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Mechanical characterization of juvenile European aspen (*Populus tremula*) and hybrid aspen (*Populus tremula* × *Populus tremuloides*) using full-field strain measurements

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Abstract Functional analysis of genes and proteins involved in wood formation and fiber properties often involves phenotyping saplings of transgenic trees. The objective of the present study was to develop a tensile test method for small green samples from saplings, and to compare mechanical properties of juvenile European aspen (*Populus tremula*) and hybrid aspen (*Populus tremula* × *tremuloides*). Small microtomed sections were manufactured and successfully tested in tension parallel to fiber orientation. Strain was determined by digital speckle photography. Results showed significantly lower values for juvenile hybrid aspen in both Young's modulus and tensile strength parallel to the grain. Average Young's moduli spanned the ranges of 5.9–6.6 and 4.8–6.0 GPa for European aspen and hybrid aspen, respectively. Tensile strength was in the range of 45–49 MPa for European aspen and 32–45 MPa for hybrid aspen. The average density (oven-dry) was 284 kg/m³ for European aspen and 221 kg/m³ for hybrid aspen. Differences in mechanical properties correlated with differences in density.

Key words Hybrid aspen · Juvenile · *Populus tremula* × *tremuloides* · Tensile strength · Young's modulus

Introduction

The possibilities of accelerated domestication of trees and enforced improvement of wood fibers and cell wall composition are under investigation. The rapid progress in identification and functional analysis of genes and proteins involved in wood formation and wood fiber properties has opened the perspective for novel approaches in tree breeding using molecular markers and/or transgenic technology.¹ *Populus* is used as a major model tree in this research.^{2–3} Gene function can be investigated by down-regulating or up-regulating mRNA availability by transgene technology. Ideally, we should also completely understand the implication of these modifications for aspects important in the final product, such as mechanical performance. The objective of this study was therefore to develop a test procedure for small hardwood samples, and to determine and compare the mechanical properties of juvenile European aspen and hybrid aspen. Experiments focused on measuring Young's modulus (E_A^*) and tensile strength (σ^*) parallel to grain.

Mechanical testing on small samples of poplars and hybrids with altered wood properties has rarely been done. Kasal et al.⁶ worked out a number of methods to test the mechanical properties of stems of 1-year-old trees, and performed experiments on control and transgenic samples of quaking aspen (*Populus tremuloides*). Coutand et al.⁷ performed three-point bending tests on small stripes of 1-year-old poplar trees (*Populus* cv. I4551), and found that tension wood showed higher modulus of elasticity (MOE) than normal wood. De Boever et al.⁸ performed tensile tests in the longitudinal direction on microtome sections of juvenile poplar trees (*Populus nigra* L.) that had been exposed to enhanced carbon dioxide (CO₂) concentrations during growth. They found that Young's modulus and tensile strength parallel to the grain increased significantly with elevated levels of CO₂. Schwab et al.⁹ examined Young's modulus, tensile strength, and compression strength parallel to the grain on specimens from logs of control and transgenic hybrid aspen trees in green condition, and found a relation between higher mechanical properties and higher

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density. The work presented here is the first known to determine the mechanical properties in the longitudinal direction of material originating from the innermost growth ring of juvenile European and hybrid aspen. This is interesting due to the potential to interpret the effects from cell wall composition on mechanical properties in wood from transgenic trees grown for just a few months.

Materials and methods

Seven saplings were selected, of which three were European aspen, all from a natural stand and the same locality. These were roughly 15 months old. The remaining four saplings were hybrid aspen that had been grown in greenhouses for 2 months before harvest. One sample per tree was cut at a height of approximately 200 mm, and placed individually in sealable test tubes for storage in damp conditions at 4°C in order to keep the material in a green condition until the tensile tests. All seven samples had a diameter of 10–14 mm (including the bark) and a length of 80–100 mm. The specimens were prepared by removing the cortex and cutting microtome sections from the samples.¹⁰ The microtome sections were then placed in deionized water. The distance between specimen location and the marrow varied between 0.7 and 2.5 mm. Consequently, microtomed sections from the European aspen material only included material from the innermost growth ring. Dimensions of specimens, 0.2 (R) × 4 (T) × 40 mm (L), were measured using a slide caliper. The actual width in the area of reduced cross section (2 mm) was cut to final dimensions with a scalpel (Fig. 1).

