

Mechanical behavior of heat-treated spruce (*Picea abies*) wood at constant moisture content and ambient humidity

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Abstract The mechanical behavior of heat-treated spruce wood was investigated in relation to the mass loss that occurs during thermal treatment. At constant wood moisture content, the strength, failure strain and toughness of wood were reduced by the heat-bath treatment, decreasing with increasing mass loss. The stiffness was unaffected up to a mass loss of about 3%, and thereafter it decreased. The mechanical properties, however, are not only dependent on the mass loss but also on the relative humidity in the heating atmosphere. As a function of mass loss, the inelastic ductility and the inelastic toughness were the lowest when wood was heated in a dry climate, as compared to a moist climate.

On the other hand, the mechanical properties of heat-treated wood were tested at constant ambient humidity. In such circumstances, the failure strain and the toughness were still reduced, but the strength and the stiffness were actually improved up to a mass loss of about 2%–3%. The improvement is due to the lower equilibrium moisture content of heat-treated wood when placed in service conditions. As a function of mass loss, wood heated at intermediate relative humidity (in the vicinity of 50%) exhibited the best mechanical behavior, which surprisingly included inelastic ductility. This is believed to be due to some irreversible hydrogen bonding.

Mechanisches Verhalten von wärmebehandeltem Fichtenholz (*Picea abies*) bei konstanter Holz- und Luftfeuchte

Zusammenfassung Das mechanische Verhalten von wärmebehandeltem Fichtenholz wurde in Zusammenhang mit dem

bei der Wärmebehandlung auftretenden Masseverlust untersucht. Durch die Wärmebadbehandlung nahmen die Festigkeit, Bruchdehnung und Zähigkeit bei konstanter Holzfeuchte mit zunehmendem Masseverlust ab. Die Steifigkeit nahm erst ab einem Masseverlust von ungefähr 3% ab. Die mechanischen Eigenschaften hängen jedoch nicht nur vom Masseverlust ab, sondern auch von der relativen Luftfeuchte bei der Wärmebehandlung. Bezogen auf den Masseverlust waren der nicht elastische Anteil der Bruchdehnung und der nicht elastische Anteil der Zähigkeit bei Holz, das in trockenem Klima wärmebehandelt wurde, niedriger als bei einer Behandlung in feuchtem Klima.

Daneben wurden die mechanischen Eigenschaften von wärmebehandeltem Holz bei konstanter Luftfeuchte untersucht. Unter diesen Bedingungen nahmen Bruchdehnung sowie Zähigkeit ebenfalls ab, jedoch verbesserte sich die Festigkeit und Steifigkeit bis zu einem Masseverlust von 2%–3%. Dies ist auf die niedrigere Gleichgewichtsfeuchte von wärmebehandeltem Holz unter Praxisbedingungen zurückzuführen. Bezogen auf den Masseverlust zeigte Holz, das bei mittlerer relativer Luftfeuchte (ca. 50%) wärmebehandelt wurde, die besten mechanischen Eigenschaften. Erstaunlicherweise gilt dies auch für den nicht elastischen Anteil der Bruchdehnung, was wahrscheinlich auf irreversible Wasserstoffbindungen zurückzuführen ist.

1 Introduction

Wood is a complex polymeric material constituted mainly of cellulose, hemicelluloses and lignin, with a minor proportion of extractives. The exposure of wood to elevated temperatures causes thermal degradation of its structure, i.e., changes in composition, often accompanied by loss of mass, and thus the properties of wood are somewhat modi-

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fied. Thermal degradation is utilized commercially to produce wood products with improved dimensional stability and reduced hygroscopicity (Bekhta and Niemz 2003, Edwardsen and Sandland 1999, Militz 2002, Stamm et al. 1946, Tjeerdsma et al. 1998, Weiss 1921). A reduction of mechanical properties, along with the mass loss, limits the use of heat-treated wood in structural applications.

