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Relationship between morphology and mechanical properties of tobacco leaf lamina

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To optimize processing parameters, a study was done of the overall mechanical properties of tobacco leaf lamina. Stress–strain tests were performed to determine modulus, yield strength, and toughness (energy to break) of the lamina when deformed under tension and simple shear loads. The cellular morphology of the lamina was obtained by using scanning electron microscopy. Microscopic observation of deformed samples showed an uneven stress distribution and anisotropy. The macroscale phenomenological results indicated a harmony in its mechanical behaviour. Lamina isotropy and homogeneity in the plane of a leaf were determined by testing samples taken from different orientation and locations. Bright and burley tobacco were studied. The relationship between tension and shear properties was also obtained. Both in shear and tension, the lamina became softer with an increase in load. The stress–strain behaviour was characterized by a two-parameter power law equation. Statistically estimated results, obtained from several leaves, did not indicate any significant effect of sample location and orientation. Tobacco lamina appeared softer but tougher in shear than tension. It also showed sensitivity to the rate of deformation, indicating the viscoelastic nature of its behaviour. Material yield during manufacturing related well with mechanical properties. Burley lamina was stiffer and broke at lower strain than bright lamina. Thus, even though at the microlevel, tobacco lamina, is heterogeneous and anisotropic, at the macrolevel it displays homogeneous and uniform mechanical properties.

1. Introduction

Natural materials are commercially important in many industries. During processing they are often subjected to severe environmental and mechanical forces. Processing conditions are mostly developed empirically, and expertise is usually associated with many years of experience. In recent years, the mechanical properties of natural materials have been systematically investigated from various perspectives [1–12]. Most of this has been prompted by the obvious importance of strength and toughness in the end uses, such as paper products, textiles, wood, etc.

This paper describes a similar investigation aimed at determining the fundamental relationships important to the processing of a commercially valuable natural material, i.e. tobacco lamina. The objective is to develop an understanding of relationships between mechanical properties of materials and processing parameters. Ultimately, this should lead to optimization of product quality as determined by processing conditions. The approach described here should be widely applicable to many natural materials.

Natural materials are multicellular polymeric composites made of various types of cells, each of which is organized in definite forms. Each cell is a complex structure of many organic polymers arranged in fixed patterns. It is interesting to find that, starting from atomic and subatomic particles, these materials are remarkably organized in several hierarchical levels with definite morphology at each scale of organization [1, 8, 10, 13]. Each level of organization presumably affects the macroscopic behaviour of the material. Theoretically, it should be possible to extrapolate from the microscopic level structure properties to the macroscopic scale. The micromechanics theory for composite materials is applicable only under sufficiently simple conditions [14–16]. In practice, however, it is virtually impossible to progress in this way because of the complexities of natural materials. Nevertheless, a knowledge of the microstructure is helpful in analysing the macroscale observations. This work will show its usefulness in the phenomenological measurements. Specifically, it will serve as the basis to generalize the observations made on a gross scale. The data will illustrate that a material can be inhomogeneous and anisotropic at one level, but be isotropic and homogeneous at the next level of observation. This behaviour is particularly important in determining the effects of processing parameters on material mechanical properties.

2. Leaf structure

The physical structure of a typical tobacco plant leaf is shown in Fig. 1. The lamina (the region between the lateral veins) is made up of four different types of cells: upper epidermis, palisade, spongy mesophyl, and lower epidermis. Importantly, the arrangement of cells is the same in all directions [1, 17, 18]. Also

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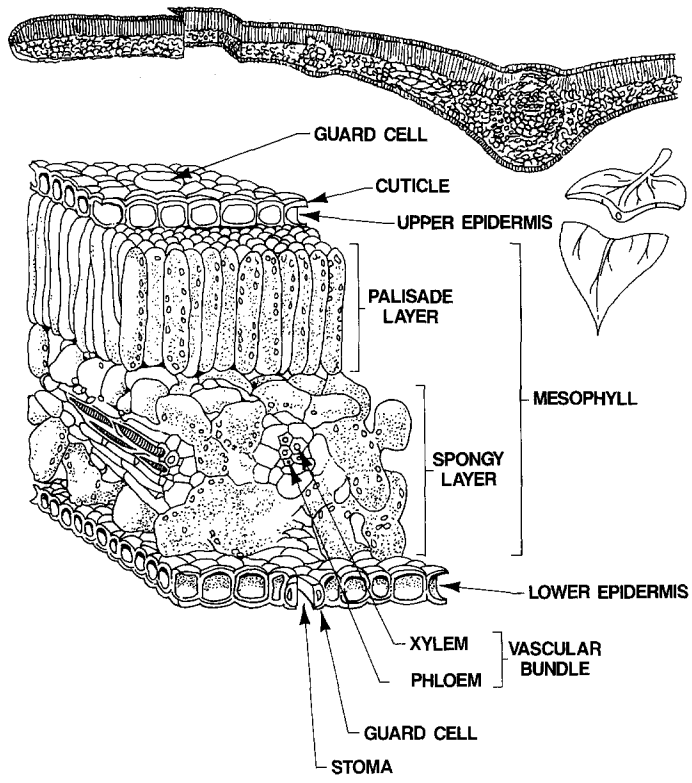

