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Strenght and Colour Response of Solid Wood to Heat Treatment

Dennis Johansson

Luleå University of Technology
Department of Skellefteå Campus,
Division of Wood Technology

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Abstract

Heat treatment is a method for improving the durability and dimensional stability of wood. The treatment method used in this work is called Thermo Wood and is industrially applied in Finland. In 2004 production was 31146 m^3. The method involves heating wood in a steam atmosphere where the heat causes chemical changes in the structure of the wood. The present work was done in order to improve the quality of the treated timber both by predicting strength through colour measurement and by understanding the mechanisms behind the internal checking in heat-treated wood. Besides the published papers there are some smaller unpublished studies presented in the thesis.

In paper I the connection between colour and the strength reduction caused by the heat treatment is studied. The results show that colour measurements are not a good way of predicting strength loss on an industrial scale. However, they also show that on an experimental level it is an effective way to study the changes that occur in the wood during treatment.

There is a problem with internal checking when heat treatment is performed on boards with dimensions greater than about 50 mm. In paper II the connection between internal checking and both mass loss and drying was investigated. The results showed that both mass loss and drying influence the development of internal checking.

Paper III focuses on the effects of drying on internal checking. From studying the results from papers II and III together with the results of some of the unpublished studies, the conclusion is that drying stress is the main cause for internal checking in heat-treated wood.

Preface

As a child I wanted to become a scientist or a rich businessman. Then, when I was fourteen, I realised that I really enjoyed going to school and learning new things all the time. So, one day I came home and told my mother that I was going to remain a student my whole life. Now sixteen years later I am a doctoral student, which means I can both do research and be a student. I haven't become rich, but I am getting paid while I am fulfilling the other two dreams, which must be considered a success. Therefore, I would like to thank Vinnova and the industrial consortium (Valutec, Stora Enso, Södra, Nordiskt Rostat Trä, Norra Skogsägarna, Martinsons Trä and IUC Trä) that have made this possible by financing my project.

The work was carried out at Luleå University of Technology, Division of Wood Science and Technology. I would like to thank my supervisor Professor Tom Morén for supporting me and trusting me enough to let me take the initiative and make my own decisions. I would like to give special thanks to everybody who has contributed to my work: Margot Sehlstedt, Joakim Wänstedt, Stefan Huszics, Aleksandra Igumnova and Yu Chen. Also thanks to Brian Reedy for reviewing the language in this thesis.

I would also like to take the opportunity to give a big thanks to Margit Sandström, Monica Tjerngren and Eva-Stina Nordlund for all their help with the administrative part of this work.

Finally, huge thanks to my beloved wife Anna-Lena and my daughters Tyra and Line for making it all worthwhile.

Skellefteå, November 2005

Dennis Johansson

List of papers

I. Johansson D, Morén T. *The potential of colour measurement for strength prediction of thermally treated wood.* Accepted for publication in Holz als Roh- und Werkstoff.

II Johansson D. (2005). *Drying and Heat Treatment of Wood: Influences on Internal Checking.* 3rd Nordic Drying Conference, Karlstad, Sweden.

III Johansson D, *Possible causes of internal checking of spruce treated above 200°C.* Submitted to Holzforschung.

Contents

Background

Historically, wood has been used for everything from needles to building churches. One might think that today's new technology and the development of new materials would make wood obsolete. But despite these new competitors, wood is still used on a very large scale. The world production of sawn timber amounted to 299 million m^3 in 2002 (*The Swedish Forest Industries 2004 – Facts and figures* 2005).

However, there are some obstacles when working with wood, and most of them are associated with its hydroscopic properties in combination with anisotropic swelling and shrinkage due to changes in moisture content (MC). In addition to this problematic dimensional instability there are other moisture-related problems, such as biological attacks. MC has a major affect on the durability of wood, since fungi and bacteria are dependent on the presence of water for their survival. As long as the MC is kept under about 20% MC there will not be a sufficient amount of water for either fungi or bacteria to grow. To physically protect wood from getting in contact with water after the lumber has been dried to below 20% MC is an environmentally friendly and efficient way of protecting it from biological degradation. This kind of protection is not always easy to accomplish, for instance when wood is used outdoors.

Impregnation of the wood with chemical substances toxic to fungi and bacteria is one of the commonly used methods of preservation. One major drawback with this method is that it is toxic to many other organisms as well. These preservatives do not prevent problems with dimensional instability. There are, however, methods to stabilize lumber chemically, but these are very expensive and the substances used can also in some cases be questionable in terms of their environmental effects.

The last decades have seen an increasing environmental awareness leading to more and more preservatives being prohibited. According to the Swedish Chemicals Inspectorate there are 92 legal preservatives in Sweden today, and 117 different preservatives have been banned since 1990. This has opened a new market for environmentally friendly ways of protecting lumber against biological degradation and increasing its dimensional stability.

Some articles and patents on how to improve durability and dimensional stability of lumber by only using heat were presented during the first half of the previous century (Stamm and Hansen 1937; Stamm, Burr and Kline 1946; Seborg, Millet and Stamm 1948; Thunell and Elken 1948). However, none of these methods resulted in a commercial breakthrough.

During the nineties, the market grew, and at present there are at least five different heat-treatment methods developed in Europe. One common problem with these methods is the risk of wood catching fire. In order to prevent this, oxygen levels need to be kept low. How this displacement of oxygen is solved is different for all these methods.

- Netherlands – Plato process
- France – Retification
- France – Les Bois Perdure
- Germany – Oil heat treatment
- Finland – ThermoWood

Plato Process

The Plato process (Ruyter 1989; Boonstra, Tjeerdsma and Groeneveld 1998) is a two-step process. The first step is performed on wood saturated with water in a hydrothermal treatment at 160°C–190°C at superatmospheric pressure. The second step is a curing step performed at 170°C–190°C in atmospheric pressure without air present, but before this step the wood has to be dried to about 10% MC.

Retification

The Retification process is performed at 210°C–240°C in a nitrogen atmosphere containing no more than 2% oxygen and is done on predried (12% MC) wood (Vernois 2000).

Les Bois Perdure

The Les Bois Perdure process starts with a drying step for green wood. After initial drying, the wood is heated in a steam environment generated by the water in the wood (Vernois 2000).

Oil heat treatment

In this method the treatment is performed in a hot oil bath. Typical treatment temperature is 180°C–220°C (Rapp and Sailer 2001). Tests are being performed on how this process can be further improved by using oil with chemically active groups added and thereby performing a combination of both chemical modification and heat treatment (Tjeerdsma *et al.* 2005).

ThermoWood

The ThermoWood treatment was developed by VTT in Finland (Viitaniemi *et al.* 1996). The process can be performed on green as well as predried wood. The first part of the process is drying and is followed by heat treatment in a steam environment at atmospheric pressure. The Thermowood method is the method that has received the biggest commercial breakthrough, and the production in 2004 was 31146 m^3 (*Thermowood Handbook* 2003). This is the method used for all work done in this thesis.

Property changes

One of the original reasons to heat-treat wood was to improve the properties of durability. Some laboratory studies has shown durability properties compared to CCA impregnation (Viitanen *et al.* 1994). Other studies have shown an improvement in the durability properties of wood, but it should not be used with ground contact and is not likely to be able to replace CCA impregnations (Kamden *et al.* 1999; Edlund 2003; Epmeier *et al.* 2003). Another problem is that high durability requires high treatment temperatures, which means greater strength loss.

In a review by Viitaniemi (1996) (cited in Viitaniemi and Jämsä 1996) it is concluded that the strength loss is from 0%–30% depending on treatment process. However, the studies in this review only look at small specimens. This is because if the small samples are studied, more information on the changes in the wood is obtained, while testing of full-length beams is more a measurement of the weak points in the beam. According to (Bengtsson, Jermer and Brem 2002), strength reduction was about 50% when testing full length spruce and pine beams treated at 220°C for 5 hours. This is reasonable since the knots are highly affected by the heat treatment and are therefore going to weaken the beams. The fifths percentile was reduced by 66% for spruce and 55% for pine. The fifths percentile is the value used for calculation in constructions.

