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The fracture toughness and properties of thermally modified beech and ash at different moisture contents

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Abstract The fracture toughness of thermally modified beech (*Fagus sylvatica* L) and ash (*Fraxinus excelsior* L) wood under Mode I loading was quantified using Compact Tension (CT) specimens, loaded under steady-state crack propagation conditions. The influence of three heat-treatment levels and three moisture contents, as well as two crack propagation systems (RL and TL) was studied. Complete load–displacement records were analysed, and the initial slope, k_{init}, critical stress intensity factor, K_{Ic}, and specific fracture energy, G_f, evaluated. In the case of both species, thermal modification was found to be significantly affect the material behaviour; the more severe the thermal treatment, the lower the values of K_{Ic} and G_f, with less difference being observed between the most severe treatments. Moisture content was also found to influence fracture toughness, but had a much less significant effect than the heat treatment.

Introduction

Thermal modification, or heat treatment, of wood is now an established alternative to other preservative treatments that may be harmful to the environment. The advantages of thermal modification include an improvement in biological resistance, enhanced dimensional stability and a reduction in hygroscopicity (Tjeerdsma et al. 1998; Rowell 1999; Bekhta and Niemz 2003). A significant

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disadvantage is, however, a loss in toughness, which limits its use in structural applications. Thus far, it has mainly been softwoods that have been heat treated, and for non-structural purposes. With the abundance of hardwoods such as beech and ash, heat treatment is one way of adding value to these species and such processes have now been commercialised.

There has been a recent interest in the use of modified wood in true structural applications (Tjeerdsma et al. 2007). However, when the use of (thermally) modified wood in structural applications is envisioned, the fracture properties become extremely important, particularly fracture in tension perpendicular to the grain. Many studies reporting the mechanical properties of thermally modified wood have appeared in the literature (Santos 2000; Kubojima et al. 2000; Kamdem et al. 2002; Bekhta and Niemz 2003; Yildiz and Yildiz 2005; Poncsák et al. 2006; Shi et al. 2007; Borrega and Kärenlampi 2008), although the findings are difficult to compare due to the variation in treatments and testing conditions.

Since pioneering work developed in the 1960s, fracture mechanics has replaced empiricism in the field of timber engineering problems (Gustafsson 1992; Valentin et al. 1991). Most of the studies employing fracture mechanical approaches to wood have so far been performed on unmodified material (Jeronimidis 1980; Tschegg et al. 2001; Tukiainen and Koponen 2006). Numerous researches in the field of wood science have applied both linear elastic fracture mechanics (LEFM) and non-linear elastic fracture mechanics (NLEFM). Through the LEFM approach, parameters like the critical stress intensity factor K_{Ic} can be obtained. K_{Ic} characterises the maximum stress state quantifying the resistance that a material possesses to crack initiation. Another important parameter is the initial slope of the load–displacement curve, k_{init}, which is proportional to the stiffness of the material and characterises the deformation behaviour before the crack starts. Some violations of the assumptions of LEFM principles (Aicher and Reinhardt 1993) are the strain softening process zones that appear around the crack-tip (Boatright and Garrett 1983). NLEFM concepts are more appropriate in several practical situations (Gustafsson 1988). Useful parameters like the specific fracture energy, G_f, which is derived from the load–displacement histories, and which represents the whole fracture process until complete separation of the specimen including crack initiation and propagation yield, can be obtained.

Different techniques for measuring macroscopic wood fracture are available. These include the wedge splitting method (Tschegg 1986), the Nordtest Build 422 method and Compact Tension (CT) tests (Boström 1992). These methods have also been used to investigate fracture in other materials such as concrete (Tschegg et al. 1995), ceramics (Harmuth et al. 1996) and wood composites (Ehart et al. 1996). Different techniques, such as digital image processing, have been used to obtain full-field displacement and strain profiles (Samarasinghe and Kulasiri 2000). As the microstructure of every wood species plays an important role in its behaviour, advanced techniques are needed to study fracture properties at the microscopic level. Nowadays, for instance, environmental scanning microscopy (ESEM) can be combined with in situ deformation allowing full-field displacement measurement to be undertaken using image processing methods (Vasic and Smith 1996; Sippola and Koponen 1999; Frühmann et al. 2003; Vasic and Stanzl-Tschegg 2007).

There have been few studies on fracture properties of modified wood reported in the literature to date. Reiterer and Sinn (2002) reported the fracture properties of unmodified spruce and spruce modified by thermal treatment and acetylation, finding reductions in toughness of 20% with acetylated wood, and from 50 to 80% for heat-treated material.

Few previous investigations have considered the dependency of fracture behaviour on moisture content (MC), and those that have, have focused mainly on untreated wood. Most of these studies have employed LEFM principles and reported values for K_{Ic} (Kretschmann and Green 1996; Prokopski 1996; King et al. 1999; Reiterer and Tschegg 2002; Wang et al. 2003) with relatively low statistical significance in the range of 12–18% MC and higher values for dry wood. Smith and Chui (1994) determined the fracture energy under Mode I loading using flexural tests at different MC. This study showed that an increase in the fracture energy was found with decreasing MC from fibre saturation point to a MC of 18%; below MC of 18%, any reduction in MC leads to a reduction in fracture energy. Reiterer and Sinn (2002) reported an increase in specific fracture energy with increasing of MC.

