28)	139 (21)
E	7.7	15 (29)	152 (23)
F	7.8	7 (43)	127 (31)
G	9.0	9 (34)	130 (19)
H	8.0	9 (50)	135 (24)
I	4.1	18 (35)	145 (21)
J	10.5	10 (36)	134 (20)
K	7.0	7 (30)	155 (24)
L	4.7	6 (31)	137 (14)
M	8.1	7 (20)	134 (14)

Table 2b: Average fibre RtC and stress properties. Value in brackets are CV%.

PART 2

Evaluation of the Single Fibre Analyser (SIFAN 3) and Laser Diffraction for Measuring Fibre Diameter

Introduction

The diameter and shape of wool fibre directly influences the mechanical and physical properties of the fibre (Collins and Chaikin, 1965, He and Wang, 2002, Xu et al., 1993, Morton and Hearle, 1993). In addition, wool fibres are irregular fibres and the diameter and shape changes along the fibre length. This non-uniformity along the fibre length is a result of seasonal growth cycles and nutrition effects (Reis et al., 1990). Due to the irregular nature of the diameter and shape of wool fibres, accurate methods for characterising single wool fibres for diameter and shape or ellipticity along the length of the fibre are of considerable interest.

Fibre diameter is the single most important wool characteristic determining quality and price and consequently many measurement techniques have been developed. The standard methods currently available to measure wool fibre diameter include the Airflow method (IWTO-6-98), the Optical Fibre Diameter Analyser (OFDA) (IWTO-47-00), the Sirolan-Laserscan (IWTO-12-03) and projection microscope (IWTO-8-97). The OFDA and Sirolan-Laserscan methods are the most commonly used methods to determine the mean fibre diameter (MFD), diameter distribution and variation (CVD) and curvature of wool fibres at all stages of the wool processing pipeline, from fleece (on-farm) to finished fabric. However these methods do not provide information on the diameter profiles or ellipticity of individual wool fibres. The diameters of single fibres are typically measured optically using a projection microscope. This is a laborious technique and provides information only about the diameter in one random orientation. Although wool fineness is always given as a diameter, the cross-sectional shape or ellipticity of wool fibres can vary. Fibre ellipticity is normally determined from the length ratio of the major and minor axis of thin, transverse fibre cross sections. The ellipticity generally varies from 1 to 2, with the average ellipticity for Merino wool about 1.3 (Ly and Denby, 1984, Naylor, 1998). The preparation of the thin transverse cross sections from wool fibre for ellipticity studies is very time consuming and tedious. Care must be taken to ensure that the wool fibres are pre-tensioned to remove any crimp prior to embedding and sectioning and that the transverse cross-sections are taken perpendicular to the fibre axis otherwise ellipticity can be induced. For example, if θ the angle of the cross-sectional face to the fibre axis is 25 degrees, then a circular fibre may appear to have an ellipticity of 1.103. Fibre ellipticity, in principle, can influence the fibre diameter results obtained from Sirolan-Laserscan and OFDA because the measurement techniques are based on projected width measurements. However Naylor (1998) reported that an ellipticity of 1.3 (i.e. average ellipticity of Australian merino wool) would not have any significant effect on the Sirolan-Laserscan measured mean fibre diameter.

Laser diffraction is a technique used to accurately measure the diameter of thin fibres such as glass fibre (Meretz et al., 1992) and wire (Khodier, 2004). These researchers have shown that laser diffraction is an accurate and rapid technique for measuring diameter. However the presence of fibre ellipticity would result in under or over estimations of fibre diameter. A novel approach being investigated in this work is to use the laser diffraction diameter measurement technique with a fibre rotating device (Collins, 1963) so that both fibre diameter profile and ellipticity can be determined from diameter measurements made at a number of different fibre orientations along the length of the fibre.