Another important reason for heat treatment is to obtain a more dimensionally stable material by reducing the equilibrium moisture content (EMC). The reduction of the EMC is about 0%–50% depending on how the treatment is performed (Thunell and Elken 1948; Schneider and Rusche 1973; Chirkova *et al.* 2005). But there are also some controversial reports on the reduction of sorption. According to Obataya *et al.* (2000) the original sorption can be almost fully restored by posttreatments such as boiling. Metsä-Kortelainen (2005) shows that sapwood of pine actually increases in water absorption when floating in water after treatment at low temperature (below 210°C).

After heat treatment the wood acquires a darker colour. This colour change is often seen as a positive effect, especially in hardwoods. The colour has the potential of reaching new markets where hardwoods that are seen as more exclusive are normally used. It has also been suggested that colour has the potential of predicting the quality of heat treatment by predicting different property changes such as chemical changes (Bourgois, Janin and Guyonnet 1991), strength loss (Bekhta and Niemz 2003) and mass loss due to thermal degradation (Patzelt, Emsenhuber and Stingl 2003).

Objectives

The main objective of this work has been to improve the quality of heat-treated wood by getting a better understanding of the process and of the material's responses. This includes an understanding of how to avoid defects as well as studying how to predict material responses. In paper I the objective is to study colour change and the use of colour as a predictor of strength. The objective of paper II and paper III is to study the reason for internal checking in heat-treated wood.

The process

Two different ways of describing the heat-treatment process have been used. In papers II and III the process schedule was divided into five parts as shown in figure 1. In paper I regimes II and III were regarded as one and the same.

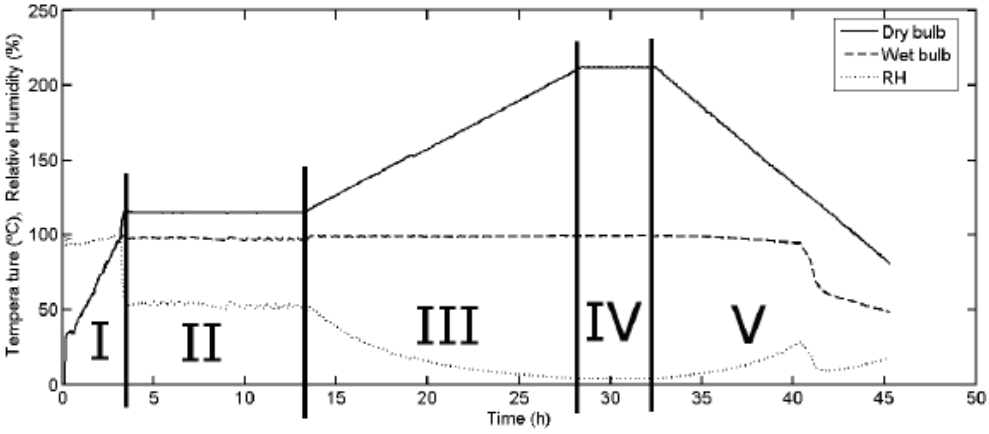

Figure 1: *Process schedules used in the experiment. Dotted line shows relative humidity. Process is divided into five regimes. I) Heating regime II) 1st Drying regime III) 2nd drying regime IV) Treatment regime V) Cooling regime (Figure from paper II)*

I) Heating regime: Heating with saturated steam generated at 130°C injected into the kiln at atmospheric pressure.
II) 1st drying regime: Drying step which can either be high- or low-temperature drying (above or below 100°C).
III) 2nd drying regime: The regime where the final drying takes place. During this regime the chemical reactions accelerate.
IV) Treatment regime: The temperature is kept constant, normally for 2–4 hours. Time and temperature during regime IV normally define the heat-treatment class.
V) Cooling regime: Cooling the wood with steam at 130°C followed by water spraying until the kiln is opened. In industrial practice it is common to include a conditioning regime for remoistening. The conditioning regime has only been used in paper I.

According to Stamm (1956) thermal degradation leading to strength loss is accelerated in the presence of oxygen, moisture and in a closed system. This means that the steam not only protects against fire, it also means less oxidation. Steam is continuously added, which means there will be a reduction of problems related to closed systems, since acidity will therefore not increase as much. The importance of keeping the pH low is also pointed out in Sundqvist, Westermark and Eriksson (2004) who show correlation between acidity

and shortening of the length of the cellulose chains. The shortening was 53% in the sample of Thermowood, which means it is believed it might affect the strength of the wood.

The relative humidity (RH) in this method is low, even though steam is used as a shielding gas. RH is calculated by dividing the partial pressure with the steam saturation pressure at the present dry-bulb temperature. For example, if the process is running at a dry-bulb temperature of 120°C and wet-bulb temperature of 100°C, the saturation pressure at 120°C is about 2 bars. If the wet bulb is 100°C at atmospheric pressure, it means there is practically only steam there, which means the partial pressure is the same as the atmospheric pressure, which is about 1 bar. This means that at 120°C/100°C (dry bulb/wet bulb) the RH is approximately 50%.

The kiln

The steam is generated at about 3.5 bars in a steam boiler connected to the kiln. At all times when dry-bulb temperature is above 130°C, steam is continuously injected to the kiln. Inside the kiln, superheated steam is created by further heating with the heating coil. The superheated steam then works as a shielding gas. The kiln used in this thesis is shown in figure 2. It is a small experimental kiln, which limits the batch sizes to a maximum of 500 x 370 x 2500 mm. The kiln is placed inside a shipping container situated at the Luleå University of Technology campus in Skellefteå.

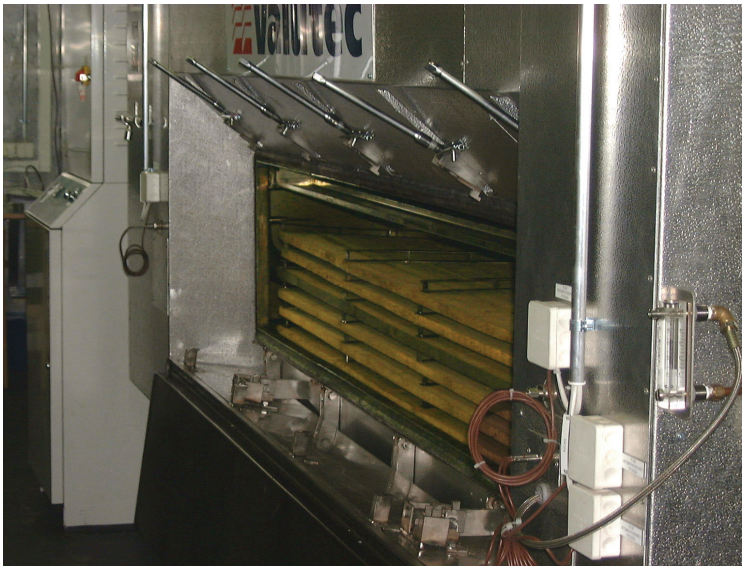

Figure 2: *The experimental kiln used for heat treatments at Luleå University of Technology. Due to the size of the kiln, the batch size is limited to less than half a cubic meter.*

In figure 3 it can be seen that the kiln in its basic design and features is very similar to an ordinary lumber-drying kiln. Obviously, there are differences, for instance the size and the fact that it can withstand temperatures of 230°C. Industrially, the direction of the airflow is alternated. This is done to achieve a more even drying and treatment. But since the experimental kiln used in this work is fitted with a radial fan, this is not possible to do. The small kiln size and the fact that it is not an energy-intensive process should mean that the air reversal is of minor importance. The idea that it is not an energy-intensive process can at first seem a bit strange. The heat capacity of wood is 1.36 kJ/kg which means it would take 272 kJ/kg to heat dry wood up 200°C. If this is compared to 2260 kJ/kg, which is the evaporation energy of water, it is easy to see that drying consumes much more energy. In order to make the kiln this small, the air flows along the boards instead of across them as in an ordinary kiln. For this purpose, special metal stickers with air passages were designed.