In the work reported in this paper, the fracture properties and fracture toughness of two hardwood species, beech (*Fagus sylvatica* L) and ash (*Fraxinus excelsior* L), thermally modified to three different levels were studied. Fracture tests were carried out using CT tests under Mode I loading and steady-state crack propagation conditions using fracture mechanics principles. The investigations were carried out in the RL (radial-longitudinal) and TL (tangential-longitudinal) crack propagation systems. The results are supplemented by scanning electron microscope (SEM) images of the fracture surfaces. In order to investigate the possible influences of environmental conditions on the fracture properties, the behaviour at different MC was also assessed.

Experimental

Material

Beech and ash were provided by Mitteramskogler GmbH, Austria. The pre-dried (to approx. 8–12% MC) wood boards were modified at heating temperatures of 180° (*Mezzo*), 200° (*Forte*) and 230° (*Forte exterior*) in a dry three-stage heat-treatment process (Mitteramskogler 2007). Both the treated and untreated wood samples were obtained from the same log (i.e. "twin" boards, with one half thermally treated and the other half left untreated) in order to reduce variability.

The colour changes resulting from the three different heat treatments were measured using a Minolta CM 2600d spectrophotometer reported according to the CIE L^*a^*b system (Patzelt et al. 2003; Esteves et al. 2008; Bekhta and Niemz 2003; Johansson and Morén 2006).

For the wood fracture tests under mode I loading, notched CT specimens were prepared with an initial notch about 5 mm long. The initial notch was then lengthened with a small band saw and finally sharpened with a razor blade cut about 2 mm long (see Fig. 1). In the RL and TL crack propagation systems studied, the
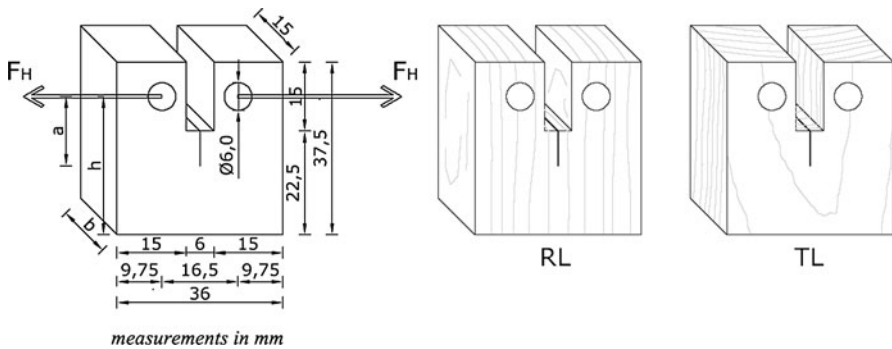
Fig. 1 Specimen geometry and orientations

first letters, R (radial) and T (tangential), respectively, indicate the direction of the normal to the crack plane. The second letter, L (longitudinal), refers to the direction of crack propagation.

According to Stanzl-Tschegg et al. (1995), there is no size dependence when the specimen dimensions are sufficiently large; however, in the present study, the specimen geometry was too small to obtain size-independent fracture values. Nevertheless, since all of the specimens had the same dimensions and geometry, a comparison of the experimental values and determination of the change in fracture properties due to the different heat treatments was possible.

Prior to testing, the specimens were conditioned at 20–22°C and 33, 65 and 95% relative humidity (RH) using salt solutions until equilibrium moisture content (EMC) was reached. The MC of each specimen was determined by oven drying for 24 h at 103°C and re-measuring the weight. Throughout, MC is expressed as the moisture contained in a sample as a percentage of the oven dry weight.

Density was measured from sets of approximately 30 cubic samples conditioned at 20–22°C and 65% RH to constant weight. Values varying from 600 to 777 kg/m³ for untreated beech and from 472 to 727 kg/m³ for heat-treated beech were obtained. In the case of ash, the density of the untreated samples varied from 617 to 694 kg/m³ and for treated samples, from 516 to 673 kg/m³.

Procedure

The Mode I CT tests were carried out using a small stepper motor driven loading device (Fig. 2). The notched specimens were loaded via two pins affixed to the cross-heads as shown in Fig. 2. The specimens were loaded up to failure under displacement control at a speed of 0.4 mm/min. Force was measured with a load cell having a maximum capacity of ±5 kN. Crack mouth opening displacement (CMOD) was recorded using a digital displacement gauge. At the end of the tests, the remaining ligament of the specimen was measured. Complete load–CMOD curves were obtained in all the tests. The specific fracture energy (G_f) was calculated from the integrated area under these curves (Fig. 3) divided by the area of the fracture surface, A, of the specimen according to Eq. (1), where F_H represents the horizontal splitting force and δ the CMOD:

Fig. 2 Testing device
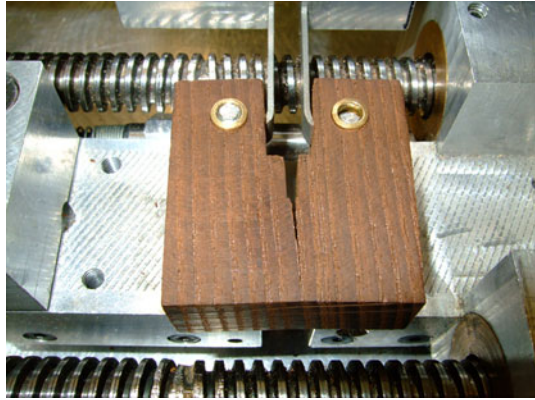
$$G_f = \frac{1}{A} \int_0^{\delta\,\text{max}} F_H(\delta)\, d\delta \qquad (1)$$

G_f is a "toughness" quantity and characterises the whole fracture process form crack initiation until complete separation of the specimen, including propagation yield. This quantity represents the energy needed to produce a unit fracture area, including the dissipated energy.