Figure 1 Stereoscopic diagram of a tobacco leaf showing the location of various cells and tissues.

present in the lamina are: a network of veins (vascular bundle), void spaces, inorganic materials, etc.; which are distributed randomly throughout the lamina. Leaves with such uniform distribution of cells and random arrangement of veins are classified as dicotyledonous (dicot) leaves [18]. Whereas those with longitudinally straight venation are called monocotyledons (monocots) [18]. Examples of the latter are leaves of pine, banana, jute, grass, etc. Each type of cell is composed, mainly, of various polymers with each having their own structure, then substructure and so on down. Bokelmann and his collaborators have done an extensive study on the structure of cell wall biopolymers [19, 20]. Most of the space inside the cell is filled with an aqueous solution. The dimensions of various cells of a green tobacco leaf are given in the work by Atkinson and Echlin [18].

To make useable products, green leaves are cured after harvesting from the fields. Two important varieties are: flue cured (also called bright or Virginia tobacco) and air cured (commonly known as burley tobacco). In flue curing, leaves are heated at several elevated temperatures, up to 90° C, for about a week. In air curing, the entire stalk is dried in air for about 4 weeks. In all methods of curing, several biochemical changes take place. A list of an approximate chemical composition of a bright an burley cured leaf is given in Table I [20]. Physically, due to loss of water and, hence, the osmotic pressure, cells collapse during the curing process. Fig. 2 shows typical scanning electron micrographs (SEMs) of the lamina cross-section of a green and a cured leaf. Notice the collapse of the cellular structure in the cured lamina. A similar surface micrograph of a cured leaf is shown in Fig. 3. The thicknesses of cured leaves can vary from 0.05 to 0.2 mm. Also, there is some thickness variation within the same leaf. It can be seen from these figures that at microlevel, tobacco laminae are anisotropic and nonuniform.

3. Material and sample preparation

Most of the work was done on bright cured lamina. Whole leaves were obtained from the 1985 bright tobacco crop grown in Southwest Virginia. All leaves were taken from the middle of the plant. A few tests were done to compare different types of lamina acquired from the Manufacturing Center of Philip Morris USA, Richmond, Virginia.

Test samples were strips cut from the lamina by using a sharp razor blade. Care was taken to avoid any major vein passing through the sample. Fig. 4 shows both tension and shear specimens. As shown in this figure, small strips of paper tape were glued at the ends

TABLE I Biopolymer cell wall compositions of bright and burley lamina (all values expressed on a percentage dry weight basis)

Tobacco component	Bright lamina	Burley lamina
Ethanol solubles	56.3	31.5
Aqueous solubles lost during dialysis	5.2	19.5
Acid detergent solubles	3.2	8.2
Pectin	10.7	11.5
Starch	3.2	trace
Protein	6.8	10.4
Hemicellulose:		
soluble	2.8	2.3
associated with cellulose	0.8	0.9
Lignin	1.7	2.1
Cellulose	5.9	6.4
Ash:		
soluble	2.8	1.2
insoluble	1.0	0.6
Total	100.4	95.2

