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# MECHANICAL CHARACTERIZATION OF HEAT-TREATED ASH WOOD IN RELATION WITH STRUCTURAL TIMBER STANDARDS

#### Simon HANNOUZ

Ph.D. Candidate – Arts et Métier ParisTech Address: LaBoMaP, rue porte de Paris, 71250 Cluny, France E-mail: simon.hannouz@ensam.eu

#### Robert COLLET

Dr.Eng – Arts et Métiers ParisTech Address: LaBoMaP, rue porte de Paris, 71250 Cluny, France E-mail: robert.collet@ensam.eu

#### Jean-Claude BUTAUD

Eng. – Arts et Métiers ParisTech Address: LaBoMaP, rue porte de Paris, 71250 Cluny, France E-mail: iean-claude.butaud@ensam.eu

#### Laurent BLERON

Prof.Dr.Eng. – Université de Lorraine Address: LERMAB, 27 rue Philippe Séguin - CS 60036, 88026 Épinal Cedex, France E-mail: laurent.bleron@univ-lorraine.fr

#### Kévin CANDELIER®

Dr.Eng. – Cirad, UR BioWooEB

Address: CIRAD-Unité de Recherches BioWooEB, TA B 114/16, Montpellier, France
Phone: (33) 3 83 68 40 00; Fax (33) 3 83 68 40 01

E-mail: kevin.candelier@cirad.fr

#### Abstract:

Heat treatment is an attractive method to enhance wood durability, and valorize local hardwood species with natural low durability. Yet no standard allows the certification of such products. This study first aims to observe the influence of heat treatment on the different mechanical properties. The standard mechanical tests; bending, tension parallel and perpendicular to grain, compression parallel and perpendicular to grain and shear, have been performed on native and heat-treated woods samples. The measurements are then compared to values of EN 338 standard. Results reveal that shear strength is the property most affected by heat treatment and that the modulus of elasticity perpendicular to grain is increased. The values given by EN 338 standard are generally safe with the exception of shear strength which is underestimated by current relationships. It is suggested that new relationships have to be provided for heat-treated wood, taking into account the loss of shear resistance.

**Key words:** hardwood; heat treatment; mechanical grading.

#### INTRODUCTION

### Heat treatment to promote hardwood species

With more than 71% of the total forest area, hardwood species is a major resource for French forest products. However, because of their faster growth, softwood species are the main raw material transformed by sawmills in France (FCBA 2013). Statistics concerning hardwood manufacturing have steadily been decreasing since 1990 while their biological production has increased (FCBA 2011). This trend can be due to the closing down of markets such as furniture and joinery, when in the meantime demand for timber has increased the use of softwoods. One reason for this tendency rises from the low natural durability of most hardwood species bringing their utilization at stake.

The heat treatment of wood is an attractive method to promote the local and non-durable wood by improving its decay resistance. It is now well known that modifying wood through exposure above 160°C in an inert atmosphere leads to the improvement of its durability (Metsa-Kortelainen and Viitanen 2009, Candelier et al. 2013a), a better dimensional stability (Lekounougou et al. 2011, Finnish Thermowood Association 2003, Korkut et al. 2008, Welzbacher et al. 2009), and darker colour (Ahajji et al. 2009). Such

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<sup>&</sup>lt;sup>\*</sup> Corresponding author

improvements enable the use of beech, ash, poplar or oak sapwood in cladding, decking, or joinery. Indoor flooring is another market for heat-treated wood as far as the aesthetic aspect is concerned.

Chemical changes occuring during wood heat treatment also affect wood strength and stiffness properties (Kocaefe *et al.* 2008, Candelier *et al.* 2013b). The mechanical classification of a thermally modified wood is therefore disrupted after heat treatment. The anatomical structure of wood may change following heat treatment intensity, however Boonstra (Boonstra 2008) notices no damage in the case of ash modified by two-stage heat treatment under optimized conditions.

#### Timber mechanical grading

Using wood, as any other building materials requires to specify its mechanical performances, particularly to obtain the CE label. European standards define visual or machine grading for untreated wood, but these standards exclude heat-treated wood. Mechanical grading of untreated timbers is based on EN 408 and EN 338. The latter standard distinguishes softwood from hardwood and provides classes for both families. These classes depend on mechanical strength properties, stiffness properties, and density. These mechanical properties are listed in Table 1. In this Table, two mechanical classes for softwood and hardwood are given as examples.