In order to characterise the stiffness of the specimens, the initial slope of the load–displacement curves in the linear elastic region k_{init} was determined (Eq. 2)

$$k_{\text{init}} = \mathrm{d}F_H/d\delta \qquad (2)$$

This quantity is proportional to the modulus of elasticity of the material.

The critical stress intensity factor K_{Ic} was also calculated, according to Eq. (3) (Zhao et al. 1991; Reiterer and Sinn 2002).

$$K_{\text{Ic}} = 3.657 F_{\text{H max}}(1 - a/h)^{-3/2}/(bh^{1/2}) \qquad (3)$$

Where F_{Hmax} is the maximum horizontal force, a is the distance from the line of action of the horizontal force to the notch tip, h is the distance from this line of action to the bottom of the specimen and b the thickness of the sample (Fig. 1). The
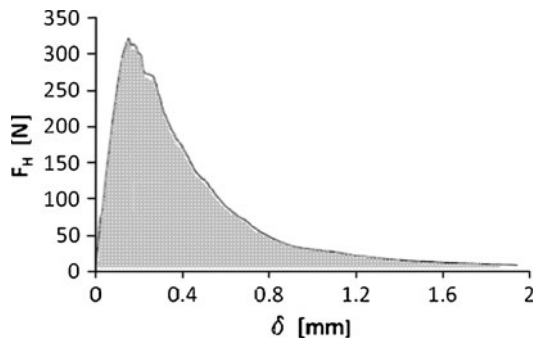
Fig. 3 Typical load–displacement *curve*

Table 1 Colour changes in heat-treated beech and ash

	n	ΔL^* [%]		Δa^* [%]		Δb^* [%]		ΔE^* [%]	
		Radial	Tang.	Radial	Tang.	Radial	Tang.	Radial	Tang.
BEECH									
Mezzo	8	−13.72 (1.84)	−15.97 (1.54)	2.21 (0.30)	3.13 (0.21)	3.68 (0.54)	3.23 (0.22)	14.40 (1.06)	16.60 (1.50)
Forte		No data available							
Forte ext.	7	−34.67 (1.42)	−36.28 (1.55)	−1.90 (0.82)	−2.09 (0.86)	−8.64 (1.51)	−10.14 (1.76)	35.81 (1.72)	37.75 (1.99)
		L^*		a^*		b^*			
Control	6	68.09 (1.55)	69.96 (1.14)	9.02 (0.38)	9.17 (0.43)	19.18 (0.40)	19.69 (0.71)		
ASH									
Mezzo	6	−21.16 (1.96)	−21.09 (1.70)	4.42 (0.34)	5.01 (1.70)	2.86 (0.67)	3.07 (1.35)	21.83 (1.84)	21.94 (1.67)
Forte	7	−39.80 (1.68)	−40.33 (1.90)	1.33 (0.60)	1.99 (0.88)	−10.85 (0.77)	−9.83 (2.00)	41.28 (1.80)	41.59 (2.27)
Forte ext.	5	−42.86 (1.94)	−43.52 (0.55)	1.32 (0.62)	1.01 (0.22)	−8.95 (1.25)	−11.51 (0.33)	43.81 (2.13)	45.03 (0.45)
		L^*		a^*		b^*			
Control	6	78.3 (1.67)	78.60 (1.28)	5.68 (0.59)	5.81 (0.38)	21.17 (0.81)	22.59 (0.22)		

Values in parenthesis indicate standard deviations. n = number of specimens

expression was applied first to isotropic materials, but recent studies have justified this approach for crack initiation parallel to the grain (Schachner et al. 2000).

Results and discussions

Colour measurements

To support the fracture studies and in order to characterise the degree of thermal modification, the colour of the radial and tangential sections was determined in accordance with the CIE L^*a^*b system. Differences between treated and untreated wood were observed in the L^*, a^* and b^* coordinates (Table 1). Total colour change, ΔE^*, was calculated using the previous colour space values. L^* (lightness) ranges from 0% (black) to 100% (white); the chromaticity coordinates a* from green ($-a$) to red ($+a$) and b* from blue ($-b$) to yellow ($+b$).

The measured averages of ΔL^*, Δa^*, Δb^* and ΔE^* are presented in Table 1.

The colour spectra are shown in Fig. 4.

With thermal treatment, the colour of the wood was altered, becoming darker as the severity of treatment increased. Colour change is associated with the formation of coloured degradation products from hemicelluloses (Sundqvist 2004) and from extractives (Sundqvist and Morén 2002). Another reason for the colour change is the formation of oxidation products (Tjeerdsma et al. 1998; Bekhta and Niemz 2003). On average, lightness decreased with increasing treatment severity, along with a^* and b^* values. The difference between tangential and radial sections was not significant.