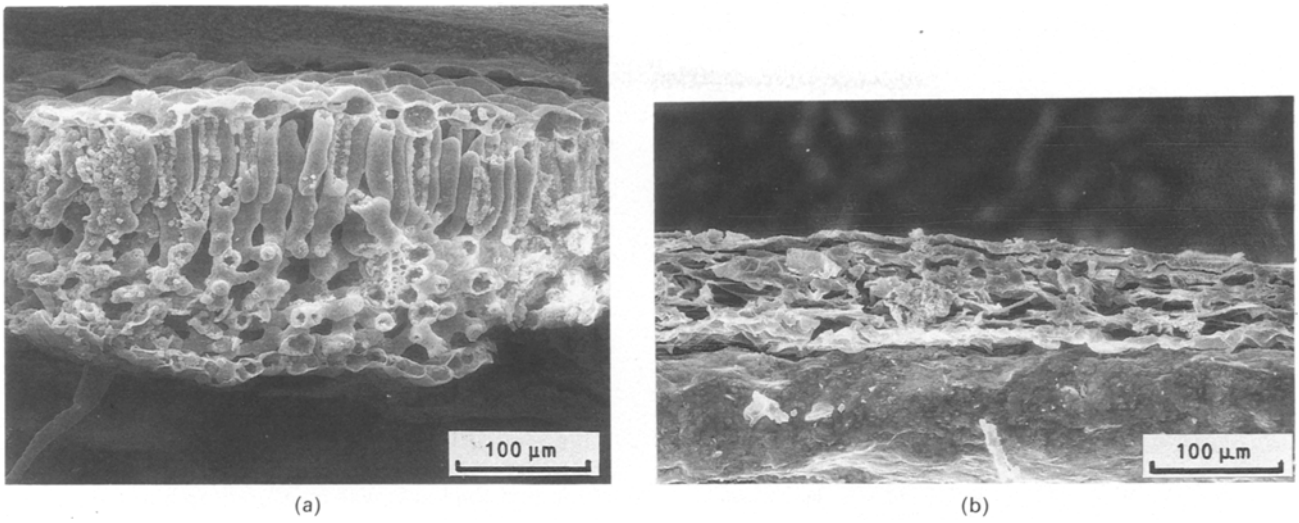

Figure 2 Scanning electron micrographs of the cross-sections of tobacco leaves. (a) Bright ripe leaf showing all four types of cell. (b) Bright flue-cured leaf showing the collapse of the cellular structure.

of all tension specimens. This was done to distribute the applied force uniformly across the entire width of the sample during a tensile test. Typical dimensions of most tensile specimens were: length $L = 40$ mm, width $W = 15$ mm, and thickness $t = 0.08$ to 0.17 mm. Sample length was kept at 40 mm to minimize the stress concentration effects [21, 22]. For shear testing, two strips of lamina of length, L, effective width, h, (overall width, W), and thickness, t, were glued to two rigid aluminium mounts, one on each surface of the mounts. The reason for mounting the lamina on two sides was to apply simple shear load [21, 22]. For the same reason, the length-to-thickness ratio (L/h) was kept greater than 8 [21]. The surfaces of both the mounts were cleaned by dipping them in acetone. A thin coat of adhesive, Loctite 404, was applied on one side of the inner and outer mounts. A tobacco strip was then gently pressed over all three metal surfaces. It took about 2 to 3 min to form a good bond between the tobacco and metal surfaces. The same procedure was used to bond tobacco on the other side of the mounts. An insert, shown in Fig. 4, was used to align the inner mount parallel to the outer mount. Sample dimensions L and h were 20 and 2 mm, respectively.

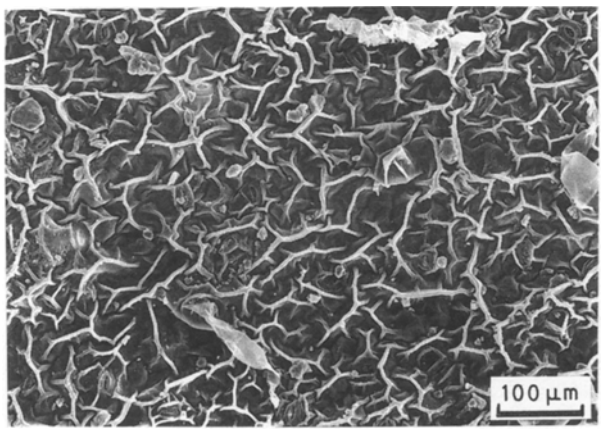

Figure 3 Scanning electron micrograph of the surface of a cured leaf lamina.

4. Experimental procedure

All tests were performed on the Dynastat, a viscoelastic property tester distributed by Imass, Inc., Hingham, Massachusetts. For stress–strain tests, a separate triangular wave generator and an oscilloscope were also required. The instrument is an electromagnetically operated feedback control system. Because of this, by using the displacement control mode, a constant strain rate was achieved simply by inputing a triangular signal to the system. All tests were performed at 23.5° C and 60% r.h. The rates of deformation for tension tests were 0.08 mm sec^{-1} (strain rate = 0.2% sec^{-1}) and 0.4 mm sec^{-1} (strain rate = 1% sec^{-1}), and that for shear was 0.006 mm sec^{-1} (strain rate = 0.3% sec^{-1}). Maximum displacement of the instrument was 5 mm, and maximum force was 5 kg. Consequently, the maximum strain in a typical 40 mm tension sample was only 12.5%. Most samples did not break at 12.5% strain. Hence, stress–strain curves to break were obtained with samples of 25 mm lengths.