Density was measured on specimens of similar size and shape, being derived from the same greenhouse population and natural stand respectively, as the ones used in the tensile tests. Wood density (ρ^*) was based on oven-dry mass and volume. Specimens were also dried from green to air-dry condition during continuous registration of weight, and under similar humidity conditions as during tensile tests in order to estimate the drying rate of specimens during the tensile tests.

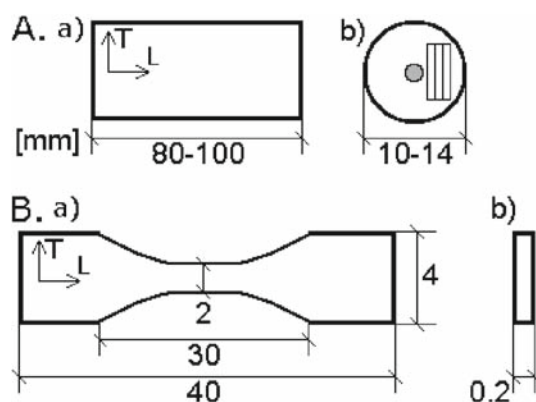


Fig. 1. A Orientation of microtome slices in samples. *a*, Side view; *b*, front view. B Specimen dimensions. *a*, Side view; *b*, front view

The project focused on measuring Young's modulus (E_A^*) and tensile strength (σ^*) in quasi-static tension parallel to the grain. A total number of 22 European aspen and 40 hybrid aspen specimens were tested, and the tensile tests were performed with a mini materials tester MiniMat 2000 (Rheometric Scientific) with a 20-N load cell. The specimens were clamped in manually tightened grips with rough surfaces to prevent slipping of the specimen during the test. The distance between grips was 30 mm (Fig. 2). The MiniMat system recorded the load (F) at a rate of 2.8 measurements per second. Displacement-controlled testing was performed with a crosshead speed of 0.5 mm/min. The average time for a specimen tested in the MiniMat was about 130 s. A CCD camera (two-dimensional analysis) registered the visible area of the specimen, and the ARAMIS software (developed by Gom, Germany) was programmed to take one photograph per second. Additional characterization of the fracture surface was done directly after tensile testing using a light microscope.

The digital speckle photography (DSP) technique is a widely used nondestructive method for measuring two-dimensional or three-dimensional surface deformations in materials, such as wood.^{11,12} The procedure is to visually identify a pattern on a surface. The surface is recorded before and during displacement, and strain calculations can be made by correlating the patterns of the surface in the unstrained and (to a different degree) strained stages. The technique requires a clearly recognizable pattern on the specimen in order to register displacement. This cannot always be provided by the microstructure of the wood.¹³ In order to apply a distinct pattern to the originally white surface of the specimen, a black high-contrast dot pattern was sprayed onto the surface. A spotlight was used to light up the sample and enhance the contrast. The ARAMIS software calculates the strain field by differentiating the displacement (δ) in the direction monitored by the camera. The specimen was meshed automatically into small facets by ARAMIS. An area measuring approximately 1 × 8 mm of the reduced cross-sectional area of the specimen was manually selected for strain calculations. Information about