The Single Fibre Analyser or SIFAN 3 developed by BSC Electronics Pty Ltd., WA, is a purpose built, commercial instrument that is able to measure fibre diameter at any number of different orientations by rotating the upper and lower jaws. The SIFAN 3 works by recording the shadow cast by the fibre. The width of the shadow is detected by a Charge Coupled Device (CCD) camera. Yu (2002) used single SIFAN scans to study the average fibre diameter, average minimum and maximum diameter as well as their coefficients of variation of single fibres extracted from top. Yu found high correlations between the mean fibre diameters of wool top measured by Laserscan, OFDA and SIFAN. Deng (2007) used the SIFAN 3 to study the effects of wool fibre irregularity and cross sectional area variations on yarn limiting irregularity. He found large variation in diameters measured from repeated single scans and suggested that multiple orientation scans allows more accurate diameter measurement. Wang (2007) examined the measurement precision of SIFAN 3001 and used the instrument to examine the diameter profiles of single wool fibres at four different orientations. They obtained a value of fibre ellipticity by averaging the two orthogonal diameter ratios obtained from the four orientations and since these orientations were not necessarily at the major and minor axes, they reported a lower than expected ellipticity for wool (1.08 ±0.01) with the typical range of ellipticity of Merino wool of 1.18 – 1.25 (Champion and Robards, 2000).

This study extends the SIFAN 3 ellipticity study by Wang (2007) and examines the diameter profiles at six different orientations instead of four different orientations. Wang (2007) used the average of the orthogonal diameter ratios as an estimate of fibre ellipticity, however it would make more sense to use the maximum ratio as an estimate of ellipticity. In this study the maximum of three orthogonal ratios is used as an estimation of fibre ellipticity. This study also examines laser diffraction coupled with a fibre rotator as an alternative way of measuring diameter at six different orientations for fibre ellipticity studies. Initially uniform wire was used to evaluate these alternative methods to establish whether they would be suitable for wool fibre studies. These methods will be used to measure the diameter of two fine wires at six different orientations around the wire. These measurements will allow the ellipticity of the two fine wires to be estimated.

Materials and Method

Two tungsten calibration wires, supplied by Graham Higgerson, a nominal 10 micron wire and a nominal 25 micron wire were used to assess the suitability of the SIFAN 3 and laser diffraction as methods of diameter measurement at multiple orientations. Three samples of each wire were used for the diameter measurements on SIFAN 3, split beam laser diffraction and single beam laser diffraction methods.

For SIFAN 3 diameter measurements, wires were mounted on to plastic tab with double sided sticky tape at a gauge length of 50mm. Each wire was pre-tensioned under a 0.5 cN force prior to scanning to remove crimp and/or fibre slack. Fibres were scanned at six orientations ie., 0°,30°,60°,90°,120° and 150°, at a motor speed of 7mm/sec with a step of 0.015mm. Outliers and spurious measurements at +/- 3 standard deviations of the mean were removed by averaging data points on either side of the outlier. The data was then smoothed by means of a moving average of five diameter results.

For the laser diffraction diameter measurements, wires were mounted onto a single fibre rotator similar to that developed by Collins (1963) and positioned perpendicular to a diode laser (λ = 656 nm). Each wire was pre-tensioned by fixing one end of the wire to the fibre rotator and applying a 200mg vibroscope weight (0.2 cN force) to the other end. A gauge length of approximately 50mm was used and the wires were held in place with magnets. The wire was positioned perpendicular to the laser, thus setting up a diffraction pattern that was displayed on a screen located 522 mm and 793mm from the 10μm and 25μm wires respectively for the split beam laser diffraction method, and 2291mm from the 10μm wire for the single beam laser diffraction method. The diffraction pattern was traced onto paper and the distance between the fringes was measured with a ruler. The diameter of the wire was calculated from the first diffraction minima using Bragg's law (Equation 1), the small angle approximation where $\sin\theta \approx \tan\theta$ (Equation 2) and is expressed as (Equation 3).

$$n\lambda = d \sin\theta \qquad (1)$$

$$n\lambda = d \tan\theta \qquad (2)$$

$$where\ \tan\theta \equiv \frac{x}{2D}, \quad \text{so}\ d = \frac{2n\lambda D}{x} \qquad (3)$$

where d is the diameter of the wire (μm), n is the order of diffraction ($n=1$, for the first diffraction minima), $\lambda = 0.656$ i.e. the wavelength of the incident laser (μm), θ is the diffraction angle, D is the distance to screen from wire (mm) and x is the distance between the diffraction fringes (mm). The diameter of the wires at four positions along the fibre were measured at six orientations ie., 0°,30°,60°,90°,120° and 150°.