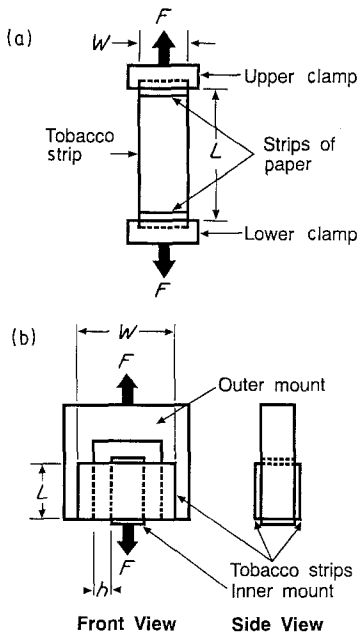

Figure 4 Schematic diagram of (a) tension and (b) shear samples.

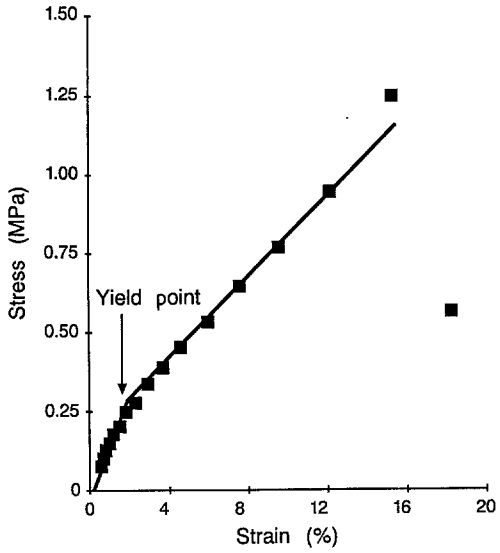

Figure 5 Tensile stress–strain behaviour of bright lamina tested at the strain rate of 0.2% sec^{-1}.

The stress and strain created in the tobacco specimen when the load was applied were calculated from the force and displacement measurements by assuming that the stress and strain were distributed uniformly throughout the sample, and edge effects were negligible. Calculations were done internally in the Dynastat by using the following formulae.

Let F be the force exerted on the tobacco sample. The normal stress, σ, and shear stress, τ, are

$$\sigma = F/Wt \text{ (tension)} \quad (1)$$

$$\tau = F/4Lt \text{ (shear)} \quad (2)$$

For small displacements, δ, the normal strain, ε, and shear strain, γ, are:

$$\varepsilon = \delta/L \text{ (tension)} \quad (3)$$

$$\gamma = \delta/h \text{ (shear)} \quad (4)$$

Note that the expression for shear strain is also good under finite strain [23]. From Equations 1 to 4, the expressions for the Young's Modulus, E, and shear modulus, G, can be written as

$$E = \sigma/\varepsilon = FL/Wt\delta \quad (5)$$

$$G = \tau/\gamma = Fh/4Lt\delta \quad (6)$$

Lamina thickness was measured with a dial gauge. Instrument resolution was 0.005 mm. Because the lamina thickness was not uniform, an average of four location thicknesses was used as the sample thickness.

5. Results and discussion

5.1. Determining the relationship between morphology and mechanical properties

Figure 5 is a typical stress–strain curve for a bright lamina sample deformed in tension. The curve is representative of several samples. All samples were 25 mm long and were deformed at a strain rate of 0.2% sec^{-1}. It can be seen from the figure that the material was nonlinear and became softer with increasing strain. Samples did not show any region of plastic deformation or the yielding associated with it. No reduction in area, or necking, was noticed. However, there appeared to be a noticeable change in the slope of the curve at about 2% strain. Additionally, it seemed that material behaviour could be divided into