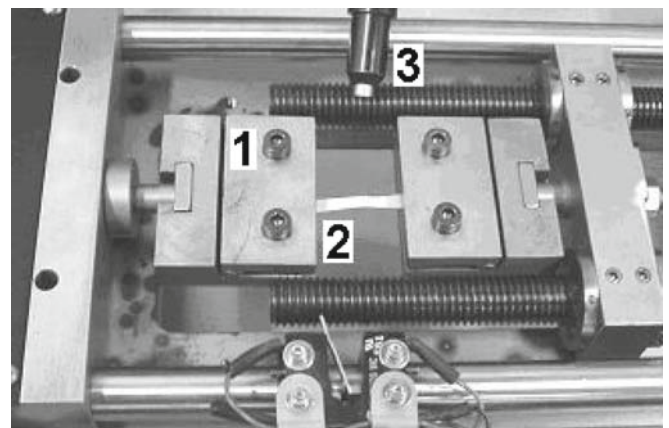


Fig. 2. Experimental setup. 1, Grips; 2, specimen; 3, spotlight. Distance between grips is 30 mm

the strain was obtained by averaging the facet strain data in the chosen area and in the longitudinal direction of the specimen. Facet size and distance between facet loci were 17×17 and 7 pixels, respectively, which correspond to approximately 0.38×0.38 mm and 0.16 mm on the specimen. Young's modulus E_A^* was calculated from the initial linear part of the stress–strain curve, and stress σ^* was obtained by simply dividing the force F by the cross-sectional area. Because the MiniMat system alone can register displacement from movement of the grips, additional tensile tests were also performed in order to compare strain values for the MiniMat system and the DSP, respectively. The results showed that strain estimations were about 20% higher for the MiniMat system when compared with DSP data due to unavoidable play and grip sliding in the mechanical equipment of the former. This technical problem could be solved because the used strain data from the DSP originated from the specimen surface alone. In this context, the application of full-field strain measurements in the form of DSP proved to be particularly important in producing more accurate data. Both the preparation and testing of the specimens were considered successful.

Results

The preparation of small specimens from juvenile wood was successfully carried out using the microtome cutting technique. In the mechanical testing step, the use of full-field strain measurements provided an advantage with respect to the quality of the data. A concern with small specimens is usually any effect of gripping constraints on estimates of E_A^* and σ^* . Because strains were measured by DSP and the meshing area was limited to that of reduced cross-sectional area on the specimens, strains occurring closest to the grips did not influence the final evaluations. Moreover, drying of specimens during mechanical testing was considered to be negligible.

Table 1 shows E_A^* and σ^* for European aspen (*Populus tremula*) and hybrid aspen (*Populus tremula* \times *tremuloides*) in the green state. Both E_A^* and σ^* was higher for the European aspen than for hybrid aspen. Average moduli spanned the range 5.9–6.6 GPa for European aspen and 4.8–6.0 GPa

Table 1. Wood properties of juvenile European aspen and hybrid aspen in green condition

Species	Sample no.	Young's modulus (GPa)	Tensile strength (MPa)
European aspen	1	6.6 (1.1)	49 (3)
	2	5.9 (1.2)	46 (8)
	3	6.1 (1.6)	45 (13)
	Average	6.2 (1.3)	47 (9)
Hybrid aspen	1	5.4 (0.9)	45 (7)
	2	6.0 (1.1)	39 (10)
	3	4.8 (0.6)	34 (3)
	4	5.6 (0.9)	32 (5)
	Average	5.5 (0.9)	38 (8)

Values in parentheses represent standard deviations

for hybrid aspen. Tensile strength σ^* was in the range 45–49 MPa for European aspen and 32–45 MPa for hybrid aspen. The lower mechanical properties of hybrid aspen correlate with lower density ρ^* (Table 2). The average European aspen density is 284 kg/m³ while hybrid aspen averages 221 kg/m³. The differences in E_A^* , σ^* , and ρ^* between species were highly significant. A Student's *t*-test analysis provided significance levels of 99.5%, 98.8%, and 100%, respectively.