For each of the diameter measurement techniques, the orthogonal diameters at 0° and 90°, 30° and 120° and 60° and 120° were used to calculate diameter ratio 1, ratio 2 and ratio 3 respectively. The maximum ratio was taken as the ellipticity of the fibre at that point. The average maximum diameter ratio for each fibre was used as an indication of the fibre ellipticity.

The diameter of the wires were also measured optically, in one random plane, using an optical microscope (Leitz DMRBE microscope) fitted with a 50x air objective and a Leica DC300F digital camera) and a Leica QWin Pro Version 3.3.1 image analysis system. Diameter measurements were made approximately every 10mm along the length of the wire.

Results:

Compared to natural fibres such as wool, wire is relatively uniform in diameter, and is the reason why wire was chosen to evaluate the SIFAN 3 and laser diffraction methods of estimating ellipticity by measuring diameter at multiple orientations. Table 3 shows the diameter results for both wires measured by SIFAN 3, laser diffraction and optical microscopy. It is clear that the diameters measured by SIFAN 3 method were lower and more variable than the diameters measured by optical microscope and laser diffraction methods. The lower diameter values may indicate an issue with the alignment of fibre samples in the jaws of the SIFAN 3. Figure 15 shows the optical microscope images of the nominal 10 micron wire and it is clear from these images that the wire is uniform in diameter for that particular orientation and that dirt contaminants on the wire could interfere with the diameter measurements. Figure 16 shows the diameter profile constructed from SIFAN 3 data for the nominal 10 micron wire at the 60 degree orientation where the diameter ranges from 4μm at one end of the wire to approximately 11μm at the other end. Clearly this indicates an error in calibration from one end to the other of the test region. This in itself would not matter if the error was independent of rotation as we are only interested in the diameter ratios. However the data for the 150 degree orientation suggests that the error is not independent of rotation, leading to a measured ellipticity that varies significantly along the length of the wire (Figure 17).

Also evident in Figure 16 is the presence of outliers that were not removed by the data smoothing algorithm. The problem is due to the high standard deviation obtained for some of the data and suggests that an iterative data smoothing process may be required.

Pre-tensioning the wire was also essential in order to obtain clear diffraction patterns for the laser diffraction diameter measurement method. The measurement method is simple, however care must be taken in measuring distance to screen (D) and fringe spacing (x). An error in measurement of D of ±10mm will result in an error of ±0.3μm whereas an error in measurement of x of only ± 1mm will result in an error of ±0.6μm for the 25μm wire.

Discussion:

The results show that the laser diffraction method is a quick and straight forward technique for measuring fibre diameter, and when coupled with a fibre rotator can be used to measure the diameter at several orientations enabling ellipticity to be estimated. The split beam system is preferred over the single beam system because it allows multiple points to be measured at each position along the fibre. The laser diffraction method however is subject to operator accuracy in measuring fringe spacing. This error can be removed if a camera and image analysis system is employed and this is being currently investigated. A further improvement also being investigated is setting up a laser diffraction system directly onto the frame of an Instron tensile tester and instead of rotating the fibre, the laser is moved through an arc, allowing diameter to be measured at any number of orientations. The SIFAN 3 diameter measurement system requires further investigations. The SIFAN 3 will be recalibrated using wires finer than 20µm and the fibre alignment and pre-tensioning will be investigated.