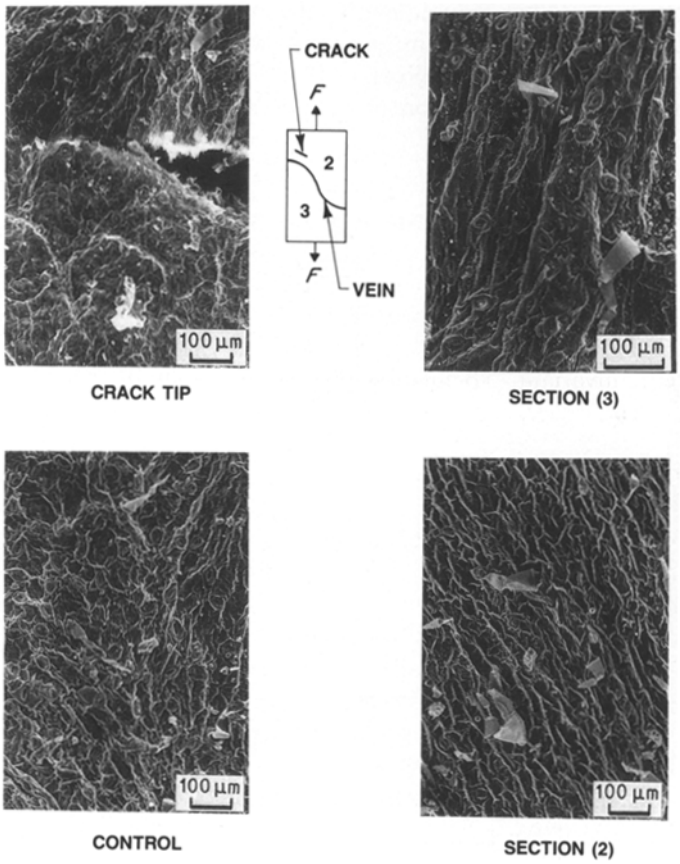

Figure 6 Scanning electron micrographs of a sample deformed in tension.

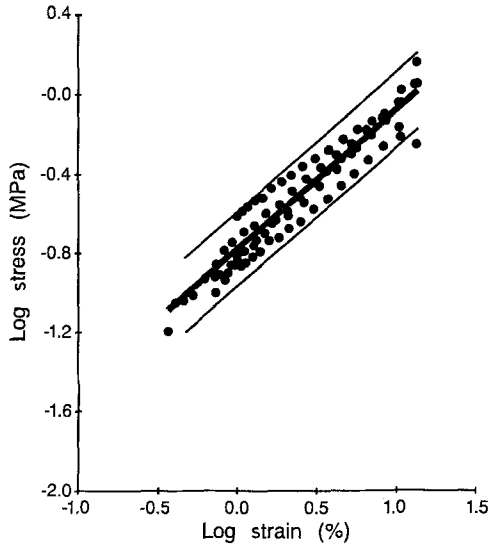

Figure 7 Tensile stress–strain data for samples cut normal to the lateral veins from mid-leaf position. Thick line is a best-fit regression line and thin lines are the 95% confidence limit on all data points.

two regions. Below 2% strain, the lamina can be approximated by a linear law, and above 2% by another linear expression. These two linear approximations were represented by straight lines shown in Fig. 5; 2% strain was the point of their intersection. Using this as a criterion, we define the 2% strain to be the onset of yielding in stretching of tobacco lamina and correspondingly, 0.3 MPa as the yield strength. This phenomenon will be addressed in detail in later work. Most of the bright laminar samples broke at approximately 16% strain.

In order to determine deformation details at the microlevel, a few stretched samples were examined under a scanning electron microscope. Figure 6 shows scanning electron micrographs of four locations taken from a stretched sample. Clearly, both stress distribution and deformation were nonuniform. In certain regions, presumably of high stress, cell walls seemed to elongate in the direction of loading. Notice how the presence of a vein distorted the cell orientation and stress distribution. This figure indicates that at the microscopic level the lamina was mechanically anisotropic and inhomogeneous. Nevertheless, its stress-strain curve was reasonably smooth and similar to many polymers [16, 24, 25].

The effects of sample orientation and location were determined next. To delineate the differences properly, it was necessary to minimize measurement errors. One obvious source of error was the edge effect due to sample length being small. For this reason, the data in the next four figures were obtained on 40 mm or longer specimens. Because of the instrument limits, the maximum strains were only 12.5% or less. Consequently, none of the samples broke. The normal stress–strain data of five samples cut perpendicular to the lateral veins from mid-leaf position are shown in Fig. 7. Data were plotted on a log–log scale. It can be seen from Fig. 7 that up to 12.5% strain the data could be fitted well linearly on a log–log scale, implying a power law relationship between the stress and strain. Thus, mathematically the curve could be represented

TABLE II Coefficients for stress–strain lines

	Mid-leaf		Base	Tip
	Normal	Parallel		
ln (a)	−1.70	−1.65	−1.72	−1.77
b	0.744	0.609	0.717	0.736
S.D.	0.13	0.04	0.15	0.19

as

$$\log\sigma = \log a + b\log\varepsilon \tag{7}$$

or

$$\sigma = a\varepsilon^b \tag{8}$$

where a represents the stress at 1% strain and b the power law exponent indicating the rate of increase of the stress relative to increase in strain. Both a and b characterize the mechanical properties of the lamina and could be used to quantitatively determine the effects of sample orientation and location.