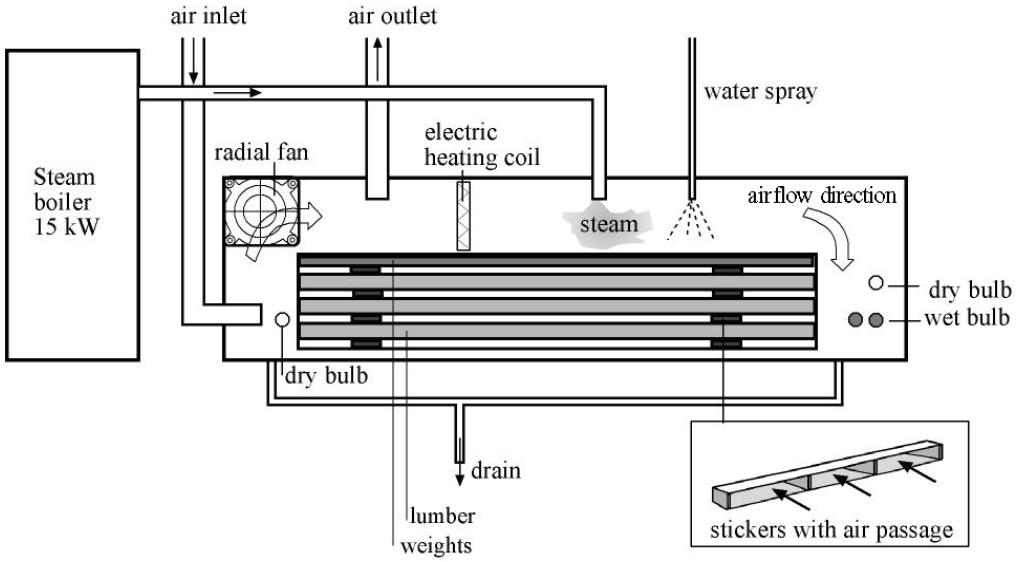

Figure 3: *Graphical description of the kiln. All basic functions of an industrial drying kiln are present. The radial fan makes it impossible to reverse the airflow as is done industrially. Due to the limited size, stickers with air passages were designed. (Figure from papers I & II)*

The rather extreme conditions during treatment make the process complicated to study. There are, for instance, not many sensors that can withstand the acidity, water vapour and high temperature. The heat also makes it impossible to remove sample from a running process. Even if it were possible to take samples out, it would be hard to tell what the effects of the sudden change in climate would be.

Prediction of strength

Strength loss due to heat treatment does not necessarily need to be a big problem as long it is possible to predict it. If predictions of strength can be made with good accuracy, the design can be changed to suite the new properties. In paper I, different ways of predicting strength in heat-treated birch were studied. The predictions were made for small, clear wood samples in both static and impact bending. The multivariate statistical method Partial Least Square (PLS) was used for establishing the prediction models.

One of the objectives with paper I was to study if colour could have potential for strength prediction industrially. Therefore, two models were created for both static and impact bending; i.e., one based on colour measurements and the other on the process parameters. Apart from the colour and the process parameters the models were also based on density, EMC, position in the board and size of specimen. In static bending, the modulus of elasticity (MOE) was also used.

The conclusion of paper I was that colour measurements are not suitable for strength prediction industrially. It is possible to use colour as a predictor, but the process parameters give better predictions. The model based on process parameters also confirms earlier studies (Stamm 1956) showing that temperature during treatment affects thermal degradation much more than treatment time. It was not possible to achieve a working prediction model for impact bending, probably due to the fact that MOE was not included in the model. It was included in the static bending model where it was the most important factor for describing the variation in bending strength.

Matched samples

In addition to the samples tested for the predictions models, matched samples were tested to get a sense of the level of strength reduction. Some of the results from the matched-sample testing are presented in paper I. The results presented are for heat-treated birch, but pine and spruce were also studied. In table 1, all results from the matched-sample testing are presented.

Table 1: *Results from static bending testing with matched samples. Each pair contained one untreated sample and one sample treated at 200°C. The testing was done with matched samples.*

Species	Number of pairs	Strength Untreated	Strength Treated	Strength loss	MOE untreated	MOE treated	MOE	Density loss
		[MPa]	[MPa]	[MPa] (%)	[MPa]	[MPa]	[MPa] (%)	[kg/m^3] (%)
Birch	20	145	82	62 (43)	13765	13500	265 (1.9)	41 (7.4)
Pine	20	93	73	20 (21)	9572	9206	366 (3.8)	25 (6.0)
Spruce	20	104	79	25 (24)	12441	12246	195 (1.6)	9 (2.1)

All three species in the matched-sample test displayed a significant loss in strength and density, but not in MOE. The results show that the process affects different species in different ways, probably due to their differences in chemical composition.

Fracture due to shearing

Even though no prediction models for strength were achievable for impact bending, it still resulted in some interesting findings. During testing, the samples sometimes acquired fractures with shearing to one of the end surfaces (figure 4), and every time this happened, it was recorded.

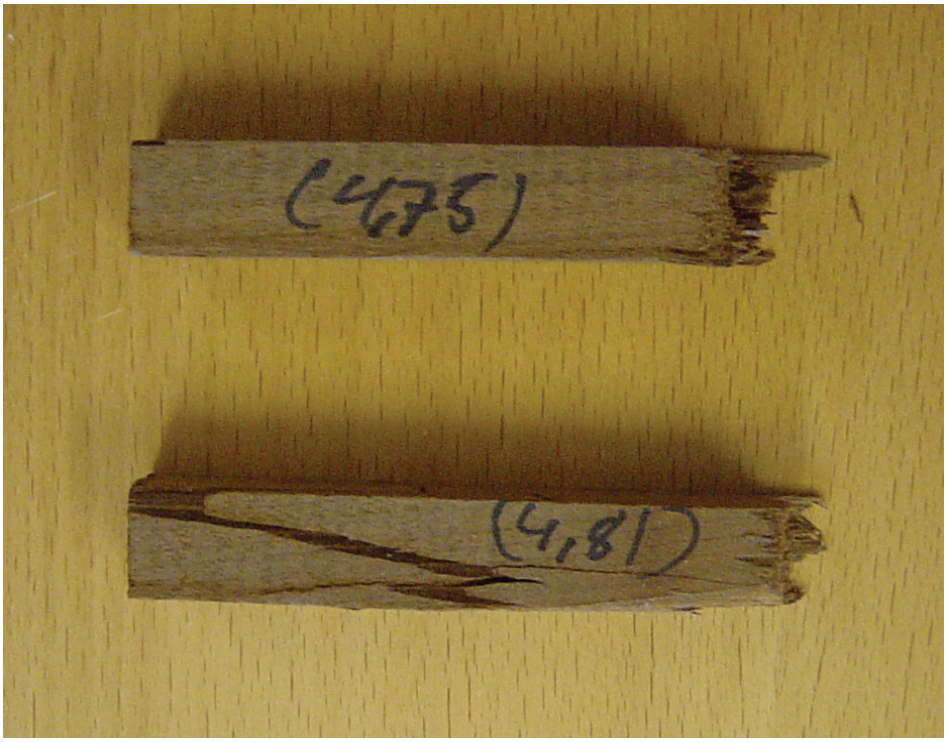

Figure 4: *The two different types of fractures defined in the impact testing. Top: ordinary fracture; bottom: shear fracture.*

There is a clear pattern in the distribution of shear fractures shown in table 2. Higher temperatures and longer treatment time spans result in a higher percentage of shear fractures. The change in type of fracture is probably due to a reduction in strength between the fibres. This indicates that strength reduction is a complex subject that should be studied further, since it shows that even if the same method is used, different types of strength might be measured. If the difference in fractural behaviour is understood, then an improved selection of raw material could give higher and more predictable strength.