Optical microscopy of juvenile wood cross sections revealed the difference in structure between trees from the two species (Fig. 3). The diameter of the European aspen was of about the same size as for the hybrid aspen, in spite of the fact that it had grown for a much longer period (approximately 15 and 2 months, respectively). Values of E_A^* of specimens with the corresponding position in the sample were generally higher for the European aspen than for the hybrid aspen (Fig. 4). With both species there is a slight increasing trend in modulus with radial distance (r). Results for the European aspen display a wider range of values than the hybrid aspen. Tensile strength σ^* also increased with r (Fig. 5). The level was generally lower in the hybrid aspen.

The stress–strain curve in Fig. 6 displays the typical mechanical behavior of European and hybrid aspen loaded in tension. The curves are initially linear, but gradually enter a nonlinear region. Both species are initially dominated by elastic behavior, followed by a plastic deformation region where the material is deformed irreversibly before fracture. This was confirmed during analysis of DSP images, where part of the strain field remained after unloading of specimens. Stress–strain curves also revealed lower values of strain-to-failure (ϵ^*) for hybrid aspen. The European aspen strain field pattern showed less uniform distribution than the hybrid aspen (Fig. 7). For European aspen, local strains exceeded 2% to a significant extent already at stage C (red color).

Table 2. Comparison of density from samples of juvenile European aspen and hybrid aspen in oven-dry condition

Species	Sample no.	Density (kg/m ³)
European aspen	i	294
	ii	298
	iii	277
	iv	274
	v	284
	vi	279
	Average	284 (10)
Hybrid aspen	i	204
	ii	220
	iii	246
	iv	224
	v	225
	vi	205
	vii	207
	viii	217
	ix	240
	Average	221 (15)

Values in parentheses represent standard deviations

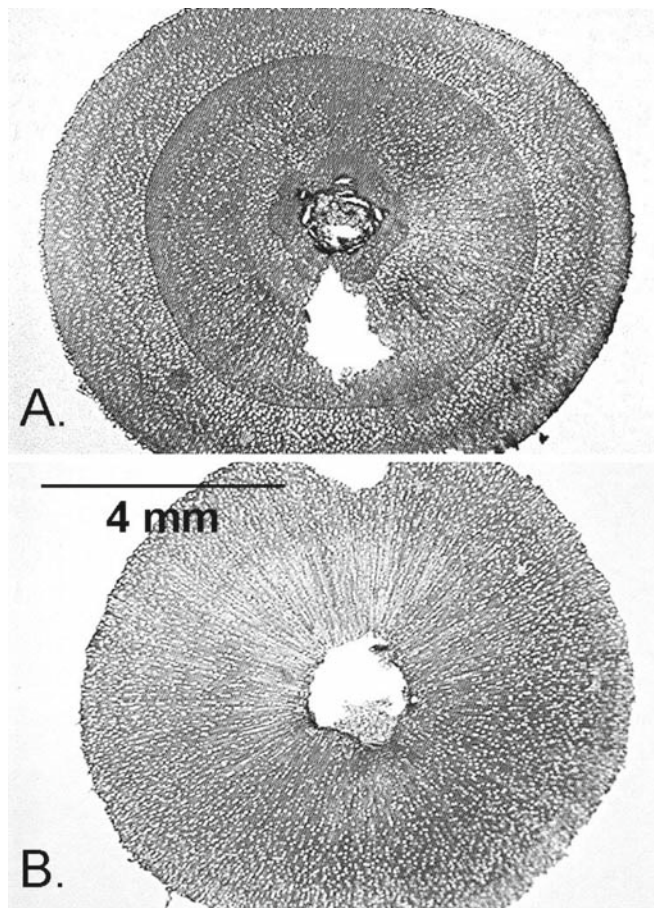


Fig. 3. Cross section of juvenile European aspen (A) and hybrid aspen (B). Note annual ring in European aspen. Bark is excluded on both samples