In Fig. 7, the solid line represents the best fit regression line for all samples; the dashed lines are the 95% confidence limit lines on all data points. There seemed to be some scattering in results from sample to sample, indicating that no two samples are alike even when taken from the same location and orientation. However, the trends were the same for all of them. Each of them could be modelled well by a power law constitutive equation. This sample-to-sample variation is an inherent property of most natural materials [1, 2, 8–10]. This implies that to obtain a representative response for a natural material, several samples need to be tested. Also, the results must be treated statistically. Table II shows the mean values of the coefficients a and b and the corresponding standard deviation of data for five samples. The means and standard deviations of a and b, obtained for samples from different locations and orientations, will be used to investigate lamina in-plane isotropy and homogeneity.

To determine the effect of orientation, similar stress–strain tests were performed on samples taken parallel to the lateral veins and from the middle of the leaves. The statistical values of parameters a and b are given in Table II. The values of a and b were within the statistical variation of samples taken normal to the lateral veins. This implies that the in-plane properties of the lamina at the middle of a leaf were direction invariant. To analyse this result, recall the lamina microstructure shown in Figs 1 and 2. In the plane of a leaf, all four types of cell were arranged uniformly throughout the leaf. This directionally uniform cell arrangement seemed to be giving the above result. Because of the random distribution of voids and veins at the microscale, their cumulative effect at the macroscopic level appeared uniform in all directions. As the cellular morphology is similar throughout the lamina, it could be assumed that, statistically, sample orientation has no effect on the mechanical behaviour of the lamina at the macroscale, irrespective of sample location. From these observations, it can be hypothesized that all dicot leaves will have directionally uniform mechanical properties and those of monocots will depend on sample orientation.

The effects of sample location were obtained next. Stress–strain tests were done on samples taken from the base and tip of the leaves, respectively. The means and standard deviations are presented in Table II. Statistically, the data compared well with those of mid-leaf samples, indicating the location homogeneity of the lamina mechanical characteristics.

Because none of the above samples broke, they were retested after adjusting their lengths. Even in the second trial, only a few broke. Unbroken samples were retested until they did break. It should be noted that during the time required to remount a sample and start the test, all samples recovered a part of their stretch. This suggests a viscoelastic nature of the lamina. In a paper to be published later, viscoelastic behaviour of the lamina will be described in detail [22, 26]. All retested data could also be fitted well to a straight line on a log–log scale. They showed similar statistical variations. Orientation as well as location effects were negligible. In every retesting, a marginal strain softening was noticed for most of the samples. This verifies the earlier observation of Fig. 5.

The above results showed that the variation in mechanical properties due to sample location and orientation was negligible. As stated earlier, however, precautions must be taken to avoid any major vein in the sample preparation. Hence it can be concluded that at the macroscale, tobacco lamina is in-plane homogeneous and isotropic. Also, a material could be anisotropic and inhomogeneous at the microscopic level and exhibit uniformity on the macroscale. Of course, this is true only in the statistical sense. A practical advantage of this finding is that one can study the effects of test variables, such as strain rate, shear-tension, dynamic loading, etc., and environmental variables, such as relative humidity and temperature, without concern for sample location and orientation. One needs to test many similar samples without regard to location and orientation and determine properties in the statistical sense.

5.2. Investigating effects of rate and shear-tension processing parameters

The effect of strain rate was examined next. Figure 8 is a stress–strain curve for samples stretched at the strain rate of 0.2 and 1% sec^{-1}. The major difference between the two was that the lamina stiffness increased, but the strain-to-break decreased with increase in strain rate. This rate effect is similar to that observed in most polymers [24, 25]. The results indicate that the mechanical behaviour of tobacco lamina needs to be studied using the theory of viscoelasticity.

Lamina behaviour in the shear deformation was rather interesting. A comparison of shear and tension responses is shown in Fig. 9. Qualitatively, material behaviour was similar in both modes of deformation. The subtle difference between the two was in their toughness and strain-to-break. The lamina was about 30% stiffer in tension than in shear. The strain-to-break in shear was much higher (35% compared to 16%) than in tension. As a result, bright lamina was approximately twice as tough in shear as in tension. Using elasticity theory, the Poisson's ratio for lamina