Table 2: *Percentage of tests in impact bending resulting in shear fracture to one of the end surfaces.*

Treatment temperature [C]	**Treatment time [h]**	**Fracture to end surface [%]**
175	0	0
175	1	5.5
175	3	4.4
175	10	5.5
185	2	0
200	0	0
200	1	13.9
200	3	27.8
200	10	44.4

Colour heterogeneity

In paper I, a method is described for determining if the colour after treatment is homogeneous. Colour heterogeneity (defined as inhomogeneity in paper I) was measured by making colour measurements with a Minolta Chroma Meter CR310 at different levels in a board (figure 5).

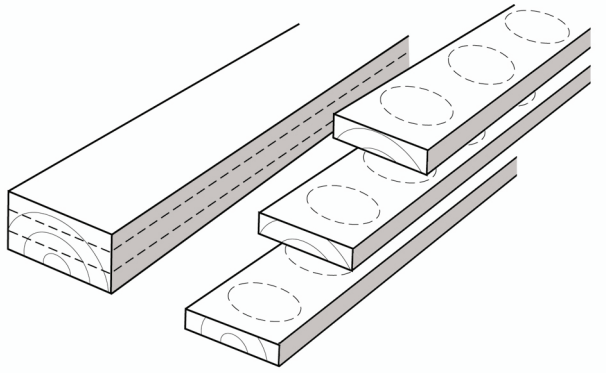

Figure 5: *Cutting of board to measure colour through the board's thickness. Colour measurements were performed on both sides of every slice through the board.*

The Minolta measures the colour as three coordinates in three-dimensional colour space, as shown in figure 6. This system is called CIE L*a*b* and works according to the CIE standard (Hunt 1995). The part of the coordinate system that is of interest in this work is the first quadrant; i.e., positive values of a* and b*.

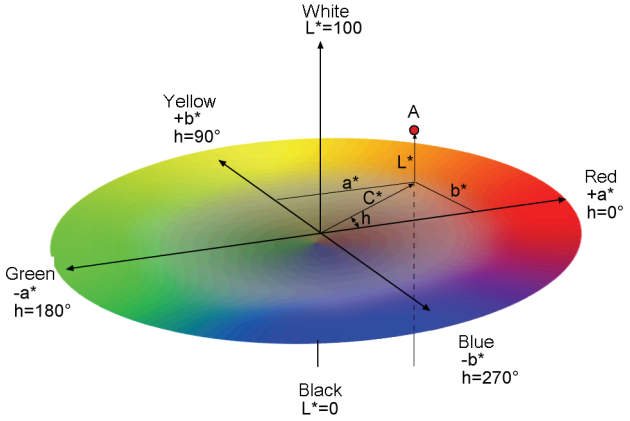

Figure 6: *The three-dimensional CIEL*a*b* colour space. The L*coordinate is the level of lightness. The a* and b*coordinates give the colour of the measurement. The figure also show how the colour can be described with the polar coordinates C* (saturation) and h (hue angle).*

Each of the colour coordinates was plotted against the distance to one of the surfaces of the board. They were then fitted with a second-degree polynomial in the same way as the example of the L coordinate in figure 7. It was assumed that an ideal treatment would generate a colour change through the board linearly for each colour coordinate. The

heterogeneity was defined as the total colour difference between the measurement and the ideal treatment. In order to calculate this difference, the constant and linear part of the second-degree polynomial has to be excluded. Since this is done for all of the coordinates, it is possible to calculate the total colour difference due to heterogeneity. All values are calculated for a distance of 22 mm.

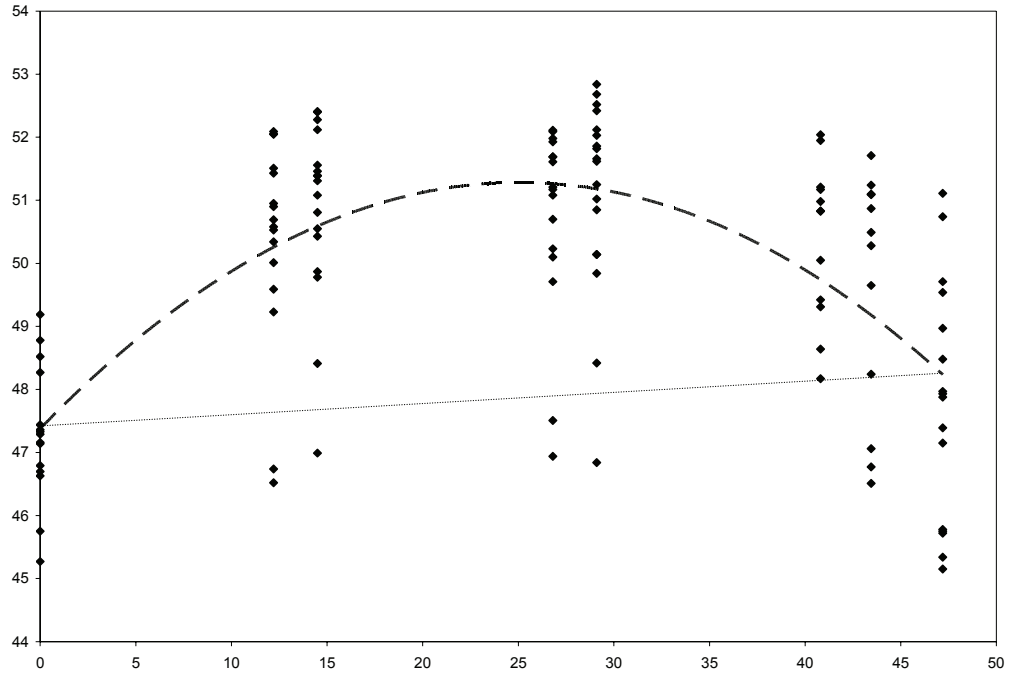

Figure 7: *The L coordinate measured at eight positions through a board. The dashed line shows the second degree polynomial fitted to the measurement, and the solid line the linear part of the polynomial.*

From the measurement performed in paper I it is possible to conclude that there is a difference in colour heterogeneity. Heat-treated boards showed five times higher heterogeneity values than untreated ones (see figure 8).

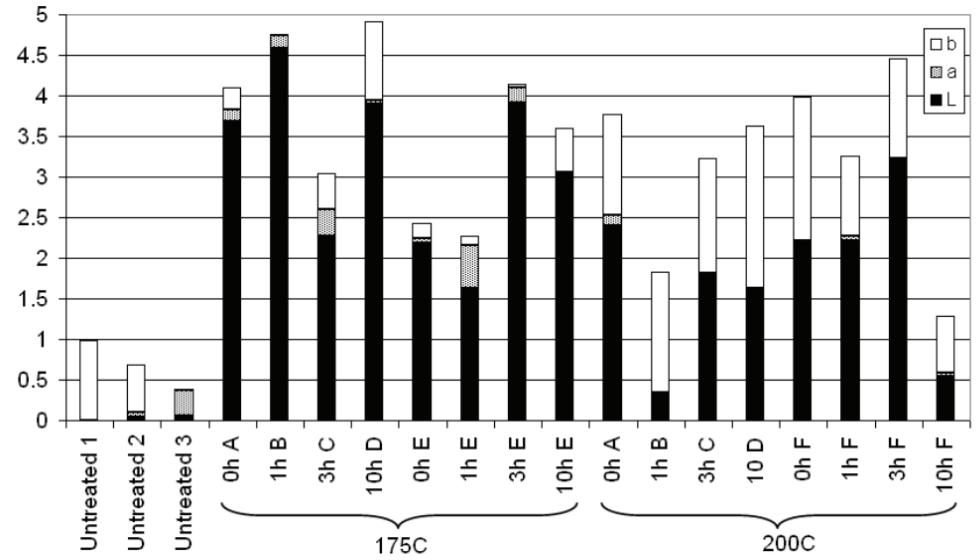

Figure 8: *Colour heterogeneity values of birch boards subjected to different heat-treatment processes. Each bar shows the heterogeneity of one board. The different colours in each bar represent the different colour coordinates' contribution to the heterogeneity. (Figure from paper I)*

There is difference between the three groups, 200°C, 175°C and untreated, as to which of the coordinates is causing the heterogeneity. In the untreated group, the heterogeneity is low, and there is almost no contribution from the L* coordinate, while in both treated groups this is the main contributor. This is understandable, since the darkening effect is connected to the heat treatment, and since the temperature at the surface of the board will be higher than at the core there should be heterogeneity due to lightness. The relation between the L* coordinate and treatment time seems to be fairly linear (see figure 9). As the L coordinate does not stagnate during these time spans, the inner parts of the board will not be able to catch up the surface, and the heterogeneity will persist.