EMC curves

Prior to the fracture studies, the equilibrium moisture contents of the samples were calculated. For all treated wood, EMC was 3.7 ± 1.1% at 33% RH, and at 65% RH, it was 5.4 ± 1.5% whilst at 95% RH, EMC was 15.5 ± 6.7%. For untreated wood, the EMC values were 7.5 ± 0.3, 11.6 ± 0.8 and 23.4 ± 1.2% at 33, 65 and 95% RH, respectively. The RH–EMC relationships of treated and untreated beech and ash are shown in Fig. 5. Each point represents the mean of 20 measurements.
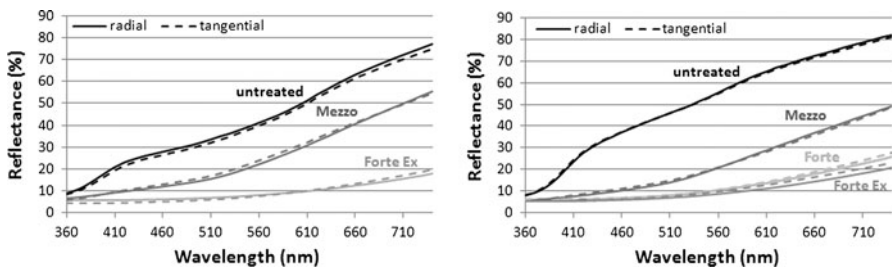

Fig. 4 Colour spectra for untreated (UT) and heat-treated (T) beech (*left*) and ash (*right*) for Mezzo (ME), Forte (FO) and Forte exterior (FE) heat treatments
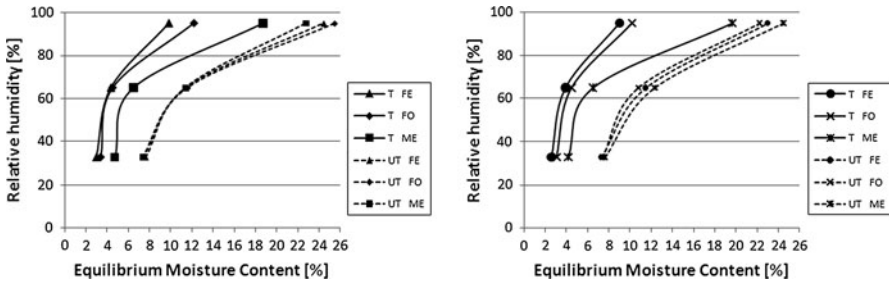
Fig. 5 Equilibrium Moisture Content curves for beech (*left*) and ash (*right*), for the treated (T) and untreated (UT) twins and Forte exterior (FE), Forte (FO) and Mezzo (ME) thermal treatments

Fracture properties

Typical load versus CMOD curves obtained from the different sets of CT tests on beech are shown in Fig. 6. In the case of ash, the load versus CMOD curves were similar to those of beech. The curves provide useful information about fracture behaviour. The top row of the group of diagrams corresponds to the results obtained in the RL propagation system and the bottom row to the TL propagation system. The solid lines represent the results for heat-treated wood, whilst the dotted lines show the untreated material. The different greys correspond to the different moisture contents. The same scale was chosen for all the graphs in order to make a direct comparison possible.

The forms of the curves for treated and untreated differ significantly. Differences can be quantified with the following parameters: maximum load, F_{max}, the initial slope, k_{init}, the critical stress intensity factor, K_{Ic}, and the specific fracture energy, G_{f}.
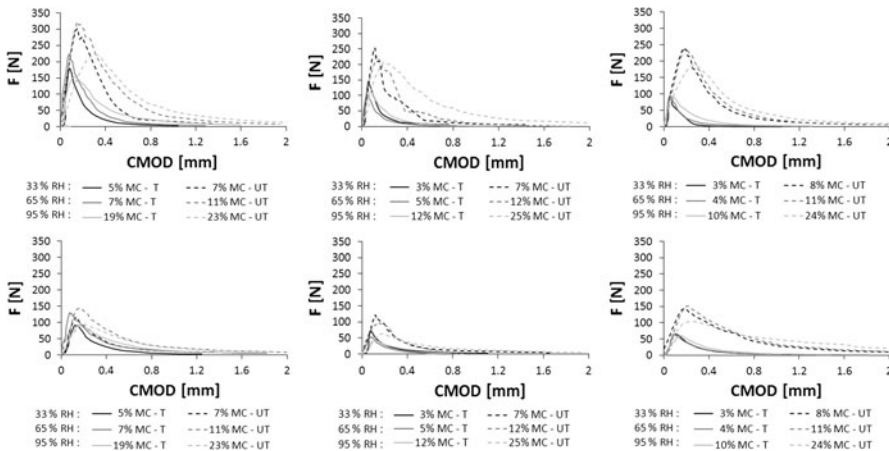

Fig. 6 Representative CMOD versus load (F) curves for untreated (UT) and heat-treated (T) beech, in the RL (*upper series*) and TL (*lower series*) systems, for Mezzo (*left*), Forte (*middle*) and Forte exterior (*right*) heat treatments, after conditioning at 33, 65 and 95% RH