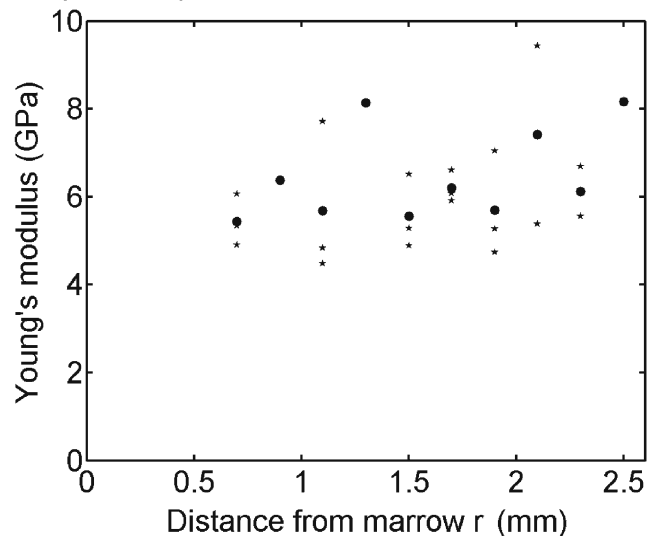
Discussion

Experimental results of E_A^* and ρ^* can be confirmed using a model presented by Gibson and Ashby,¹⁴ which is based on an idealized two-dimensional honeycomb structure of wood. When a wooden material is loaded in the axial direction, the only type of deformation is stretching of the cell walls in the solid honeycomb material. A rule-of-mixtures type of model is therefore applied where the relative modulus (E_A^*/E_s) in the cell (E_s is the axial modulus of the solid) scales linearly with the relative density (ρ^*/ρ_s) where ρ_s is the density of the cell wall;

$$\frac{E_A^*}{E_s} = C \left(\frac{\rho^*}{\rho_s} \right) \quad (1)$$

C is an empirical constant meant to compensate for deviations in real wood material compared with assumptions in the model (here set to $C = 1$). Cell wall density ρ_s is virtually the same for all wooden species, and can be used in combination with experimental results of E_A^* and ρ^* to estimate E_s and compare the results with previous studies. Measured density ρ^* was converted for evaluation of a material in

European aspen



Hybrid aspen

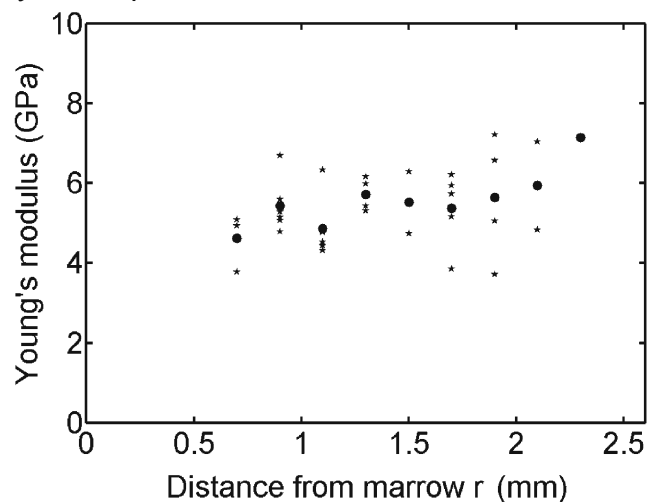


Fig. 4. Young's modulus related to specimen distance from marrow in juvenile European aspen (upper) and hybrid aspen (lower). Asterisks, data; dots, mean values

green condition during calculations¹⁵ assuming a moisture content of 28%. Density in the green material $\rho_s = 1300 \text{ kg/m}^3$ was based on cell wall density values of 1500 kg/m^3 in dry material.¹⁴ Altogether this resulted in approximate values for axial modulus of the solid E_s of 24 and 27 GPa for European and hybrid aspen, respectively, which are fully comparable with a number of hardwood species for which E_s spans the range of 17–36 GPa.^{16–18}