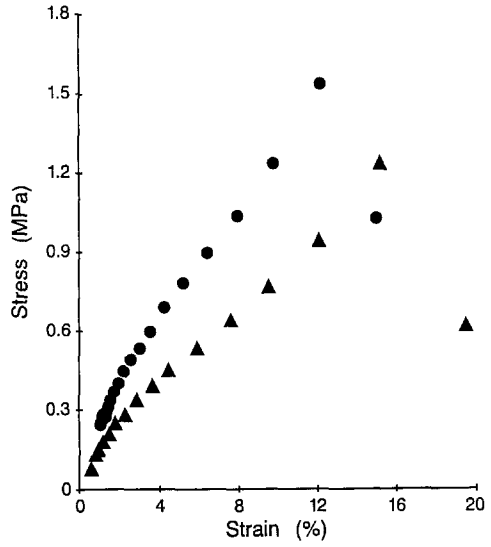

Figure 8 Effect of the strain rate on the stress–strain behaviour of bright lamina in tension. Strain rates (●) 1% sec^{-1}, (▲) 0.2% sec^{-1}.

can be determined by using the following formula [27]

$$E = 2G(1 + \nu) \tag{9}$$

where E and G are the tension and shear moduli and ν the Poisson's ratio. The Poisson's ratio is defined as the ratio of lateral strain over axial strain. In short, it is a measure of the change in the lateral dimension of a sample during an axial test. Substituting E = 14 MPa and G = 9.2 MPa, for strain less than 2%, we get $\nu = -0.2$, implying the increase in sample lateral dimensions during a tension test. The visual observation did not give such an impression. This phenomenon will be examined later.

5.3. Comparing mechanical properties of different types of laminae

For this study, both bright and burley lamina materials were obtained from the Manufacturing Center of Philip Morris USA, Richmond, Virginia. Each type of tobacco included the highest and the lowest yield laminae. In the subsequent discussion, these will be referred to as the high- and low-yield materials. The term yield implies the percentage of material not

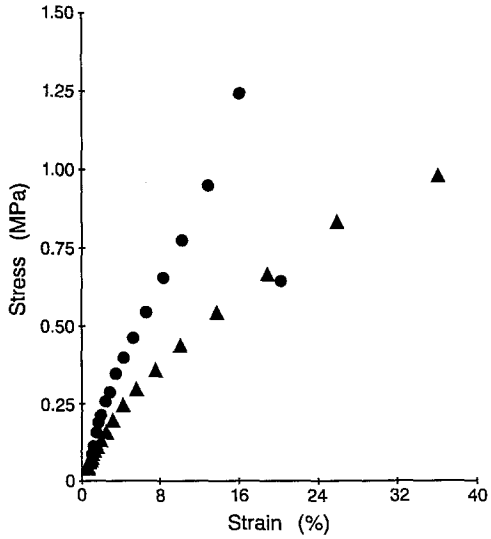

Figure 9 Comparison of the stress–strain behaviour of bright lamina in tension and shear deformations. (●) Tension, (▲) shear.

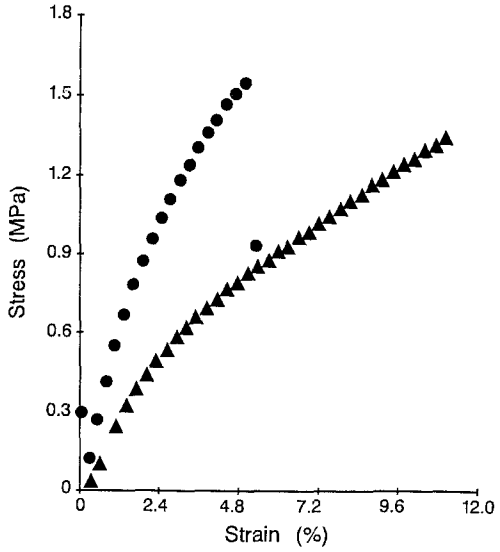

Figure 10 Comparison of the stress–strain behaviour of (▲) high- and (●) low-yield bright lamina.

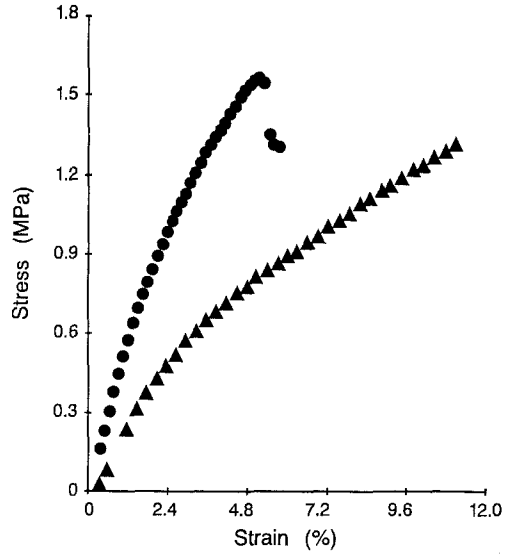

Figure 12 Comparison of the stress–strain behaviour of the high-yield (●) bright and (▲) burley laminae.

broken into unusable small pieces and dust. Tension tests were run on many samples of both classes of bright and burley laminas. Test conditions were similar to that of low strain rate tests of the previous study.