Extract from table 1 of the standard EN 338

Table 1

		Softwood		Hardwood	
		C30	C35	D30	D35
Strength properties (in N/mm²)					
Bending	f <sub>m,k</sub>	30	35	30	35
Tension parallel	f <sub>t,0,k</sub>	18	21	18	21
Tension perpendicular	f <sub>t,90,k</sub>	0.4	0.4	0.6	0.6
Compression parallel	f <sub>c,0,k</sub>	23	25	23	25
Compression perpendicular	f <sub>c,90,k</sub>	2.7	2.8	8	8.1
Shear	$f_{v,k}$	4.0	4.0	4.0	4.0
Stiffness properties (in kN/mm²)					
Mean modulus of elasticity parallel	E <sub>0,mean</sub>	12	13	11	12
5% modulus of elasticity parallel	E <sub>0,05</sub>	8.0	8.7	9.2	10.1
Mean modulus of elasticity perpendicular	E <sub>90,mean</sub>	0.40	0.43	0.73	0.80
Mean shear modulus	G <sub>mean</sub>	0.75	0.81	0.69	0.75
Density (in kg/m³)					
Density	$\rho_k$	380	400	530	540
Mean density	ρ <sub>mean</sub>	460	480	640	650

However, not all properties of Table 1 are measured. Today, for non-treated wood and wood whose treatment does not affect its mechanical properties, only characteristic bending strength  $f_{m;k}$ , mean modulus of elasticity parallel  $E_{0;mean}$ , and characteristic density  $\rho_k$  are determined. The other properties are estimated by the equations given in EN 338. These relationships are given in Table 2.

Relations given by standard EN 338

Table 2

		Softwood	Hardwood	
Strength properties (in N/mm²)	•		•	
Bending	f <sub>m,k</sub>	measured		
Tension parallel	f <sub>t,0,k</sub>	0.6 f <sub>m,k</sub>		
Tension perpendicular	$f_{t,90,k}$	0.4	0.6	
Compression parallel	f <sub>c,0,k</sub>	5( f <sub>m,k</sub>	0.45	
Compression perpendicular	f <sub>c,90,k</sub>	$0.007 \rho_k$	$0.0015 \rho_k$	
Shear	f <sub>v,k</sub>	given by EN 338		
Stiffness properties (in kN/mm²)				
Mean modulus of elasticity parallel	E <sub>0,mean</sub>	measured		
5% modulus of elasticity parallel	E <sub>0,05</sub>	0.67 E <sub>0,mean</sub>	0.84 E <sub>0,mean</sub>	
Mean modulus of elasticity perpendicular	E <sub>90,mean</sub>	E <sub>0,mean</sub> /30	E <sub>0,mean</sub> /15	
Mean shear modulus	Gmean	E <sub>0,mean</sub> /16		
Density (in kg/m³)				
Density	$\rho_k$	measured		
Mean density	ρ <sub>mean</sub>	measured		

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The aim of this study is to observe the evolution of each property of EN 338 under heat treatment and to verify the validity of the equations given by EN 338, first on non-treated European ash wood, and then on a twin batch of heat-treated European ash wood.

#### **MATERIAL AND METHODS**

#### Wood samples

The studied hardwood species is European ash wood (Fraxinus exelsior). This species is known to have a low natural decay resistance. Heat treatment is thus a suitable means to give it an added value. The tests achieved for this study are already published within the frame of a master thesis (Mainguy 2013).

#### Heat treatment protocols

One thermal treatment has been performed with the ThermoWood<sup>®</sup> technology (Finnish Thermowood Association 2003) by the French company Bois Durables de Bourgogne. To perform wood heat treatment, the oven temperature was held at 210°C for 2 hours (Fig. 1).

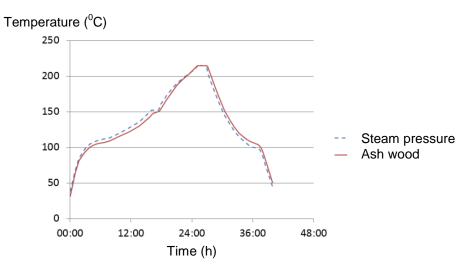


Fig. 1.