In comparing mechanical properties and densities of species, features and behavior of American and European aspen are similar to those of hybrid aspen and therefore are of interest. Young's modulus E_A^* for fully grown American aspen¹⁶ is 8.9 GPa, while the corresponding value for European aspen¹⁹ is 8.3 or 8.7 GPa depending on the method used (acoustic measurements and tensile tests, respectively). Tensile strength parallel to the grain (σ^*) for fully grown European and hybrid aspen spans the ranges of 64–103 and

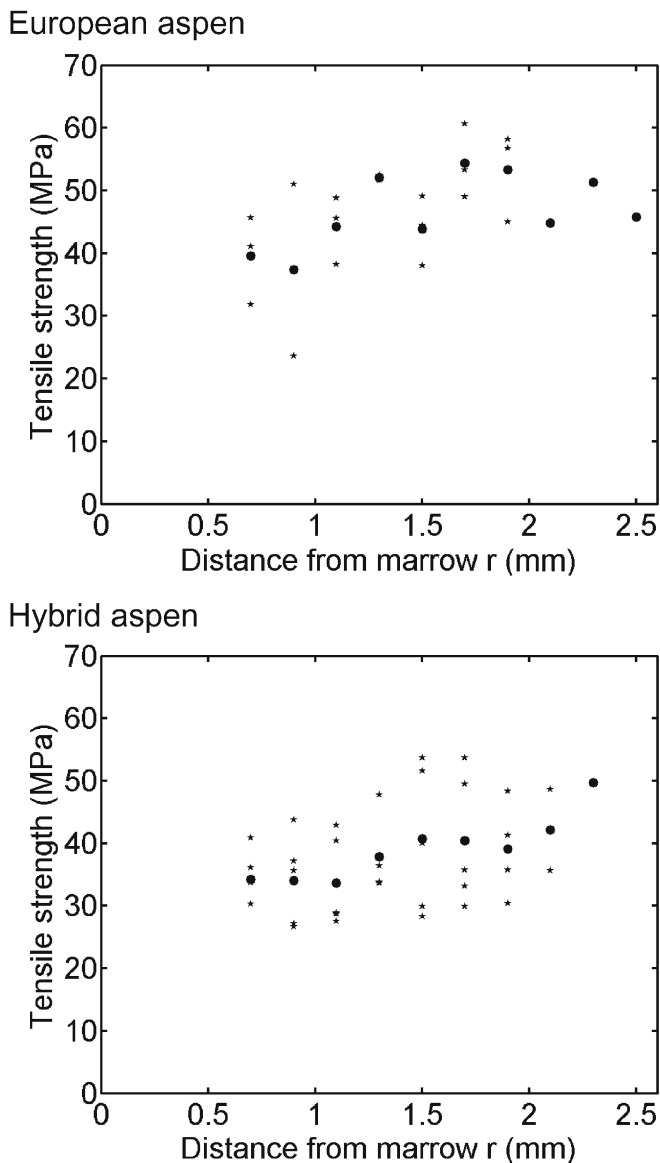


Fig. 5. Tensile strength related to specimen distance from marrow in juvenile European aspen (*upper*) and hybrid aspen (*lower*). Asterisks, data; dots, mean values

58–93 MPa, respectively, (depending on the drying method used).²⁰ The corresponding value for American aspen could not be found. Heräjärvi and Junkkonen²¹ stated that basic density for full grown hybrid and European aspen grown in Finland was 363 and 376 kg/m³, respectively. A study on European aspen grown in Sweden²² and Norway presented air-dry density values of 423 and 440 kg/m³, respectively. A value presented by Tsoumis²³ of air-dry density for European aspen was found to be higher than that for American aspen (460 and 380 kg/m³, respectively); the latter value corresponding well to observations made by Lang et al.²⁴ Recalculation²³ of these results (assuming moisture contents of 28% and 12% for basic and air-dry density, respectively) renders oven-dry density values of approximately 300 kg/m³ (hybrid aspen), 320, 390, 410, and 430 kg/m³ (European aspen) and 350 kg/m³ (American aspen).