Between the two temperature levels of the treated boards there is a difference in all of the colour coordinates. According to figure 8, the heterogeneity of the 175°C group has a greater influence of the yellow a* coordinate. This agrees with the previous reasoning, since, as figure 10 shows, yellowness increases with time, and then the yellow a coordinate stagnates at about 11, making it possible for the core to become as yellow as the surface.

In figure 11, the same phenomenon can be seen in the red b* coordinate. One difference, though, is that the b*coordinate (redness) increases from about 17 in untreated boards and stagnates between 22 and 23 for the 175°C group. As in the case with the yellowness, there will not be heterogeneity as long as the redness is stagnated and not changing with time. But in the 200°C group, redness decreases back towards its original value. This decrease of the b* coordinate is what is causing the contribution to heterogeneity.

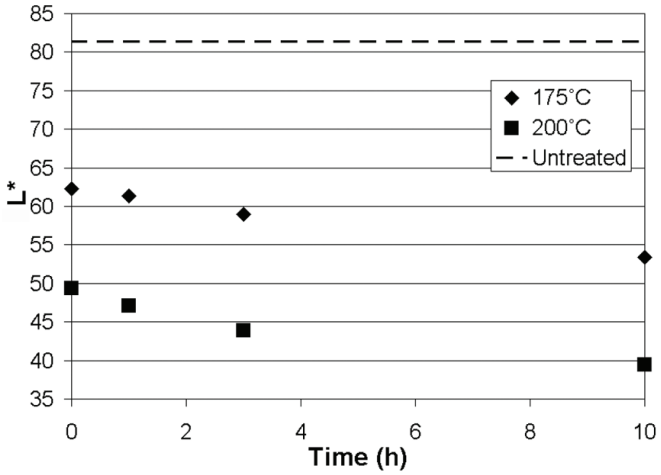

Figure 9: *Average values in lightness (L*) for all measurement done in the different heat-treatment processes. (Figure from paper I)*

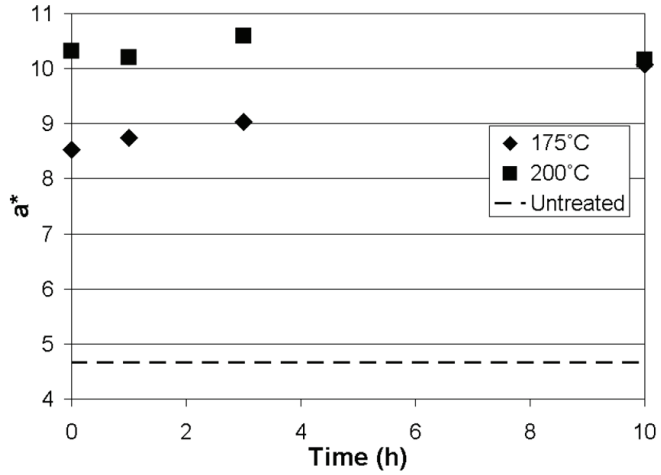

Figure 10: *Average values in yellowness (a*) for all measurement done in the different heat-treatment processes. (Figure from paper I)*

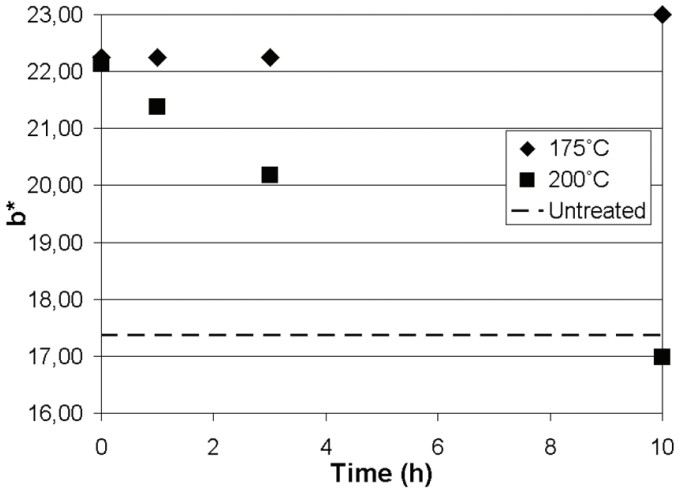

Figure 11: *Average values in redness (b*) for all measurement done in the different heat-treatment processes. (Figure from paper I)*

In paper I, an exothermic reaction was detected by measuring the internal temperature using thermocouples inserted into the wood. The reduction in the b* coordinate (redness) seems to coincide with activation of an exothermic reaction inside the wood during treatment. The effect of the exothermic reaction can also be seen in how lightness contributes to heterogeneity. Due to the exothermic reaction, the temperature at the core will actually be higher than the surface temperature for some time. This means that the core will at this point darken faster then the surface and thereby reduce the contribution from the L* coordinate to heterogeneity.

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Simulation of exothermic reaction

The internal temperature measured was used to study the energies generated inside the lumber. This study used Femlab, a program for numerically solving partial differential equations. In this case, the heat equation (1) is solved

$$C\rho\frac{\partial T}{\partial t} = \lambda\nabla^2 T + Q \qquad (1)$$

where T is temperature, C is the heat capacity, ρ is the density, λ is thermal conductivity and Q is the heat source. It was assumed that the thermal conductivity was isotropic and the surface temperature was approximated to be the same as the temperature measured by thermocouple closest to the surface (see figure 12b). Since the temperature in the middle of the board was measured, it was possible to minimize the error between measured and calculated value by changing the value of Q.

The calculated values of Q are shown in figure 12a. There is some instability in the calculated value of Q, but it gives an indication of the energy levels. In figure 12c, the difference between the calculated value and the measured one is plotted. This error is constantly kept below ± 0.03C, but it is probably the compensation for this error that is creating the instability. A number of factors might cause the error. For instance, there is always going to be some small fluctuation in measured temperatures due to the measuring error of the logger. Since the fluctuation at the surface is not synchronized with the one in the middle, it will cause instability.

By studying the energy generation throughout the treatment process, some interesting tendencies can be seen. The first part indicates a positive source term. This is probably because the condensation of steam on the wood causes the real heat transfers to be greater than the calculated one. It gradually reduces until it becomes negative due to drying, which is consuming energy. The value then starts going up again and stops at around zero, which can be seen as an indication of the wood reaching its equilibrium moisture content. When the temperature increase in the 2nd drying regime is started, it goes down once again because of the evaporation of water. But during this regime it becomes positive and reaches its maximum of about 1000W/m^3 in the transition to the treatment regime. After this it goes slowly down until the cooling regime is started, at which point it drops faster. Two possible explanations for this drop could be that negative relative pressure during cooling is adding convection to the heat flow or that the exothermal reaction is stopped by the cooling. Directly after the cooling regime, the source term increases once more. This could be due both to condensation of water or to the fact that the exothermic reaction has not stopped.