Temperature kinetics of ash wood and steam pressure during heat treatment performed at 210 °C for 2 hours.

In order to compare the behavior of non-treated and heat-treated wood in similar batches, each mechanical test has been performed on the same two-meter-long board for both samples the initial board is cut into two parts.

#### Bending tests

Four-point bending tests are achieved according to EN 408 standard. The size of specimens is 0.40x0.20x0.20 (L\*R\*T). The apparatus used is an INSTRON 4467 Universal Mechanical Test Machine with a capacity of 100kN fitted with a comparator to measure the bending radius at the centre span of the specimen. Samples were conditioned in a room with 65% RH and 22°C during the necessary time to stabilize the samples weights. The moving head speed and the span length were respectively 1.8mm.s<sup>-1</sup> and 160mm. The load deformation data obtained were analyzed to determine the modulus of elasticity (MOE) and the modulus of rupture (MOR).

43 specimens are tested for the non-treated wood and 38 concerning the heat-treated wood. The difference comes from specimens presenting defects or singularities, then discarded.

#### Other mechanical properties

In order to determine all properties described in Table 1 the following tests are performed: tension parallel to grain, tension perpendicular to grain, compression parallel to grain, compression perpendicular to grain, shear. All these tests have been performed with the same INSTRON 4467 Universal Mechanical Test Machine (capacity of 100kN). Samples were conditioned in a room with 65% RH and 22°C during the necessary time to stabilize the samples weights.

The standards used for the different tests as well as the number of specimens for each test are given in Table 3.

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Table 3

Number of specimens for each mechanical test (bending not included)	uded)
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Test	Standard	S (mm²)	Non-treated wood	Heat-treated wood
Tension	B51-017	16*4	41	45
parallel				
Tension	B51-010	20*20	43	42
perpendicular				
Tension	-	13*20	38	34
perpendicular				
Compression	B51-007	20*20	41	43
parallel				
Compression	EN408	40*90	39	41
perpendicular				
Shear	B51-012	20*20	41	51

Characteristic values are calculated on the basis of EN 14358. Finally, the uncertainty of measurement has been estimated around 4.5% concerning the modulus of elasticity obtained with bending tests, and less than 2% concerning strength properties.

#### **RESULTS AND DISCUSSION** Characteristic values

The calculation of characteristic values is allowed since strength tested values follow a logarithmic normal distribution whereas stiffness tested values follow a normal distribution. The hypothesis of logarithmic normal or normal distribution has been validated in regard to standard X 06-050.

Results from calculations and mechanical grading corresponding to each characteristic value are displayed in Table 4.

Table 4 Characteristic values from mechanical tests and from European Standard EN 338

		Measurements		Estimated values			
		Non-treated	Heat-treated	Non-treated	Heat-treated		
Strength properties (in N/mm²)							
Bending	f <sub>m,k</sub>	71.2 (D70)	35.6 (D35)	71.2 (D70)	35.6 (D35)		
Tension parallel	f <sub>t,0,k</sub>	56.9 (D70)	36.0 (D60)	42.7 (D70)	21.3 (D35)		
Tension perpendicular	f <sub>t,90,k</sub>	2.4 (D70)	1.3 (D70)	0.6 (D70)	0.6 (D70)		
Compression parallel	f <sub>c,0,k</sub>	44.6 (D70)	49.5 (D70)	34.1 (D70)	24.9 (D30)		
Compression perpendicular	f <sub>c,90,k</sub>	9.3 (D50)	6.7 ( <d18)< td=""><td>4.3 (<d18)< td=""><td>3.6 (<d18)< td=""></d18)<></td></d18)<></td></d18)<>	4.3 ( <d18)< td=""><td>3.6 (<d18)< td=""></d18)<></td></d18)<>	3.6 ( <d18)< td=""></d18)<>		
Shear	f <sub>v,k</sub>	4.8 (D60)	1.2 ( <d18)< td=""><td>4.0 (D30)</td><td>4.0 (D24)</td></d18)<>	4.0 (D30)	4.0 (D24)		
Stiffness properties (in kN/mm²)							
Mean modulus of elasticity parallel	E <sub>0,mean</sub>	11.3 (D30)	10.9 (D30)	11.3 (D30)	10.9 (D30)		
5% modulus of elasticity parallel	E <sub>0,05</sub>	8.6 (D24)	8.4 (D18)	7.6 ( <d18)< td=""><td>7.3 (<d18)< td=""></d18)<></td></d18)<>	7.3 ( <d18)< td=""></d18)<>		
Mean modulus of elasticity perpendicular	E <sub>90,mean</sub>	1.1 (D60)	2.1 (D18)	0.4 ( <d18)< td=""><td>0.4 (<d18)< td=""></d18)<></td></d18)<>	0.4 ( <d18)< td=""></d18)<>		
Mean shear modulus	G <sub>mean</sub>	-	-	0.7 (D30)	0.7 (D30)		
Density (in kg/m³)							
Density	$\rho_k$	621 (D50)	515 (D24)	621 (D50)	515 (D24)		
Mean density	ρ <sub>mean</sub>	708 (D60)	609 (D40)	708 (D60)	609 (D40)		