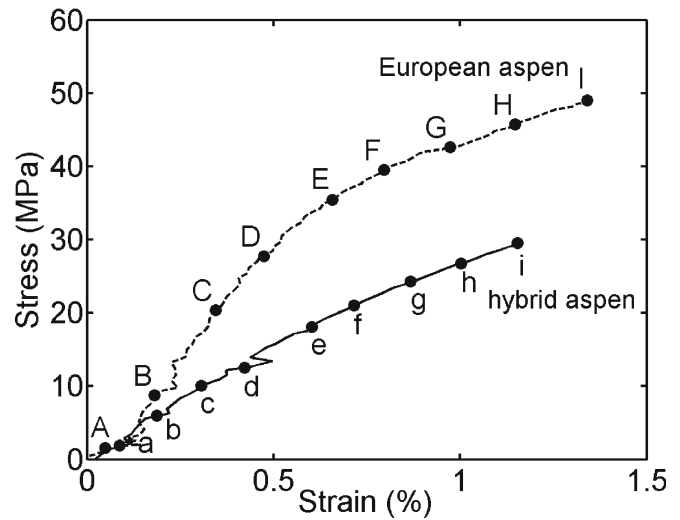


Fig. 6. Stress–strain curves for juvenile European aspen (*uppercase*) and hybrid aspen (*lowercase*). Uppercase and lowercase letters refer to corresponding strain stages shown in Fig. 7

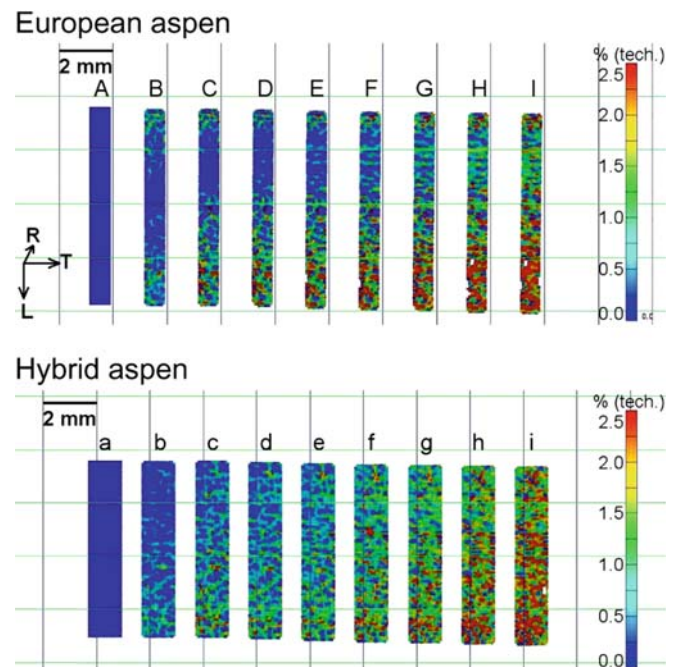


Fig. 7. Digital speckle photography strain fields at different stages of global strain for juvenile European aspen (*upper*) and hybrid aspen (*lower*) in tension parallel to grain

To sum up, values of E_A^* and ρ^* for American aspen are in the same range as for European aspen, while values of σ^* and ρ^* for European aspen are higher than those for hybrid aspen. Inferior mechanical behavior and low density are known to be closely related. This has also been shown for European aspen alone,²⁵ European aspen in comparison with hybrid aspen,²⁶ and hybrid aspen in comparison with transgenic hybrid aspen.⁹