The extent of mass loss depends on the process conditions and on the tree species used in the heat treatment. The mass loss increases with increasing the process temperature, duration of the treatment and relative humidity in the heating atmosphere (Alén et al. 2002, Borrega and Kärenlampi 2007, Esteves et al. 2007). Mass loss is mainly due to the degradation of the hemicelluloses, which are the most thermal sensitive polymers of the wood components (Bourgois and Guyonnet 1988, Zaman et al. 2000). Hardwoods contain a higher proportion of hemicelluloses than softwoods and thus are degraded more extensively (Esteves et al. 2007, Zaman et al. 2000). Furthermore, the degradation of hemicelluloses has been shown to correlate well with the loss of strength in wood (Esteves et al. 2007, Sweet and Winandy 1999, Winandy and Lebow 2001).

The reduction of mechanical properties also depends on the tree species and on the process conditions of the heat treatment. According to Bekhta and Niemz (2003), the bending strength of spruce wood decreases nearly 50% as the temperature is raised from 100 °C to 200 °C, whereas the modulus of elasticity (MOE) is hardly affected (4%–9% reduction). Similar results have been reported regarding the mechanical behavior of birch heated at 220–230 °C (Poncsák et al. 2006). Kamdem et al. (2002) studied the mechanical properties of spruce and beech after heat treatment between 200 °C and 260 °C. Spruce showed a reduction in modulus of rupture (MOR) of 8% and in MOE of 11%, whereas beech showed a reduction in MOR of 40% and in MOE of 20%. Tjeerdsma et al. (1998) reported a two-step heat treatment to decrease the MOR of beech and Scots pine by 3% and 20%, respectively. For eucalyptus wood, the MOE after a high-temperature treatment was actually found to be improved (Santos 2000). These observations, although not explicitly specified, appear to correspond to wood tested at constant ambient conditions.

On the other hand, the mechanical properties of wood strongly depend on moisture content, increasing with decreasing moisture content below the fiber saturation point (Gerhards 1982, Haygreen and Bowyer 1996). Since heat-treated wood is less hygroscopic than untreated wood, its mechanical properties in service conditions may even be higher than at constant moisture content.

The objective of this study is to investigate the mechanical behavior of spruce wood subjected to heat-bath treatments under different process conditions. The mechanical

properties of heat-treated wood are analyzed in relation to the mass loss that occurs during the thermal treatment. The mechanical properties are tested at constant moisture content on the one hand, and at constant ambient humidity on the other hand.

2 Experimental

2.1 Material and heat-bath treatments

The wood material used in this study was Norway spruce (*Picea abies*) felled in Joensuu, Finland. Wood specimens with dimensions 320 × 24 × 24 mm³ (longitudinal, radial, tangential) were prepared. All the specimens were clear of visible flaws. Eighteen groups containing 12 specimens each were formed, and each group was subjected to a particular heat-bath treatment. The equipment used was a stainless steel pressure vessel, equipped with a temperature gauge and a pressure gauge. To block direct radiation from the steel onto the specimens, a sheet of aluminum was positioned at the bottom and around the walls of the vessel.

Heat-bath treatments were conducted as described in our previous study (Borrega and Kärenlampi 2007). The specimens were placed in the vessel along with a predetermined amount of water, and the temperature was raised to the setup temperature. Water vapor pressure and consequent relative humidity were determined by subtracting air pressure from the total pressure. Once the setup temperature was reached, the specimens were subjected to an isothermal treatment, at the end of which the vessel was allowed to cool to room temperature. The mass loss of any specimen was determined on a dry mass basis, after oven-drying at 103 °C for 24 hours. The experimental parameters and the mass loss for each treatment are shown in Table 1.

2.2 Mechanical properties

Heat-treated specimens were conditioned in a climate controlled room at 19 °C temperature and 65% relative humidity for a minimum period of 3 months, thus the specimens attaining equilibrium moisture content (EMC). Seven specimens from each group were then placed in a climate chamber at 19 °C temperature, and the air humidity was increased in order to reach an EMC of 10.3 ± 0.1%, which was the EMC of the reference specimens.