#### Influence of heat treatment

The evolution of standard EN 338 mechanical properties with heat treatment is illustrated on Fig. 2. Strength properties are globally affected by heat treatment. Only compression parallel increases by 11%. Similar results have been found by Boonstra (Boonstra et al. 2007) in a previous study. With a decrease of 74%, shear strength is the most negatively impacted mechanical property by heat treatment. Given the uncertainty of measurement, the modulus of elasticity parallel to grain remains unchanged. However the modulus of elasticity perpendicular to grain is increased by 72% after heat treatment. Due to the degradation of hemicelluloses (Boonstra and Tjeersma 2005), density is thus diminished. The increase of the www.proligno.ro

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compressive strength in longitudinal direction might be due to a lower amount of bound water in heat-treated wood. An increased cross-linking of the lignin polymer network could be another reason for this improvement (Boonstra *et al.* 2007). This result may indicate that the lignin polymer network contributes directly to the strength properties of woo (Banoub and Delmas 2003).

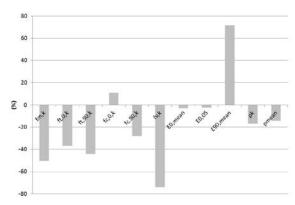


Fig. 2.
Variations of mechanical properties of wood due to heat treatment.

The weakening of shear strength may be due to the formation of radial cracks during thermal treatment which leads to an increased failure when external forces, causing internal stresses, are applied on wood (Boonstra *et al.* 2007). Native ash is assigned to D30 grade if one only considers the bending strength  $f_{m;k}$ , the mean modulus of elasticity  $E_{0;mean}$ , and the density  $\rho_k$  as the classifying parameters, the modulus of elasticity  $E_{0;mean}$  being the limiting parameter. On the other hand, D24 is the grade assigned to the heat-treated ash with the same considerations, the density  $\rho_k$  being the limiting parameter. However if all of the experimental values are taken into account, the grade of non-treated ash remains the same whereas the heat-treated ash is downgraded below D18 because of both compression strength perpendicular to grain  $f_{c:90:k}$  and shear strength  $f_{v:k}$ .

These observations show that heat treatment does not proceed to a simple degradation with the same level on all mechanical properties: some properties are more affected than others by the heat treatment. Similar conclusions were found by Boonstra (Boonstra *et al.* 2007). In the case of heat-treated ash at 210°C, shear strength is the most sensitive property. The density has already been found as an important parameter regarding shear strength. (Stretenovic *et al.* 2004) find a loss of 2N/mm² for a decrease of 0.1g/cm³ concerning spruce and larch wood (Stretenovic *et al.* 2004). In the present work, the shear strength decreases by more than 3N/mm² for a decrease of 0.1g/cm³ due to heat treatment.

Ratio between axial and transverse elastic moduli is used to assess the anisotropy level of the material: an inorganic crystal is supposed to have a ratio r lower than 2. The ratio r being given by Equation 1.

$$r = \frac{\left(\frac{E_{0,mean}}{E_{90,mean}}\right)_{\text{max}}}{\left(\frac{E_{0,mean}}{E_{90,mean}}\right)_{\text{min}}}$$
(Eq.1)

The same ratio is between 12 and 62 for hardwood species. The higher r is, the higher the anisotropy of the material. Values extracted from our tests give a ratio of 20 for non-treated ash, which is within the range given by Kollmann (Kollmann and Côté 1984). However, heat-treated ash has a ratio r=8 that is below the range of all non-treated wood. This is explained by the important increase of the transverse modulus of 164 elasticity  $E_{90;mean}$  while the axial modulus of elasticity  $E_{0;mean}$  does not vary a lot. This observation leads to the conclusion that heat-treated wood is less anistropic than non-treated wood. The increasing crystallinity already observed with heat-treatment of wood can be one reason for the reduction of anisotropy (Bhuiyan *et al.* 2001, Akgül *et al.* 2006).