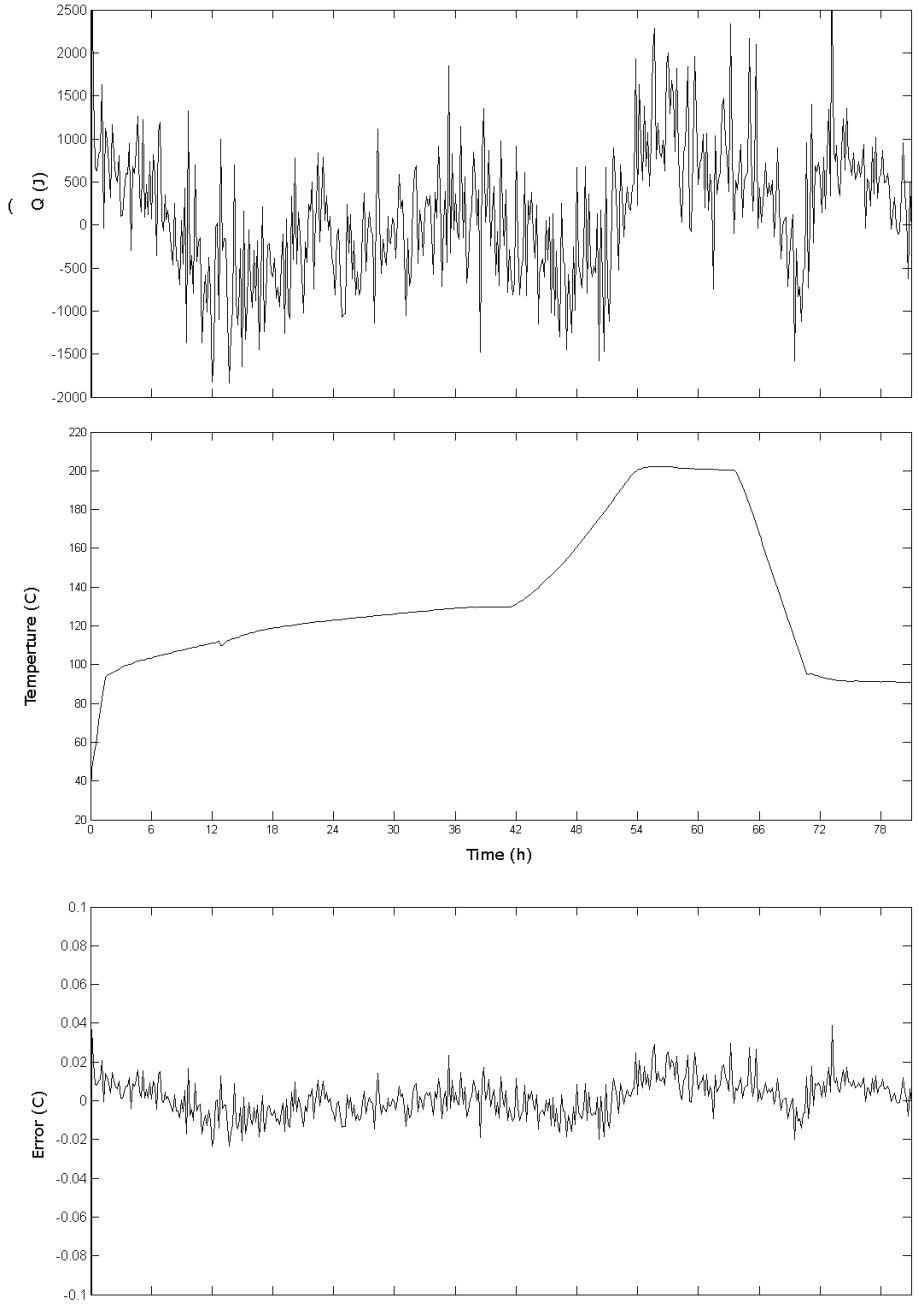

Figure 12a-c: *a) The calculated source term Q. b) The temperature measured closest to the surface. c) Shows the error; i.e., the difference between calculated and measured internal temperature after calculating Q.*

It should be remembered that this study has not been published and has not yet been validated; it needs more work in order to determine how accurate it is. But if this method of estimating the energy released during treatment is found valid, it might become useful in acquiring important information on the chemical changes that occur during treatment.

Internal Checking

Internal checking is often a problem when drying wood that is subject to cell collapse during the capillary regime of drying. According to Booker (1994), both internal checking and cell collapse are due to the same mechanism. It is negative relative pressure during the capillary regime of drying, which leads to tension in the cell walls. The later stress reversal then leads to tension in the internal parts of the lumber, resulting in propagation of the small checks initiated during the capillary regime. There are problems with internal checking in heat treatment as well, but since no free water is present, the checks should not be initiated by the same phenomenon.

Measuring internal checking

In papers II and III, internal checking was measured by cutting the boards into slices and then visually grading the cross sections as shown in figure 13. In the examinations, the presence of knots, pith, internal checking and checking associated with knots were documented. This method of evaluating the results is time consuming and subjective. If further studies are to be done, there is a need for a more objective method. This method might be based, for instance, on computerized image analysis to measure the amount of internal checking.

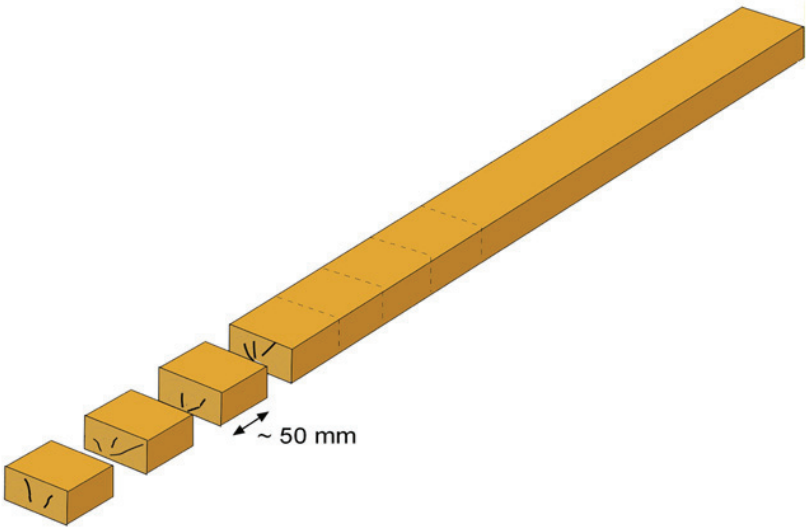

Figure 13: *Method used for detecting internal checking. The board is cut and one cross-section surface is visually graded. For each visually graded surface, the number of internal checks, knots, checks in knots and presence of pith are documented.*

Internal pressure

When the heat treatments in paper III were performed, pressure sensors were fitted to the boards to measure the internal pressure. The result was not included in paper III. These measurements were performed because it was thought that there might be a connection between positive pressure and internal checking. Each heat-treatment batch in paper III consisted of six 50- x 125-mm spruce boards. Three boards were fitted with a pressure sensor limited to 0.6 bars, and the other three with a sensor limited to 1.6 bars. A pipe with a maximum length of 1 meter was connected at one end to the pressure sensor and to a connector screwed into a hole drilled into the board at the other.