Mechanical properties of heat-treated wood were tested at constant moisture content on the one hand and at constant ambient humidity on the other hand. Prior to implementing any bending tests, all the specimens were planed to dimensions of 20 × 20 mm² in cross section. Bending tests were conducted in a three-point bending apparatus with a span

Table 1 Process parameters, mass loss and EMC for each heat-bath treatment. Standard deviation is in parentheses

Tabelle 1 Versuchsparameter, Masseverlust und Gleichgewichtsfeuchte für verschiedene Wärmebadbehandlungen. Standardabweichung in Klammern

Setup temperature [°C]	*Relative humidity [%]/ Vapor pressure [kPa]	Isothermal treatment [h]	Mass loss [%]	**EMC [%]
150	8/38	0	–	9.3 (0.3)
	8/38	2	0.2 (0.3)	9.0 (0.3)
	8/38	8	0.4 (0.3)	8.7 (0.3)
	60/285	0	0.6 (0.2)	7.8 (0.2)
	51/241	2	1.0 (0.2)	7.3 (0.2)
	57/269	8	2.1 (0.3)	6.4 (0.2)
	100/465	0	1.8 (0.5)	8.1 (0.1)
	100/472	2	3.0 (0.7)	7.6 (0.1)
	100/461	8	6.9 (1.3)	6.9 (0.2)
	170	10/85	0	1.5 (0.5)
15/117		2	2.1 (0.7)	7.8 (0.3)
15/117		8	2.8 (0.9)	7.3 (0.3)
55/440		0	2.4 (0.4)	6.5 (0.2)
50/397		2	3.2 (0.5)	6.2 (0.2)
61/486		8	7.2 (0.5)	5.6 (0.1)
100/794		0	6.2 (0.9)	7.2 (0.1)
100/790		2	10.9 (1.0)	6.6 (0.1)
100/802		6	15.3 (0.8)	6.2 (0.1)

* Relative humidity and corresponding vapor pressure computed at the instant of reaching the setup temperature

** EMC of reference specimens is 10.3 ± 0.1 . This value is the mean of 24 observations

length of 300 mm. The displacement of the cross-head was 0.2 mm per second, and the force was applied to the middle of the face of the specimen nearest the pith. The MOR and MOE were determined according to the equations below:

$$MOR = \frac{3P_{max}L}{2bh^2} \tag{1}$$

$$MOE = \frac{PL^3}{4bh^3\delta} \tag{2}$$

where P_{max} is the load at failure, P and δ are any load and its corresponding displacement below the proportional limit, L is the span length, and b and h are the width and the height of the specimen, respectively.

The strain at failure was approximated according to Eq. 3:

$$\epsilon_{max} = \frac{6\delta_{max}h}{L^2} \tag{3}$$

where ϵ_{max} and δ_{max} are the strain and displacement at failure, respectively. The failure strain was then divided into elastic failure strain (ϵ_e) and inelastic failure strain (ϵ_i) as shown in Fig. 1. The elastic failure strain was computed as:

$$\epsilon_e = \frac{MOR}{MOE} \tag{4}$$

and the inelastic failure strain was computed by subtracting the elastic failure strain from the failure strain.

The area under the stress-strain curve up to failure, i.e., toughness of wood, was determined according to Eq. 5:

$$Toughness = \int_0^{\epsilon_{max}} \sigma d\epsilon \tag{5}$$

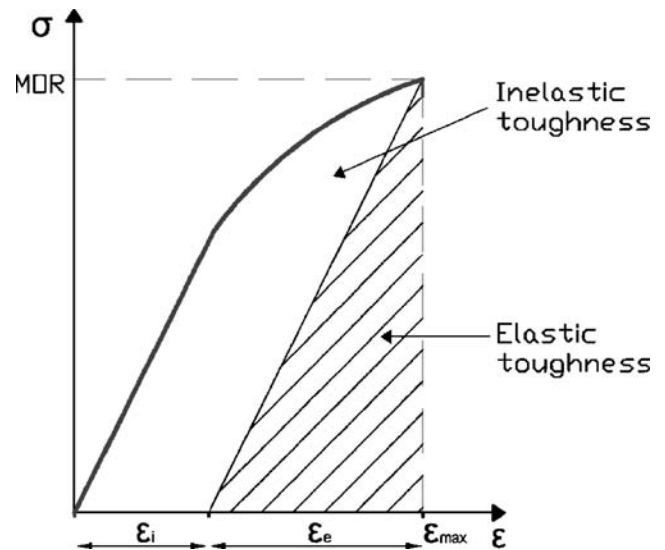


Fig. 1 Stress-strain curve showing the strength (MOR), failure strain (ϵ_{max}), elastic failure strain (ϵ_e), inelastic failure strain (ϵ_i), and elastic and inelastic toughness