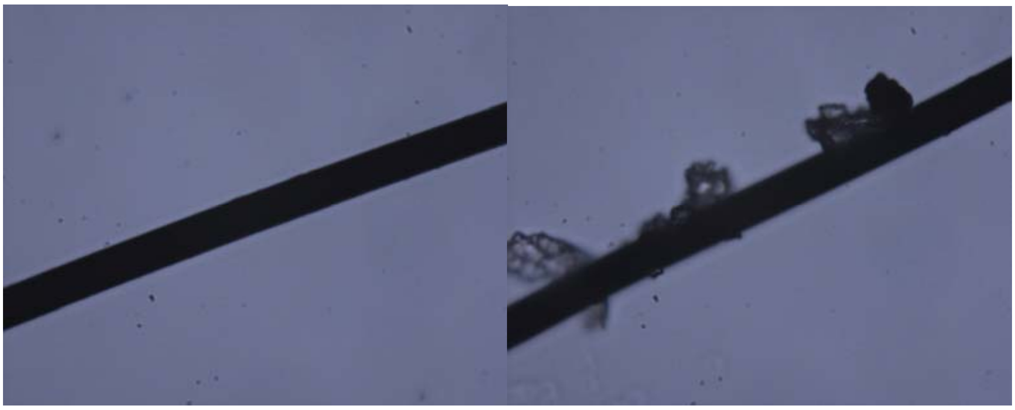

Figure 15: Nominal 10 micron tungsten wire

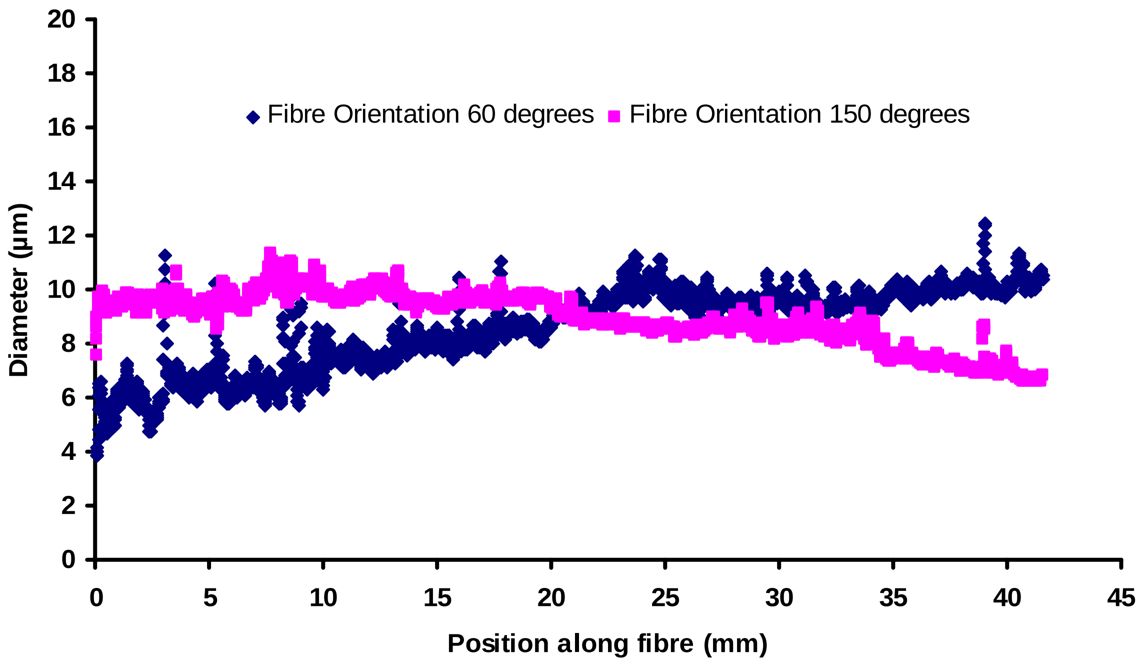

Figure 16: Orthogonal diameter profile of nominal 10 micron wire obtained from SIFAN 3 at an orientation of 60 degrees and 150 degrees.