Table 2 Average values and standard deviations for the initial slope k_{init} of heat-treated and untreated beech and ash, in RL and TL systems at different MC. The values represent the mean of 10 measurements

Treat.	Heat-treated beech						Untreated beech					
	RH (%)	ρ	k_{init} (RL)	SD	k_{init} (TL)	SD	RH (%)	ρ	k_{init} (RL)	SD	k_{init} (TL)	SD
ME[a]	33		2308	293	1086	438	33		2250	432	1061	261
	65	706	2726	322	1639	62	65	730	2010	533	1263	391
	95		1176	297	696	59	95		1001	201	655	153
FO[b]	33		3361	259	1514	449	33		3081	260	1554	501
	65	594	2680	634	989	223	65	744	2387	383	970	84
	95		1554	774	603	119	95		1372	146	455	128
FE[c]	33		1981	666	1091	138	33		1611	253	880	176
	65	574	1450	423	1012	254	65	743	1440	127	947	92
	95		1236	353	736	197	95		804	73	593	108

Treat.	Heat-treated ash						Untreated ash					
	RH (%)	ρ	k_{init} (RL)	SD	k_{init} (TL)	SD	RH (%)	ρ	k_{init} (RL)	SD	k_{init} (TL)	SD
ME[a]	33		2.267	304	1.266	201	33		1.896	400	1.072	242
	65	706	1.795	340	1.184	100	65	730	1.503	299	889	184
	95		905	101	610	111	95		977	33	569	125
FO[b]	33		2.306	358	1.071	211	33		1.440	325	1.095	280
	65	594	1.835	398	1.065	259	65	744	1.824	282	1.103	97
	95		1.459	260	1.096	121	95		901	158	693	109
FE[c]	33		2.023	623	990	382	33		2.085	342	1.217	121
	65	574	1543	145	720	73	65	743	1.338	205	983	32
	95		1.392	249	702	227	95		905	79	534	87

[a] Mezzo, [b] Forte, [c] Forte Exterior; MC: Moisture content [%]; ρ: Density [kg/m^3]

k_{init}: Initial slope [N/mm]; SD: Standard deviation

F_{max} is a characteristic strength parameter. The F_{max} attained in the tests on the treated and untreated twins was entirely different, being highest for the untreated raw material and decreasing as the severity of thermal treatment increased. The difference between the two species is not particularly pronounced. As may be observed (Fig. 6), the load–CMOD curves for the heat-treated wood show smoother and more uninterrupted behaviour than those exhibited by the unmodified raw material, which displays more uneven behaviour. The explanation for this could lie in the complex structure of hardwoods, which consists on a high proportion of radially oriented cells (rays) acting as reinforcement that compel the crack to take a more winding trajectory (Reiterer et al. 2002a, b). The maximum load is evident as an angular peak, typical of linear elastic brittle hardwoods (Stanzl-Tschegg 2006). A deviation is noticeable in the case of untreated wood at higher MC which exhibits a more rounded peak at the maximum load.

The results of the initial slope, k_{init}, are shown in Table 2.

The differences in k_{init} between treated and untreated wood are small and no clear, general, trend can be seen. Nevertheless, slightly higher values can be

Table 3 Average values and standard deviations for the critical stress intensity factors, K_{Ic}, of heat-treated and untreated beech and ash, in the RL and TL systems at different MC

Treat.	Heat-treated beech						Untreated beech						% Reduction	
	RH (%)	ρ	K_{Ic} (RL)	SD	K_{Ic} (TL)	SD	RH (%)	ρ	K_{Ic} (RL)	SD	K_{Ic} (TL)	SD	RL	TL
ME[a]	33		0.70	0.07	0.36	0.03	33		1.15	0.06	0.48	0.02	39	23
	65	706	0.73	0.05	0.43	0.03	65	730	1.01	0.07	0.47	0.04	28	8
	95		0.58	0.04	0.35	0.05	95		0.78	0.01	0.36	0.02	26	2
FO[b]	33		0.49	0.02	0.25	0.05	33		0.91	0.02	0.48	0.09	46	47
	65	594	0.38	0.03	0.19	0.03	65	744	0.87	0.06	0.44	0.02	57	56
	95		0.37	0.02	0.14	0.02	95		0.72	0.02	0.24	0.02	49	39
FE[c]	33		0.38	0.03	0.26	0.02	33		0.96	0.06	0.63	0.10	60	58
	65	574	0.36	0.02	0.25	0.02	65	743	0.92	0.09	0.59	0.06	61	57
	95		0.33	0.04	0.23	0.01	95		0.70	0.06	0.39	0.03	53	41

Treat.	Heat-treated ash						Untreated ash						% Reduction	
	RH (%)	ρ	K_{Ic} (RL)	SD	K_{Ic} (TL)	SD	RH (%)	ρ	K_{Ic} (RL)	SD	K_{Ic} (TL)	SD	RL	TL
ME[a]	33		0.48	0.06	0.48	0.01	33		0.90	0.03	0.68	0.06	47	29
	65	706	0.47	0.05	0.48	0.04	65	730	0.88	0.11	0.65	0.03	47	25
	95		0.46	0.07	0.43	0.01	95		0.69	0.04	0.43	0.01	33	0.6
FO[b]	33		0.35	0.02	0.26	0.02	33		0.76	0.01	0.66	0.14	53	60
	65	594	0.32	0.01	0.28	0.02	65	744	0.77	0.02	0.64	0.03	58	57
	95		0.33	0.01	0.26	0.01	95		0.65	0.02	0.43	0.01	49	40
FE[c]	33		0.32	0.02	0.22	0.01	33		0.69	0.02	0.56	0.03	53	61
	65	574	0.32	0.02	0.22	0.01	65	743	0.69	0.04	0.55	0.01	53	60
	95		0.30	0.03	0.25	0.01	95		0.59	0.03	0.37	0.01	48	34