Abb. 1 Spannungs-Dehnungs-Verlauf mit Biegefestigkeit, Bruchdehnung (ϵ_{max}), elastischem (ϵ_e) und nicht elastischem (ϵ_i) Anteil der Bruchdehnung sowie elastischem und nicht elastischem Anteil der Zähigkeit

where σ is the stress at strain ϵ . The toughness was then divided into elastic and inelastic toughness (Fig. 1). The elastic toughness was computed according to Eq. 6:

$$\text{Elastic toughness} = \frac{\epsilon_e}{2} MOR \tag{6}$$

and the inelastic toughness was computed by subtracting the elastic toughness from the total toughness.

3 Results and discussion

Mechanical properties of heat-treated wood were analyzed as a function of the mass loss that occurred during the thermal treatment. At constant wood moisture content, the MOR of heat-treated wood was reduced in comparison to that of the reference specimens, decreasing with increasing mass loss (Fig. 2a). Moreover, no differences among treatments were found. The mass loss is believed to be mainly due to the degradation of the hemicelluloses (Bourgois and Guyonnet 1988, Zaman et al. 2000), and thus the results support the idea that hemicelluloses play a major role in the strength of wood (Esteves et al. 2007, Sweet and Winandy 1999, Winandy and Lebow 2001). At constant ambient humidity, however, the MOR of heat-treated wood was im-

proved up to a mass loss of about 2% (Fig. 2b). This may be explained by the lower EMC of heat-treated wood when placed in service conditions, as shown in Table 1. As a function of mass loss, wood heated at intermediate relative humidity (in the range 50%–61%) attains the lowest EMC (Borrega and Kärenlampi 2007), and accordingly, this material appears to be the strongest (Fig. 2b).

Heat treatments have been reported to have a stronger impact on the strength than on the stiffness of wood (Bekhta and Niemz 2003, Esteves et al. 2007, Poncsák et al. 2006). This is also clearly observed in Fig. 2c. At constant wood moisture content, the MOE of heat-treated wood was 0.9–1.1 times that of the reference specimens up to a mass loss of about 3%, and thereafter it decreased. This indicates that stiffness is less sensitive than strength to the degradation of