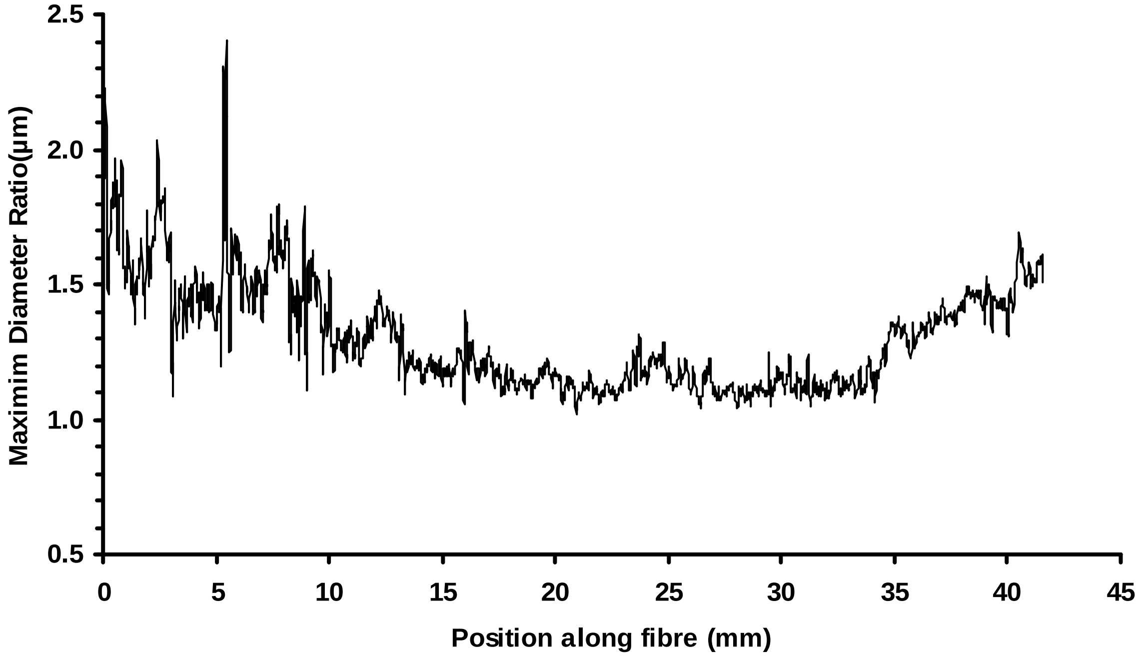

Figure 17: Maximum diameter ratio (ellipticity) of nominal 10 micron wire obtained from SIFAN 3 at an orientation of 60 degrees and 150 degrees.

Table 3: Nominal 10 and 25 micron wire - comparison of average fibre diameter (FD, µm) and standard deviation (SD, µm) along the length of wire by optical microscopy (OM), laser diffraction split beam (LDSplit), laser diffraction single beam (LDSingle) and SIFAN 3 and the average maximum orthogonal diameter ratio (Max ratio)

Orientation	OM		LDSplit		LDSingle		SIFAN 3	
	FD	SD	FD	SD	FD	SD	FD	SD
Nominal 10 micron wire								
Random	11.9	0.3						
0 °			11.6	0.2	11.9	0.3	9.1	0.8
30 °			11.8	0.1	12.4	0.2	8.9	1.1
60 °			11.8	0.2	11.9	0.1	8.6	1.5
90 °			11.9	0.2	11.9	0.1	9.1	1.2
120 °			11.8	0.2	12	0.1	9.3	0.6
150 °			11.7	0.2	12	0.0	8.9	1.0
Ave. Diameter			11.7	0.1	12.0	0.3	9.0	1.1
Ave.Max Ratio			1.02	0.01	1.03	0.03	1.30	0.2
Nominal 25 micron wire								
Random	25.9	0.4						
0 °			25.4	0			24.1	0.4
30 °			25.5	0.5			24.7	0.4
60 °			25.3	0.5			25.4	0.4
90 °			25.1	0.6			25.4	0.9
120 °			25.4	0.6			24.7	0.8
150 °			25.2	0.2			23.9	1.1
Ave. Diameter			25.3	0.4			24.7	0.9
Ave.Max Ratio			1.03	0.01			1.09	0.03

Number of points along the fibre length at each orientation where fibre diameter was measured by the different methods: OM: 5 positions (random orientation), LDSplit: 32 (positions with each point split into 8 points), LDSingle: 3 positions and SIFAN 3: 2694 positions.