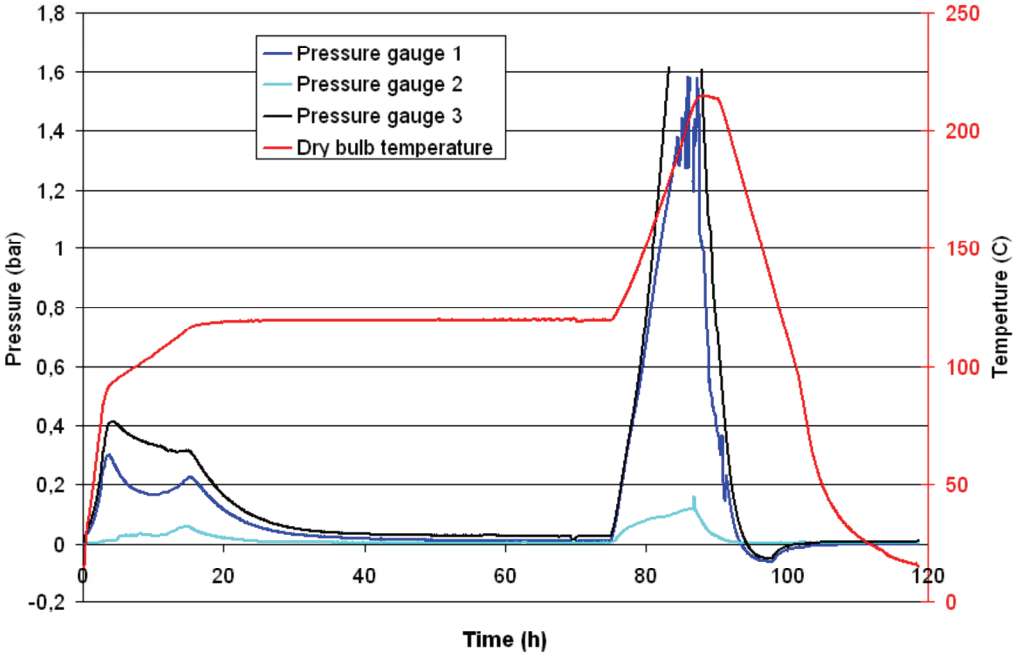

Figure 14: *Treatment temperature plotted together with pressure. Maximum pressure is reached in the second drying regime, which suggests that the pressure is most likely due to vapour and thermal expansion. The low value of the pressure gauge may be an affect of leakage.*

The result from the pressure measurement during the heat treatment of predried boards with a 2nd drying-regime length of 12 hours is shown in figure 14. In the beginning the pressure goes up due to the generation of steam inside the board. (Perre 1996) reported a maximum pressure of 2.7 atmospheres during high temperature drying at 160°C. The maximum pressure during the heat treatment was reached during the 2nd drying regime. In some of the boards the pressure was higher than the sensor could measure; i.e., more than 1.6 bars above atmospheric pressure. It seems that most of the pressure is generated through thermal expansion, since the pressure drops as soon as the heating stops. In these measurements it was not possible to make any connection between pressure and internal checking.

Loss of dry mass

Heat treatment of wood results in a loss of dry mass, which causes shrinkage of the wood. According to Chang and Keith (1978) there is a strong correlation between loss of dry mass and volumetric shrinkage. They also showed that the anisotropy of the shrinkage is similar to the shrinkage due to changes in MC.

Since Chang and Keith (1978) only studied hard woods, a small test was performed to study the anisotropic shrinkage of spruce. Seven samples with the dimensions of approximately 35 x 35 x 400 mm were used. All samples were first oven-dried for 24 hours, and then each sample was measured at three different positions approximately 100 mm apart. Then heat treatment was performed at 212°C for 3 hours, after which the samples were measured once more.

The results showed an average shrinkage of 1.78% in the tangential direction and 0.68% in the radial. This gives anisotropy of 2.6, which is higher than anisotropy due to moisture change. The tangential shrinkage due to the heat treatment is comparable with a moisture loss of 7%. This means there will either be deformation or stress generated through thermal degradation. The results in paper II strengthen this hypothesis further by a statistically significant correlation between dry-mass loss and boards developing internal checking.

Drying

It is a well-known phenomenon in ordinary wood drying that there will be tensional stress in the internal parts of boards. This stress is caused by MC gradient, anisotropy in shrinkage and mechanosorptive creep. The effects of mechanosorptive creep are often referred to as case hardening or set. Both paper II and paper III indicate that drying has a major influence on the development of internal checking.

In paper III, six batches of spruce boards were heat treated with three different processes (see figure 14). Three of the batches were predried from 18% MC to 6% MC in a climate with 90°C dry-bulb temperature and 70°C wet-bulb temperature, and the other three batches started the process with 18% MC.

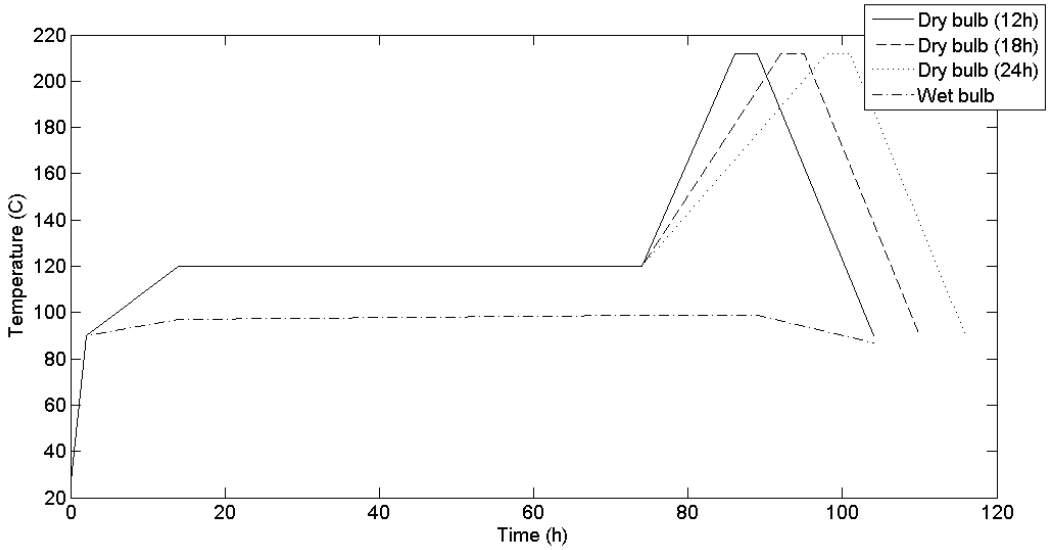

Figure 15: *The three different process schedules used in paper III. For each process with different time span, two batches were treated, one with 18% initial MC and one with 6% initial MC. A seventh process was used as reference. Both initial MC levels were included, but the treatment was stopped at the end of the 1st drying regime.*

The results in paper III showed that both predrying and longer time in 2nd drying regime result in less internal checking. It was concluded that reduced drying stress is the most likely explanation as to why the predried batches developed less internal checking. As reference, two additional groups were exposed to the same process up to the starting point of the 2nd drying regime. One of the groups had 18% initial MC and the other was from the predried boards with 6% initial MC. Slicing tests were performed after approximately four months of storage. The average split gap directly after cutting was about 0.4 mm. In paper III, some simple calculations were made to estimate the level of stress in the wood. Under the assumptions made in paper II, the measured split gap corresponds to an approximate stress of 0.4 MPa. This stress is not sufficient to generate internal checking

on its own, but could very well explain the difference between the two initial moisture-content groups.

In paper III there is a discussion of whether it might also be an effect of microcracks generated in the boards through fast drying in high temperatures. Therefore, a small test was performed on the tangential strength of the reference boards. If there are microcracks present in a board, it should have a lower strength. The test samples were cut as shown in figure 16.