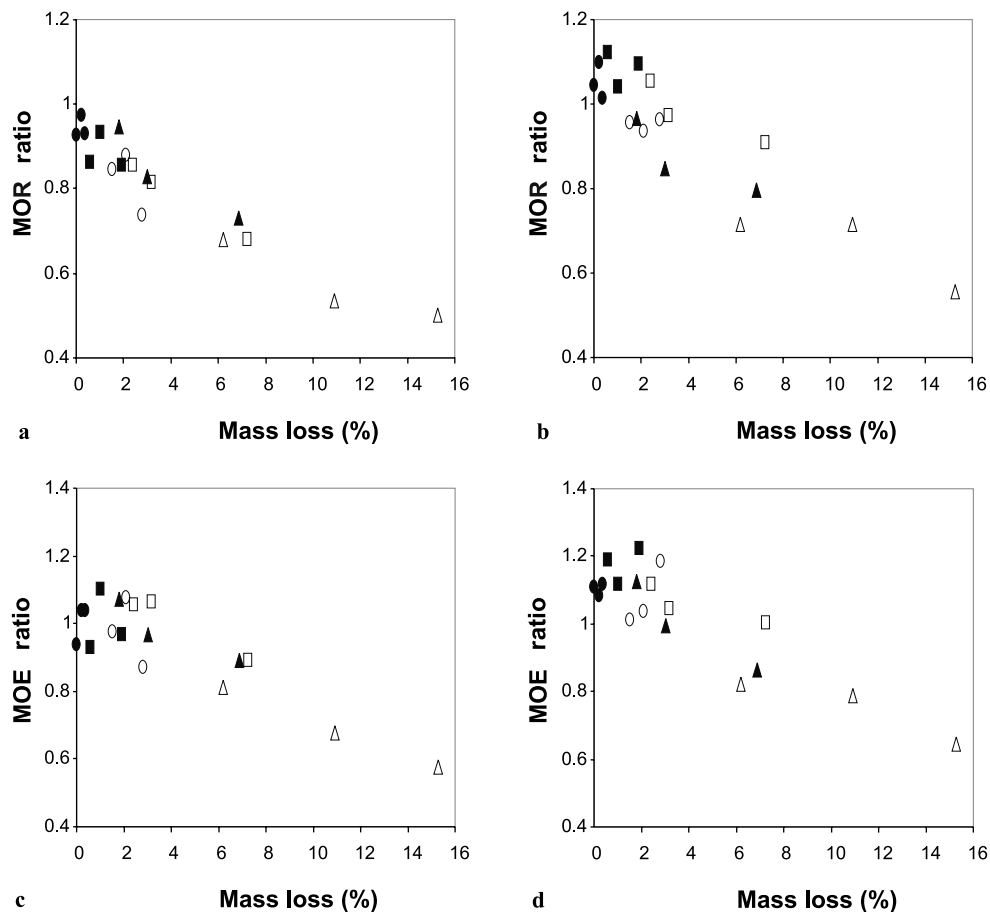


Fig. 2 Ratio of MOR and MOE of heat-treated wood to that of reference specimens as a function of mass loss. **a** MOR at constant wood moisture content. **b** MOR at constant ambient humidity. **c** MOE at constant wood moisture content. **d** MOE at constant ambient humidity. *Filled circles*, dry conditions at 150 °C; *open circles*, dry conditions at 170 °C; *filled squares*, intermediate relative humidity at 150 °C; *open squares*, intermediate relative humidity at 170 °C; *filled triangles*, water-saturated conditions at 150 °C; *open triangles*, water-saturated conditions at 170 °C

Abb. 2 Relative Biegefestigkeit und Elastizitätsmodul von wärmebehandeltem Holz im Vergleich zu Kontrollproben in Abhängigkeit vom Masseverlust. **a** Biegefestigkeit bei konstanter Holzfeuchte, **b** Biegefestigkeit bei konstanter Luftfeuchte, **c** Elastizitätsmodul bei konstanter Holzfeuchte, **d** Elastizitätsmodul bei konstanter Luftfeuchte. *Ausgefüllter Kreis*: trockenes Klima, 150 °C; *Leerer Kreis*: Trockenes Klima, 170 °C; *Ausgefülltes Rechteck*: mittlere relative Luftfeuchte, 150 °C; *Leeres Rechteck*: mittlere relative Luftfeuchte, 170 °C; *Ausgefülltes Dreieck*: wassergesättigt, 150 °C; *Leeres Dreieck*: wassergesättigt, 170 °C

the hemicelluloses. When heat-treated wood was tested at constant ambient humidity, the MOE was up to 20% higher for a mass loss up to 3% (Fig. 2d). As in the case of MOR, the increase in stiffness may be attributed to the lower EMC of heat-treated wood in service conditions.

The amount of energy absorbed before failure, or toughness, can be computed as the area under the stress-strain curve. Kubojima et al. (2000) reported that the reduction in toughness of heat-treated wood can be attributed to its lower plastic ductility. We have computed the elastic and inelastic components of the failure strain and toughness according to Fig. 1. At both constant wood moisture content and constant ambient humidity, the elastic failure strain of heat-treated wood was within the range of 0.7–1 times that of the reference specimens (Figs. 3a and 3b). However, the elastic failure strain at constant ambient humidity (Fig. 3b) appears to be greater than at constant moisture content (Fig. 3a). This seems to indicate that elastic ductility increases with decreasing moisture content. On the other hand, no major differences were found among treatments regarding the elastic failure strain, whilst the inelastic failure strain was clearly lower for wood heated in a dry climate (relative humidity range 8%–15%), as shown in Figs. 3c

and 3d. At constant moisture content, it is interesting to observe that wood heated at intermediate relative humidity showed higher inelastic ductility than that of the reference specimens (Fig. 3c). At constant ambient humidity, the inelastic ductility was reduced, being predominantly less than that of the reference specimens (Fig. 3d).