The values represent the mean of 10 measurements

[a] Mezzo, [b] Forte, [c] Forte Exterior; RH: Relative Humidity [%]; ρ: Density [kg/m³]

K_{Ic} : Critical Stress Intensity Factor [MPam$^{1/2}$]; SD: Standard deviation

associated with the heat-treated sets, especially in beech. However, there are some cases where the k_{init} of heat-treated samples is only as high as the raw material itself and also other cases where a higher k_{init} is seen in the untreated wood. This mostly occurs in ash in the TL system. As the initial slope, k_{init}, characterises stiffness and is proportional to an effective modulus of elasticity, these findings indicate that a generally higher modulus of elasticity would be expected in the treated wood (Santos 2000; Kocaefe et al. 2008). Some researchers have, however, reported an inverse trend (Schneider 1971; Yildiz et al. 2002; Reiterer and Sinn 2002).

The critical stress intensity factors, K_{Ic}, obtained are shown in Table 3.

In this case, clear differences in K_{Ic} were found between the heat-treated sets and the unmodified material; the lower values seen in the case of the heat-treated wood indicate that crack initiation occurs more readily in this material.

Table 4 Average values and standard deviations for the specific fracture energies G_f, of heat-treated and untreated beech and ash, in RL and TL systems for different MC

Treat.	Heat-treated beech						Untreated beech						% Reduction	
	RH (%)	ρ	G_f (RL)	SD	G_f (TL)	SD	RH (%)	ρ	G_f (RL)	SD	G_f (TL)	SD	RL	TL
ME[a]	33		207.14	17.3	123.82	11.7	33		461.57	62.7	203.20	19.6	55	39
	65	706	242.25	39.0	191.85	24.5	65	730	563.75	86.9	318.60	60.7	57	40
	95		299.60	27.0	204.22	10.5	95		637.65	91.9	281.48	37.7	53	24
FO[b]	33		74.09	21.4	66.30	10.7	33		300.48	36.1	192.66	25.9	75	66
	65	594	75.80	10.4	74.99	12.2	65	744	310.16	51.2	263.65	48.4	76	72
	95		106.81	34.4	75.23	9.4	95		556.88	58.4	166.90	41.4	81	55
FE[c]	33		65.83	13.9	75.15	6.6	33		472.52	32.8	462.08	74.3	86	83
	65	574	67.01	7.6	77.65	7.3	65	743	488.50	61.4	489.84	65.9	86	84
	95		90.92	18.3	99.60	20.5	95		570.94	54.8	450.47	71.5	84	78

Treat.	Heat-treated ash						Untreated ash						% Reduction	
	RH (%)	ρ	G_f (RL)	SD	G_f (TL)	SD	RH (%)	ρ	G_f (RL)	SD	G_f (TL)	SD	RL	TL
ME[a]	33		131.56	23.6	181.97	19.3	33		421.68	38.8	396.31	79.5	69	54
	65	651	159.08	29.6	202.10	15.0	65	655	529.38	80.6	457.61	71.9	70	56
	95		235.77	56.0	282.37	23.5	95		594.65	57.8	365.56	48.4	60	23
FO[b]	33		62.21	12.5	69.69	11.4	33		306.85	41.5	242.85	36.6	80	71
	65	573	63.12	11.3	89.98	11.8	65	663	361.29	22.5	316.88	33.3	82	72
	95		96.70	12.1	106.28	15.6	95		412.29	39.9	312.20	35.4	76	66
FE[c]	33		69.47	10.1	52.59	5.8	33		277.31	36.5	214.03	19.6	75	75
	65	534	83.53	9.4	72.75	12.0	65	654	340.39	39.6	304.38	30.9	75	76
	95		100.79	18.7	91.97	9.4	95		365.90	34.7	249.89	26.6	72	63

The values represent the mean of 10 measurements

[a] Mezzo, [b] Forte, [c] Forte Exterior; RH: Relative Humidity [%]; ρ: Density [kg/m^3]

G_f : Average Specific Fracture Energy [J/m^2]; SD: Standard deviation

In Table 4, the results for the specific fracture energies G_f of the beech and ash twins are shown.

As expected, in both species and in both crack propagation systems, G_f was found to be higher in the case of untreated wood than in treated wood. No significant differences in G_f could be observed between the wood subjected to the two severest treatments, FO and FE.