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#### Mechanical grading according to EN 338

This section compares experimental results with calculated values from EN 338 which provides relationships for all mechanical properties as a function of bending strength, modulus of elasticity parallel to grain and density. These relationships are given in Table 2. Fig. 3 shows differences between our experimental values and calculated values with EN 338. It should first be noticed that differences between experimental values and calculated values still exist for non-treated ash. These differences are particularly great for perpendicular to grain properties  $f_{t:90}$ ,  $f_{c:90}$  and  $E_{90;mean}$ . Other properties are estimated with an error lower than 100%. In any case experimental values are higher than the calculated ones: this means that the European standard EN 338 provides safe values with a view of design.

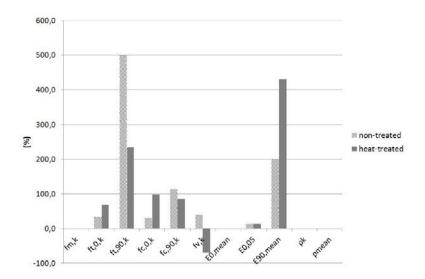


Fig. 3. Variations between experimental values and calculated values from standard EN 338.

Conclusions on heat-treated ash wood are approaching those with non-treated ash: test values of tensile strength f<sub>t:90</sub> and modulus of elasticity perpendicular to grain E<sub>90:mean</sub> are much higher than calculated values with standard EN 338. The observed negative difference with shear strength confirms that this property is the limiting variable for the mechanical grading of heat-treated ash.

EN 338 provides safe characteristic values for native wood. It should be noticed that our tests have been performed on wood clear from defects, whereas relations of standard EN 338 are specified for any piece of wood used for structural purposes which may contain defects or singularities affecting mechanical performances. Moreover, the effect of these natural defects, such as knots, resin pockets, slope of grain and reaction wood, on the strength properties of wood appeared to be amplified by heat treatment (Boonstra et al. 2007).

Results on ash wood confirm previous study of Widmann (Widmann et al. 2012) who has observed similar behavior with heat-treated beech: heat treatment is performed between 180 and 190°C for 16 hours. Conclusions stand that strength properties decrease with the exception of compression parallel to grain, while  $E_{0;mean}$  do not vary and  $E_{90;mean}$  is significantly higher for heat-treated wood.

#### **CONCLUSION**

This study highlights the following points:

- Heat treatment decreases strength properties with the exception of compression parallel to grain, and increases modulus of elasticity perpendicular to grain of ash.
- Mechanical tests fulfilled on small specimens clear from defect of non-treated ash give higher values than estimated values by the EN 338 standard.
- The same equations used to determine a low limit of strength and stiffness properties can be applied to heat-treated wood with the exception of shear strength, which is the property that most suffers from heat treatment, and limits mechanical grading of heat-treated ash.
- Heat treatment performed on hardwood species with low natural decay resistance allows its development in non-structural applications such as decking, cladding, and joinery, where no strong strength resistance is needed. The influence of heat treatment on strength properties points out the need to develop nondestructive controls for other structural uses.

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• Since no standard for visual or machine grading exists for heat-treated wood yet, it is suggested that shear strength should be taken into account for future non-destructive controls. These considerations should help to promote the use of heat-treated hardwood species in outdoor use.

#### **REFERENCES**

Ahajji A, Diouf P, Aloui F, Elbakali I, Perrin D, Merlin A, George B (2009) Influence of heat treatment on antioxidant properties and colour stability of beech and spruce wood and their extractives. Wood Sci Technol, 43:69–83.

Akgül M, Gümüskaya E, Korkut S (2006) Crystalline structure of heat-treated scots pine [pinus sylvestris I.] and uldag fir [abies nordmanniana (stev.) subsp. bornmuelleriana (mattf.)] wood. Wood Sci Technol, 41:281–289

Banoub J, Delmas M (2003) Structural elucidation of the wheat straw lignin polymer by atmospheric pressure chemical ionization mass spectrometry and matrix-assisted laser desorption/ionization time-of-flight mass spectrometry. Journal of mass spectrometry, 38(8):900–903.