The effect of mass loss on the elastic toughness of heat-treated wood is shown in Figs. 4a and 4b. At constant wood moisture content, the elastic toughness decreased with increasing mass loss, irrespective of the process conditions (Fig. 4a). The elastic toughness was computed according to Eq. 6. Therefore, the result is a direct consequence of the MOR and the elastic failure strain decreasing with increasing mass loss, and no differences being found among treatments (Figs. 2a and 3a). At constant ambient humidity, the MOR as a function of mass loss was the highest for wood heated at intermediate relative humidity, whereas the elastic failure strain did not differ much among treatments (Figs. 2b and 3b). Consequently, as a function of mass loss, the elastic toughness was the highest for wood heated at intermediate relative humidity (Fig. 4b). At both constant wood moisture content and constant ambient humidity, the inelastic toughness was predominantly reduced by the heat-bath treatment

Fig. 3 Ratio of elastic and inelastic failure strain of heat-treated wood to that of reference specimens as a function of mass loss. **a** Elastic failure strain at constant wood moisture content. **b** Elastic failure strain at constant ambient humidity. **c** Inelastic failure strain at constant wood moisture content. **d** Inelastic failure strain at constant ambient humidity. Symbols are the same as in Fig. 2

Abb. 3 Relativer elastischer und nicht elastischer Anteil der Bruchdehnung von wärmebehandeltem Holz im Vergleich zu Kontrollproben in Abhängigkeit vom Masseverlust

a Elastischer Anteil bei konstanter Holzfeuchte

b Elastischer Anteil bei konstanter Luftfeuchte, **c** Nicht elastischer Anteil bei konstanter Holzfeuchte, **d** Nicht elastischer Anteil bei konstanter Luftfeuchte. Die Symbole sind identisch mit denen in Abb. 2