Appendix 1: Anova testing of RtC of the four bulk fibre property groups

Bulk fibre properties MFD 16μm and MFC 53°/mm – Sheep "L and M"

```
Source   DF        SS       MS     F      P
C1        1   3270016  3270016  4.22  0.043
Error   100  77532023   775320
Total   101  80802039

S = 880.5   R-Sq = 4.05%   R-Sq(adj) = 3.09%


                          Individual 95% CIs For Mean Based on
                          Pooled StDev
Level   N    Mean  StDev  ---+---------+---------+---------+------
1      51  6023.2  897.2              (---------*---------)
2      51  5665.1  863.5  (---------*--------)
                          ---+---------+---------+---------+------
                          5500      5750      6000      6250

Pooled StDev = 880.5
```

Result: Reject Ho – there is sufficient evidence to suggest that the average Young's modulus of Sheep L and M are not all equal at α = 0.05.

Bulk fibre properties MFD 16.9μm and MFC 51°/mm – Sheep "A and B"

```
Source   DF         SS        MS     F      P
C1        1   11262159  11262159  5.47  0.021
Error   100  206043132   2060431
Total   101  217305291

S = 1435   R-Sq = 5.18%   R-Sq(adj) = 4.23%


                        Individual 95% CIs For Mean Based on
                        Pooled StDev
Level   N  Mean  StDev  --------+---------+---------+---------+-
1      49  6746   1289                  (----------*---------)
2      53  6081   1559  (---------*---------)
                        --------+---------+---------+---------+-
                             6000      6400      6800      7200

Pooled StDev = 1435
```

Result: Reject Ho – there is sufficient evidence to suggest that the average Young's modulus of Sheep A and B are not all equal at α = 0.05.

Bulk fibre properties MFD 16.9µm and MFC 73°/mm – Sheep "I, J and K"

```
Source   DF         SS        MS      F      P
C1        2   69345061  34672531  14.24  0.000
Error   150  365282126   2435214
Total   152  434627188

S = 1561   R-Sq = 15.96%   R-Sq(adj) = 14.83%


                        Individual 95% CIs For Mean Based on
                        Pooled StDev
Level   N  Mean  StDev  ----+---------+---------+---------+-----
1      51  6644   1761                       (-----*-----)
2      51  5071   1121  (-----*------)
3      51  6287   1717                  (-----*-----)
                        ----+---------+---------+---------+-----
                         4900      5600      6300      7000

Pooled StDev = 1561
```

Result: Reject Ho – there is sufficient evidence to suggest that the average Young's modulus of Sheep I, J and K are not all equal at α = 0.05.

Bulk fibre properties MFD 16.9µm and MFC 59°/mm – Sheep "C,D,E,F,G and H"

```
Source   DF         SS        MS     F      P
C1        5  102061671  20412334  8.00  0.000
Error   318  811343077   2551393
Total   323  913404748

S = 1597   R-Sq = 11.17%   R-Sq(adj) = 9.78%


                        Individual 95% CIs For Mean Based on
                        Pooled StDev
Level   N  Mean  StDev  ------+---------+---------+---------+---
1      51  6527   1852                       (-----*------)
2      51  6054   1416                (-----*------)
3      51  6067   1534                (------*-----)
4      58  4913   1915  (-----*-----)
5      54  5194   1172      (-----*-----)
6      59  5424   1554          (----*-----)
                        ------+---------+---------+---------+---
                           4900      5600      6300      7000

Pooled StDev = 1597
```

Result: Reject Ho – there is sufficient evidence to suggest that the average Young's modulus of Sheep C,D,E,F,G and H are not all equal at α = 0.05.

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