Heat-treatment produces a degradation of the hemicelluloses, which is the wood component with the highest thermal sensitivity (Zaman et al. 2000). Hemicelluloses play an important role in strength (Esteves et al. 2007) and their degradation, which is expected to take place at temperatures above 120°C (Fengel and Wegener 1989), can therefore affect the strength properties of wood. It follows then that the fracture properties are likely to be affected and this contention is supported by the results of

this study. The reduction in the toughness of heat-treated wood can be attributed to its lowered plastic ductility. Crack initiation is easier in thermally modified wood, as shown by the lowered K_{Ic}, and the crack propagation phase consumes less energy and takes place in a more brittle manner in the treated wood.

For the untreated material, the generally higher fracture parameters values obtained in the RL crack propagation system indicate that in both species the stiffness in this orientation is higher than in the TL direction. When the structure of both hardwoods is considered, the higher G_{f} values seen in the untreated material in the RL system could, once more, be attributed to a high proportion of rays acting as reinforcement. According to several authors (Beery et al. 1983; Burgert and Eckstein 2001; Reiterer et al. 2002a), before macro-crack initiation, as well as during the crack propagation phase, the rays carry tensile stresses in the developing process zone (comprising micro-cracks and irreversibly deformed regions) around the crack-tip. Moreover, additional fracture energy is consumed by the rays by creating fibre bridges behind the crack-tip during crack propagation. The ratios between RL and TL system are higher for K_{Ic} than for the equivalent G_{f}. This may signify that the rays have a greater influence on the crack initiation phase than on the propagation phase. In the case of the treated material, the fracture values were also generally higher in RL system than in the TL system, but the differences were especially slight for the most severe treatments.

In both species, as the severity of the treatment increases, the percentage reductions in K_{Ic} and G_{f} relative to the untreated materials also increase. These reductions are higher in the RL system than in the TL system, though between the two severest treatments, FO and FE, the percentage reductions are similar. The difference in these reductions between beech and ash is not large.

The structural features are clearly visible on the fracture surfaces shown in Figs. 7 and 8 which support the differences described above.

In Fig. 7a, the RL fracture surface of untreated ash can be seen. It is clearly visible how the multiseriate rays are severed by the propagation of the crack. Figure 7b shows the TL fracture surface of untreated ash. Regions where the multiseriate rays are separated from the axial tissue are clearly seen. Figure 8a, b shows the fracture surface of FE-treated ash in the RL and TL directions,
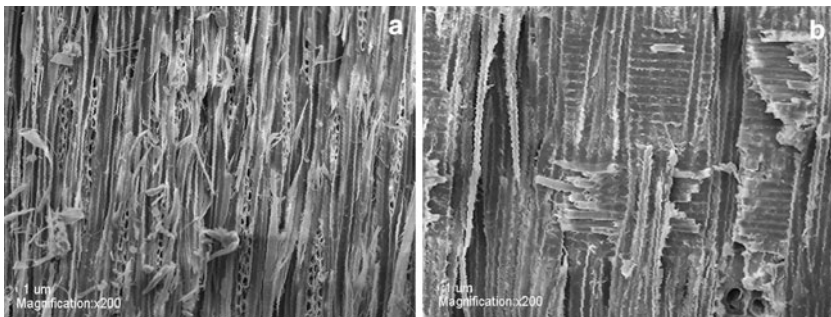

Fig. 7 SEM micrographs of the fracture surface of untreated ash in the RL (**a**) and TL (**b**) crack propagation system
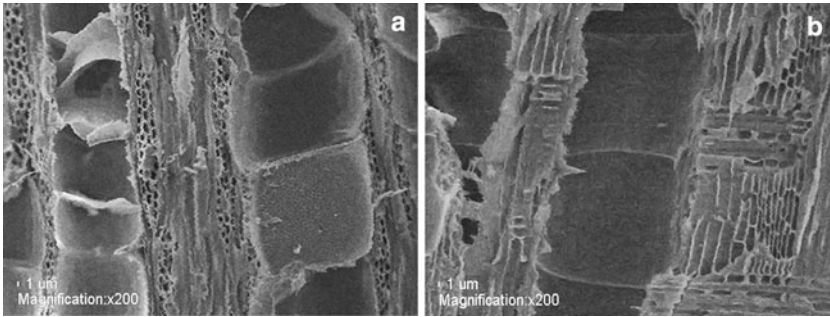
Fig. 8 SEM micrographs of the fracture surface of treated ash (FE) in the RL (**a**) and TL (**b**) crack propagation system
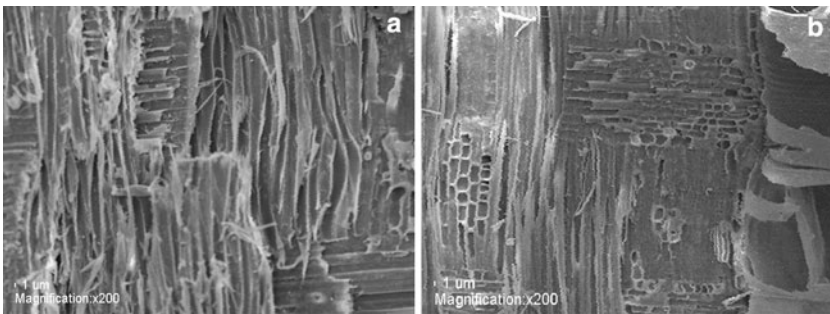