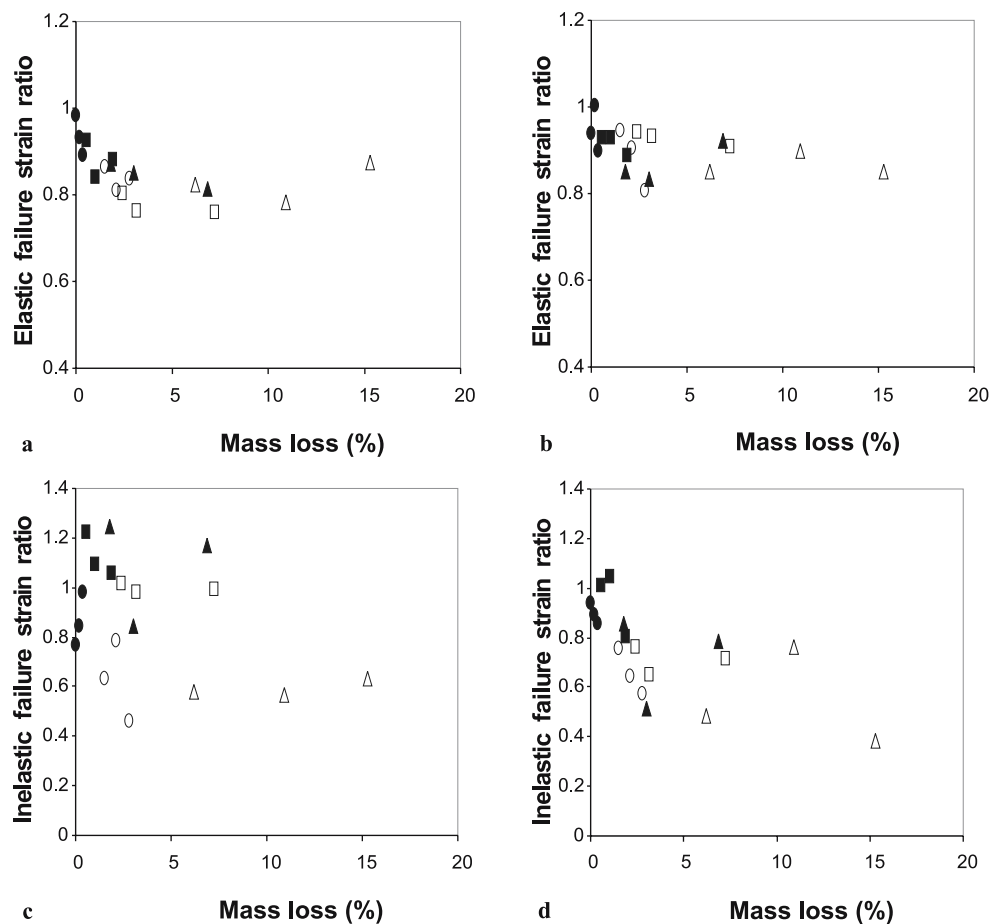
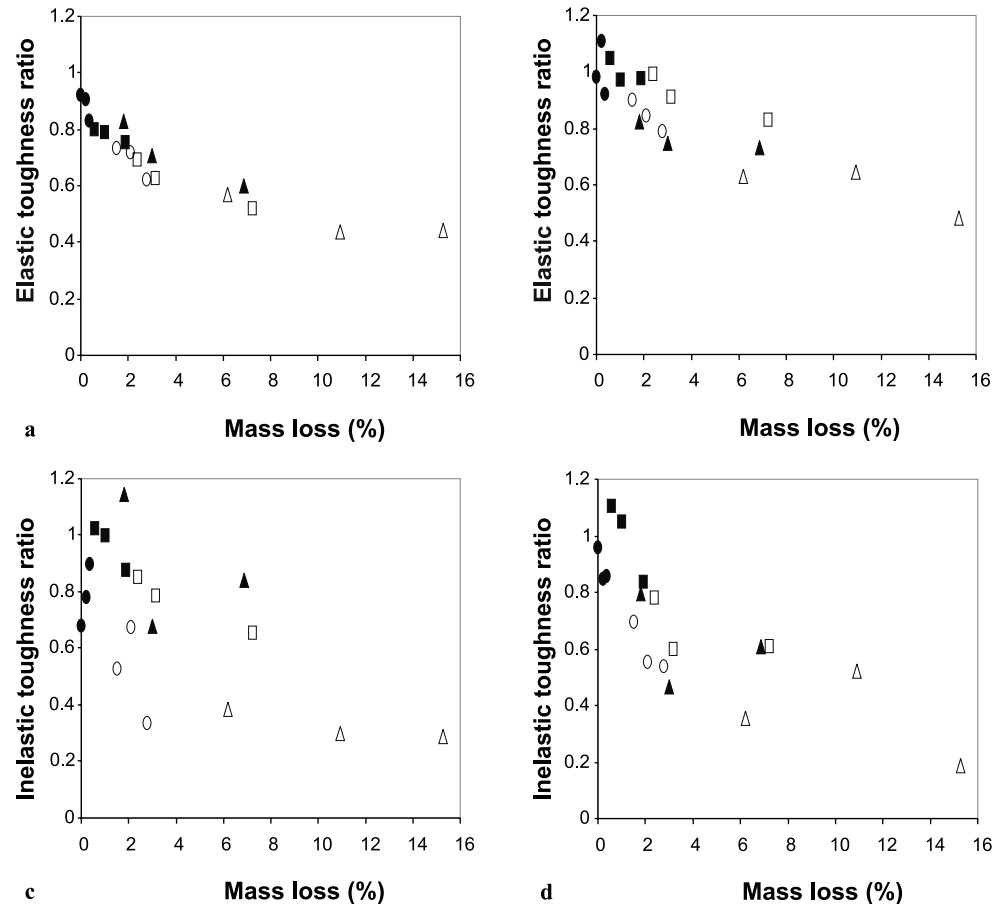