Figures 10 and 11 are the stress–strain curves of both classes of bright and burley laminae. All four curves were representative of many samples of the respective types of lamina. It can be seen that for both types of tobacco, high- and low-yield materials were mechanically different. Low-yield material was stiffer, but less extensible than high-yield lamina. Representative initial Young's moduli of high-yield samples were 50 MPa (burley) and 30 MPa (bright), and those of low-yield were 80 MPa (burley) and 50 MPa (bright). For both types of tobacco, low-yield samples broke at about half the strain of high-yield material (5.5% compared to 12% for bright and 2.5% compared to 5% for burley).

Figure 12 is a comparison of the stress–strain curves of high-yield bright and burley laminas. Figure 13 is a similar comparison of low-yield materials. For each case, there were significant differences in mechanical properties of bright and burley laminae. Burley was always stiffer, but less extensible than bright. Its breaking strain was about half of that for the other.

The above results indicated that the mechanical properties of bright lamina differed from those of burley. There was a wide spread in properties of high- and low-yield laminae of both bright and burley tobacco. Interestingly, the stress–strain behaviour of all four types of lamina seemed consistent with the material yields observed in the manufacturing of cigarettes. Thus, a simple stress–strain test could give a qualitative comparison of material yield during processing.

6. Conclusions

The results of this study showed that, on a microscale, tobacco lamina was anisotropic and inhomogeneous, but its inplane phenomenological mechanical properties were invariant with sample location and orientation. This implies that a material can be anisotropic and inhomogeneous at the microscopic level and

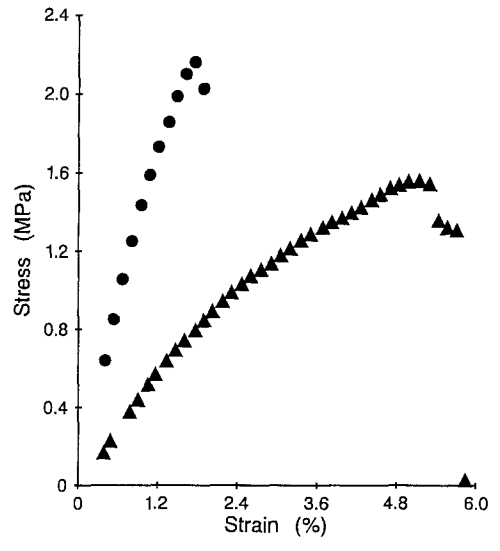

Figure 11 Comparison of the stress–strain behaviour of (▲) high- and (●) low-yield burley lamina.

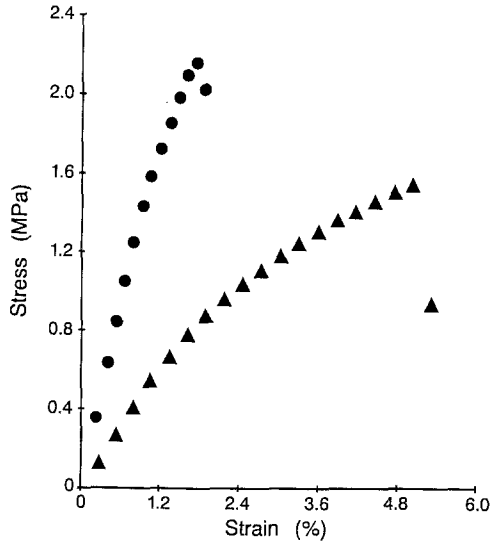

Figure 13 Comparison of the stress–strain behaviour of the low-yield (▲) bright and (●) burley laminae.

exhibit uniformity on the macroscale. Thus, the effects of processing parameters on mechanical behaviour of lamina can be examined without undue concern for sample location and orientation. However, due to sample-to-sample variability, many samples need to be tested to determine material properties with adequate statistics. We also described how a knowledge of lamina microstructure can help to generalize and develop a better understanding of the macroscopic observations.

The stress–strain data indicated that tobacco lamina mechanical behaviour was nonlinear with it becoming softer with increasing strain. Additionally, material was softer but tougher in shear than in tension. Like many polymers, the lamina became stiffer and strain-to-break decreased with the increase in strain rate. Burley lamina was stiffer and broke at lower strain than bright. Interestingly, lower yield materials also had lower breaking strain and higher stiffness than higher yield lamina. This shows that a simple stress–strain test can provide a qualitative comparison of material yield during processing.

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