Fig. 9 SEM micrographs of the fracture surface of ME-treated ash (**a**) and FO-treated ash (**b**) both in TL system

respectively. It can be clearly seen how the fracture surface of the untreated material (Fig. 7) is significantly more deformed, with many parts of the cell walls being pulled-out, than the fracture surface of the heat-treated sample, which exhibits predominantly cleanly cut cell walls (Fig. 8). A similar finding may be observed from Fig. 9a, b that show the fracture surfaces of ME- and FO-treated ash, respectively, in the TL system. The behaviour of ME-treated ash is closer to that of untreated ash, with some pulled-out cell walls, whereas FO exhibits a predominance of clean-cut cell walls, similar to those shown in FE-treated ash (Fig. 8b). These observations closely support the results from fracture tests. These findings agree with those of other authors (Reiterer and Sinn 2002; Reiterer et al. 2002a, b), who reported greater reductions over the raw material in heat-treated spruce than in acetylated spruce; differences between the RL and TL systems were also noted, being similar to the results obtained in this study.

Moisture content is another factor which is known to influence most of the mechanical properties of wood. Increasing MC leads to a reduction in stiffness and strength. In this study, it has been clearly shown that MC also affects the fracture properties. As shown in EMC subsection, heat-treated wood is less hygroscopic than untreated wood, with the EMC values of the treated wood being lower than those of the untreated material at the same relative humidity. This in turn influences the

crack propagation stage; for a low MC, a more brittle crack propagation phase can be expected.

With increasing MC, the load–CMOD curves showed a decrease in the maximum stress state and an increase in the displacements (Fig. 6). For untreated beech and ash, the decrease in the maximum load (F_{max}) and K_{Ic} values in the 7–8 and 11–12% MC ranges (corresponding to 33 and 65% RH, respectively) are not significantly different. However, a more pronounced decrease in maximum load was obtained at a MC of 22–24% (corresponding to 95% RH). For the heat-treated beech and ash, the differences in F_{max}, as well as in K_{Ic}, between all moisture contents were slight. This result agrees with mostly of the related investigations in this field (Kretschmann and Green 1996; Prokopski 1996; King et al. 1999; Reiterer and Tschegg 2002).

The influence of MC on G_f is more pronounced than on K_{Ic}. An increase in MC was found to result in an increase in G_f, which is in agreement with the results reported by Reiterer and Tschegg (2002). This means that at higher MCs, more energy per unit area is needed to separate a wood sample into two halves. The energy needed to create the process zone for crack propagation increases due to the higher ductility, more energy is consumed in fibre bridging behind the crack-tip and also the dissipation energy increase because of irreversible deformation. The CMOD prior to the crack start is larger with increasing MC, which means that wood can be strained more until crack propagation initiates. This increase in ductility accompanies the reduction in stiffness argued by the findings of K_{Ic}. A deviation from these results was found for untreated wood in the TL orientation, which showed the highest G_f at intermediate moisture content levels.

For heat-treated wood, the increase in G_f in the 7–8 and 11–12% MC ranges are not significantly different either. ME sets in the TL system and in the highest MC, 18–20%, reached G_f close to the values obtained for untreated wood in the same system and MC condition.

Conclusion

The influence of three different heat treatments (ME, FO and FE), three moisture contents and two crack propagation systems, RL and TL, on the Mode I fracture behaviour of beech and ash have been evaluated using compact tension tests. The load–displacement diagrams were used to determine the basic fracture parameters. Heat-treated specimens showed a decrease in K_{Ic} and G_f in comparison with the unmodified raw material. The more severe the treatment, the lower both K_{Ic} and G_f values, as crack initiation was easier and the crack propagation phase consumed less energy and took place in a more brittle way. The initial slope indicated that the stiffness values did not differ significantly between untreated and heat-treated wood.

Most of the fracture parameters were found to be higher in the RL crack propagation system than in the TL system and may be accounted for by the high proportion of rays acting as reinforcement in the crack plane. This apparently has a strong influence, especially on K_{Ic}.

ME treatment in both species showed the lowest K_{Ic} percentage reductions varying between 25 and 46% in the RL system and 0.6–28% in the TL system. FO and FE K_{Ic} reductions varied between around 44 and 61% compared to the untreated raw material in the RL system and between 34 and 60% in the TL system. G_f percentage reduction values were higher than those of K_{Ic}. In the RL direction, ME showed G_f values reduced by approximately 53–70% in both species, and 72–86% in the case of FO and FE, compared to the unmodified material. In the TL direction, the reduction was in the range of 23–56% for ME and 63–84% for both FO and FE. No huge differences were found between species. Therefore, it can be concluded that crack initiation and crack propagation are easier in beech and ash subjected to the more severe treatments.

Increasing moisture content leads to an increment of ductility, which also produces a significant increase in the specific fracture energy, which characterises the entire fracture process. For the unmodified wood in the TL orientation, the highest G_f values were obtained at intermediate moisture contents and not at the greatest MC. It must be noted that for K_{Ic}, the opposite trend occurred; a decrease in this parameter being observed with increasing MC.

The greater brittleness of the thermally treated wood is expressed in the G_f and K_{Ic} parameters. The significant decrease in the fracture properties is mainly due to the heat-treatment process and to a lesser extent to changes in MC. Thus, severe treatment penalises the potential of thermally modified beech and ash in structural applications. Further studies are currently underway on the application of the fracture properties of TMT to find a fracture model for this material.

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