Fig. 4 Ratio of elastic and inelastic toughness of heat-treated wood to that of reference specimens as a function of mass loss.
a Elastic toughness at constant wood moisture content. **b** Elastic toughness at constant ambient humidity. **c** Inelastic toughness at constant wood moisture content. **d** Inelastic toughness at constant ambient humidity. Symbols are the same as in Fig. 2
Abb. 4 Relativer elastischer und nicht elastischer Anteil der Zähigkeit von wärmebehandeltem Holz im Vergleich zu Kontrollproben in Abhängigkeit vom Masseverlust
a Elastischer Anteil bei konstanter Holzfeuchte **b** Elastischer Anteil bei konstanter Luftfeuchte, **c** Nicht elastischer Anteil bei konstanter Holzfeuchte, **d** Nicht elastischer Anteil bei konstanter Luftfeuchte. Die Symbole sind identisch mit denen in Abb. 2



(Figs. 4c and 4d). Moreover, as a function of mass loss, the inelastic toughness was the lowest for wood heated in a dry climate, which is in accordance with its lower inelastic ductility.

We have found that specimens treated at intermediate relative humidity, when tested at constant ambient humidity, are the strongest and the stiffest (Figs. 2b and 2d). This is likely to be related to the hygroscopicity of these specimens being reduced most by the heat-bath treatment (Table 1). However, these specimens also show the greatest inelastic ductility and inelastic toughness (Figs. 3d and 4d). Thus the greater mechanical performance of wood heated at intermediate relative humidity hardly can be solely explained by the reduction of equilibrium moisture content (cf. Obataya et al. 2006, Stone 1955). It has been suggested that irreversible hydrogen bonding occurs during a sequence of wetting and drying processes within the porous structure of the cell wall (Borrega and Kärenlampi 2007), in particular in the case of experiments at intermediate relative humidity, where added free water first moisturizes the specimens, and increased temperature then induces drying of the specimens. The formation of irreversible hydrogen bonding may account for the greater inelastic ductility of wood treated at intermediate relative humidity.

It is also found that the modulus of elasticity, in comparison to that of the reference specimens, is significantly higher than the corresponding modulus of rupture (Fig. 2). We suspect that the degradation of structural components produces stress-enhancing voids within the cell wall, thus reducing strength in relation to stiffness. Furthermore, the inelastic ductility and the inelastic toughness are clearly the worst when the specimens are heated in a dry climate (Fig. 3c, 3d, 4c and 4d). This may be related to the fact that the presence of water during thermal treatments reduces the glass transition temperature of the wood polymers (Back and Salmén 1982). In dry conditions, the molecular chains are quite stiff, and thus their mobility is rather limited. However, the authors would like to invite any suggestions regarding the mechanism of the effect of relative humidity on inelastic ductility.

4 Conclusions

At constant moisture content, the mechanical properties of wood such as strength, failure strain and toughness are mostly reduced by the heat-bath treatment, decreasing with increasing mass loss. The stiffness appears to be unaffected up to a mass loss of about 3%, and thereafter it decreases. As

a function of mass loss, no differences among treatments are found for the reduction in strength, stiffness, elastic ductility and elastic toughness. However, the inelastic ductility and the inelastic toughness are the lowest when the treatment is conducted in dry conditions, as compared to moist conditions.

At constant ambient humidity, the failure strain and the toughness of wood are still mostly reduced by the heat-bath treatment, decreasing with increasing mass loss. However, the strength and the stiffness are improved up to a mass loss of about 2%–3%. This is at least partly due to the lower equilibrium moisture content of heat-treated wood when placed in service conditions. As a function of mass loss, wood heated at intermediate relative humidity exhibits the best mechanical performance, which surprisingly includes inelastic ductility. In addition, wood heated at intermediate relative humidity has the additional benefit of attaining a significantly lower EMC.

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