Bhuiyan TR, Hirai N, Sobue N (1999) Changes of crystallinity in wood cellulose by heat treatment under dried and moist conditions. J. Wood Sci., 46:431–436.

Bhuiyan TR, Hirai N, Sobue N (2001) Effect of intermittent heat treatment on crystallinity in wood cellulose. J. Wood Sci., 47:336–341.

Boonstra M (2008) A two-stage thermal modification of wood. PhD thesis, University Henri Poincaré - Nancy 1.

Boonstra MJ, Acker JV, Tjeerdsma BF, Kegel EV (2007) Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents. Ann. For. Sci., 64:679–690.

Boonstra MJ and Tjeersma B (2005) Chemical analysis of heat treated softwoods. Holz als Roh und Werkstoff, 64(3):204-211.

Candelier K, Dumarçay S, Pétrissans A, Desharnais L, Gérardin P, Pétrissans M (2013a) Comparison of chemical composition and decay durability of heat treated wood cured under different inert atmospheres: Nitrogen or vacuum. Polymer degradation and Stability, 98:677–681.

Candelier K, Dumarçay S, Pétrissans A, Gérardin P, Pétrissans M (2013b) Comparison of mechanical properties of heat treated beech wood cured under nitrogen or vacuum. Polymer degradation and stability, 98:1762–1765.

FCBA (2011) Perspectives de valorisation de la ressource de bois d'œuvre feuillus en France (Outlook for the promotion of hardwood resource in France). Technical report, FCBA.

FCBA (2013) Fcba - mémento. Technical report, FCBA.

Finnish Thermowood Association (2003) Thermowood handbook.

Kocaefe D, Poncsak S, Boluk Y (2008) Effect of thermal treatment on the chemical composition and mechanical properties of birch and aspen. Bioresources, 3(2):517-537.

Kollmann FF, Côté WA (1984) Principles of wood science and technology, volume I. Springer-Verlag.

Korkut DS, Korkut S, Bekar I, Budakci M, Dilik T, Cakicier N (2008) The effect of heat treatment on the physical properties and surface roughness of turkish hazel (corylus colurna I.) wood. Int. J. Mol. Sci., 9:1772–1783.

Lekounougou S, Kocaefe D, Oumarou N, Kocaefe Y, Poncsak S (2011) Effect of thermal modification on mechanical properties of Canadian white birch (Betula papyrifera). International Wood Products Journal, 2(2):101-107.

Mainguy P (2013) Measure of thermal treatment intensity and mechanical characterization of heat-treated wood [In French]. Master's thesis, Arts et Métiers Paris-Tech.

Metsa-Kortelainen S, Viitanen H (2009) Decay resistance of sapwood and heartwood of untreated and thermally modified scots pine and norway spruce compared with some other wood species. Wood Material Science and Engineering, 4(3-4):105–114.

NF B 51-007 Standard (1985) Wood - axial compression test [In French].

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# **PRO LIGNO**

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NF B 51-010 Standard (1942) Tensile test perpendicular to the fibers [In French].

NF B 51-012 Standard (1942) Wood - Shear test [In French].

NF B 51-017 Standard (1988) Wood - Traction parallel fibers - Determination of breaking strength in tension parallel to the grain of small specimens flawless [In French].

NF EN 338 Standard (2009) Structural timber - Strength classes [In French].

NF EN 408 Standard (2012) Timber structures - Structural timber and glued laminated timber - Determination of some physical and mechanical properties [In French].

Stretenovic A, Müller U, Gindl W (2004) New shear assay for the simultaneous determination of shear strength and shear modulus in solid wood: finite element modeling and experimental results. Wood and Fiber Science, 3(36):302–310.

Welzbacher C, Brischke C, Rapp A (2009) Estimating the heat treatment intensity through various properties of thermally modified timber (tmt). In The international research group on wood protection. IRG/WP 09-40459.

Widmann R, Fernandez-Cabo JL, Steiger R (2012) Mechanical properties of thermally modified beech timber for structural purposes. Eur. J. Wood Prod., 70:775–784.