Compared with mature wood, juvenile wood is characterized by wider annual rings, shorter fibers, larger micro-

fibril angle (MFA), and relatively lower mechanical properties.²⁷ This holds true also for European and hybrid aspen, for which density and wood properties increase markedly as a function of the distance from the marrow.^{21,26} Kasal et al.⁶ found that values for E_A^* and σ^* of 1-year-old small-diameter (3–10 mm) air-dry American aspen samples were 8.2 GPa and 41.3 MPa, respectively, which are in line with the results presented here. One important aspect that must be taken into account is the fact that tests were performed on green material. In the absence of water, microfibrils are able to draw closer together, which leads to stronger attractive forces between cellulose chains. When moisture is reduced, strength and stiffness increase, and vice versa.²³ In this case, not only the juvenescence but also the presence of water in test specimens are explanations of the low values of E_A^* and σ^* for both European and hybrid aspen, compared with results presented previously in the text.

Values for both E_A^* and σ^* increased with distance from the marrow for both species, while the trend was more ambiguous for European aspen than for hybrid aspen (Figs. 4 and 5). Cown and Parker²⁸ displayed the intraring density profile for American aspen, which clearly showed an initial increase in ρ^* from the inner part to the outer part of the growth ring. The current results, indicating increasing mechanical properties with distance from the marrow in one specific growth ring, are in line with this and other studies on radial profiles of diffuse porous (DP) species.²³ It is likely that MFA also decreases with distance from the marrow.

Results displayed a wider range of values for both E_A^* and σ^* for European aspen (Figs. 4 and 5), which was probably due to a less homogeneous structure of these saplings, given that data are sensitive to position in the growth ring and the corresponding local density. European aspen saplings originated from a natural stand growing under uncontrolled conditions, which also increases the difference between individuals.

Difference in fracture behavior between European aspen and the hybrid was observed. DP hardwoods do not exhibit any growth rings clearly visible to the naked eye, but the apparently homogenous structure can still conceal differences in mechanical properties within a single growth ring. Koch²⁹ states that tensile failure in hardwoods parallel to the grain results from breaking fiber walls. In cells having a thick S2 layer, tension produces intrawall failure that follows the S2 fibrillar angle. In cells with lesser S2 cell wall area, abrupt failures may be found. Thus, the latewood zone typically shows a combination of tension and shear failure, while in earlywood failure takes the form of brittle tension fracture. The high rate of splintering tension in European aspen and brittle tension in hybrid aspen was significant. This is also in line with the fact that the content of latewood was higher in the specimens of European aspen than in the hybrid, due to the presence of a complete growth ring in the former. Division of characterizing fracture surfaces was done outgoing from Bodig and Jayne¹⁶ (see Table 3).

Finally, local failure appears more heterogeneously distributed in European aspen, as interpreted from the DSP

Table 3. Characterization of fracture behavior from fracture surfaces on specimens of juvenile European aspen and hybrid aspen

Species	Splintering tension (%)	Combination tension-shear (%)	Diagonal shear (%)	Brittle tension (%)
European aspen	50	0	13	37
Hybrid aspen	5	15	10	70

images. The more heterogeneous structure in the European aspen mentioned previously can be one possible explanation for this behavior, although further investigation on the subject is needed.

Conclusions

A method for preparation and mechanical testing of green specimens with dimensions of 0.2 (R) × 4 (T) × 40 mm (L) was developed. The application of full-field strain measurements in the form of DSP was particularly important, in order to produce accurate data for stiffness parameters. Results showed significantly higher Young's modulus E_A^* and tensile strength σ^* for European aspen than for hybrid aspen, primarily due to the higher density ρ^* of the former. Results also displayed increasing mechanical properties with increasing distance from the pith, within a single growth ring. This is in line with previous studies. A study of cross sections revealed morphological differences between the species, probably originating from differences in growth rate. Full-field strain measurements performed using the DSP technique showed that the strain field pattern on European aspen specimens had a less uniform distribution than that of the hybrid aspen. This behavior might be explained by the more homogenous structure of the former.

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