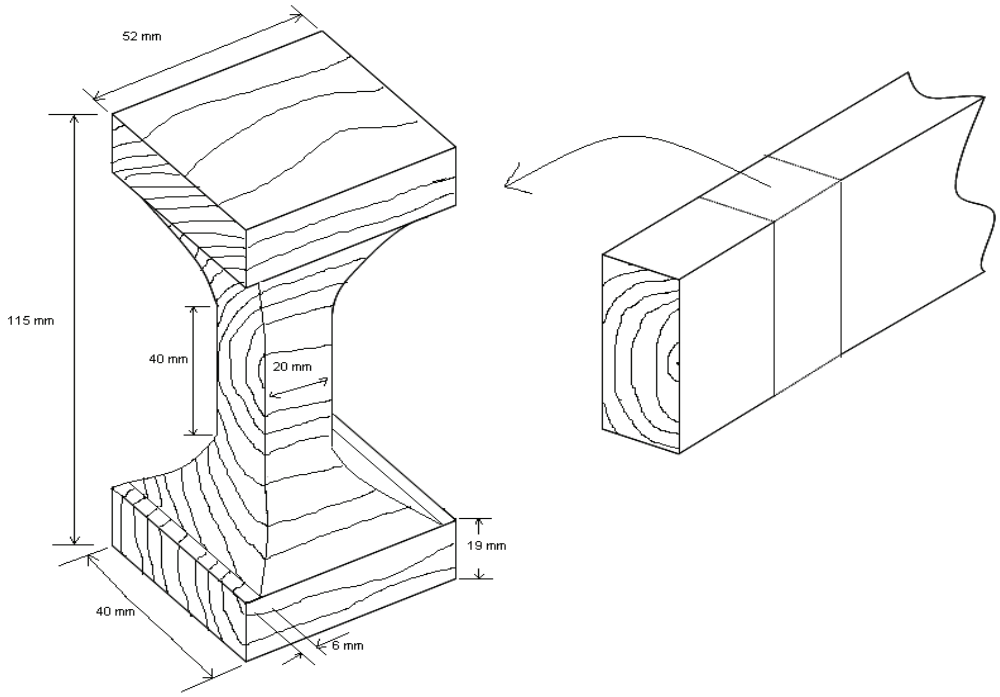

Figure 16: *Samples for tensional strength test in tangential direction. This design was used both for comparison of the two different reference groups in paper III and in a small study of the effect of high temperatures on tensional strength.*

The results, shown in figure 17, do not indicate any reduction in the average tangential strength. However, the standard deviation of the predried group is only half that of the group with 18% initial MC, and the difference is statistically significant. No explanation of the difference in standard deviation has been found. It might simply be the difference in the material, since it is a quite small test set. But the results do indicate that there is no decrease in tangential strength in the group with 18% initial MC.

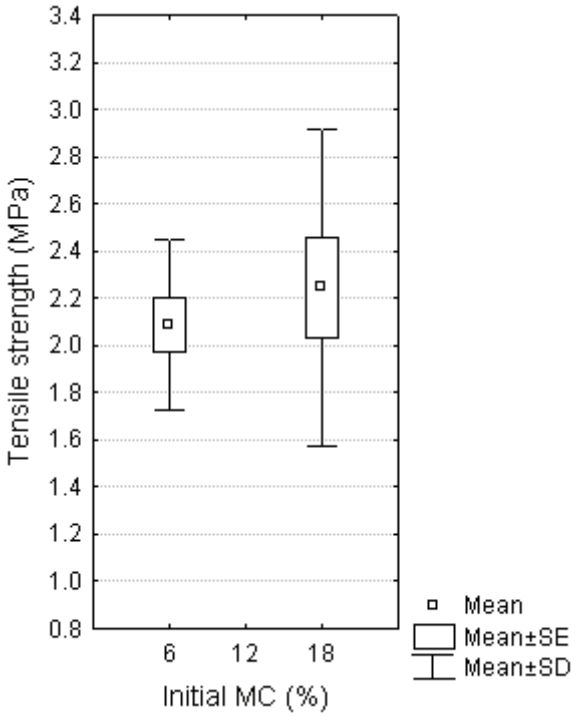

Figure 17: *Tensile strength in the tangential direction for the two reference groups in paper III.*

There is also a discussion in paper III of the possibility that there might be property changes at high temperatures that can affect the development of internal checking, for example if there is a sudden drop in strength or a drastic change in the MOE. Therefore, the tangential strength of a set of matched spruce samples was tested at 150°C, 180°C and 210°C. The test samples were produced in the same way as the previous test. The tests were performed inside an oven. In order to know when the internal temperature of the wood had reached testing temperature without damaging the test sample, a reference piece was placed right next to the sample. This reference piece was fitted with a thermocouple. The internal temperature of this reference piece was used to determine when the testing should be started.

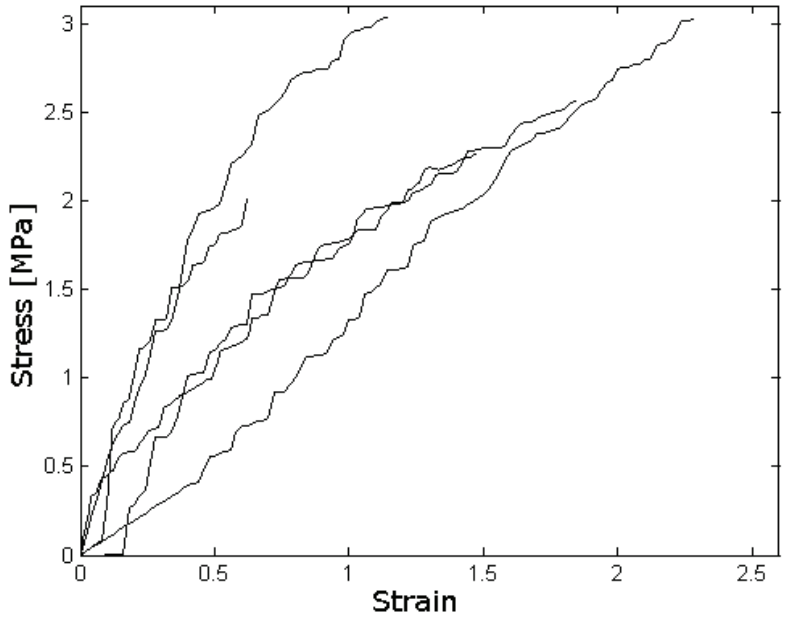

Figure 18: *Testing of tensile strength in the tangential direction in spruce. Testing was performed in an oven heated to 150°C.*

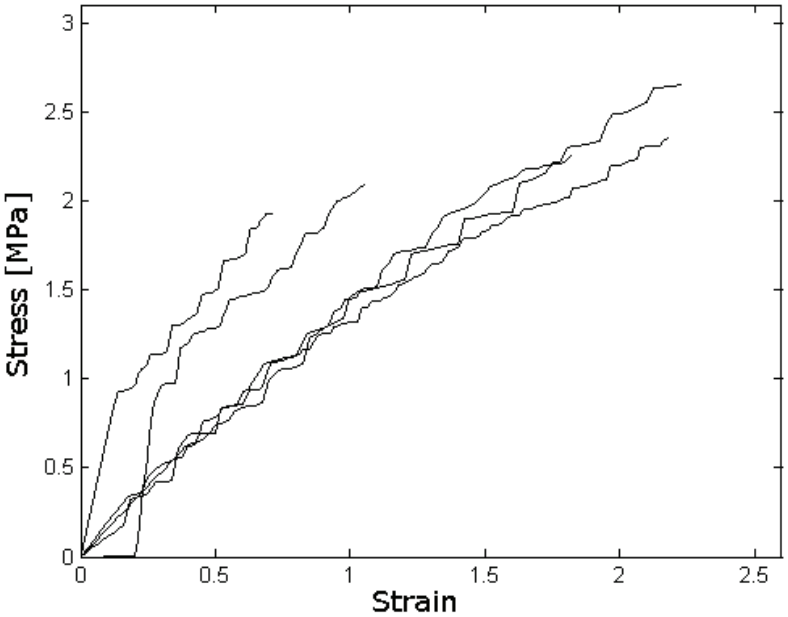

Figure 19: *Testing of tensile strength in the tangential direction in spruce. Testing was performed in an oven heated to 180°C.*

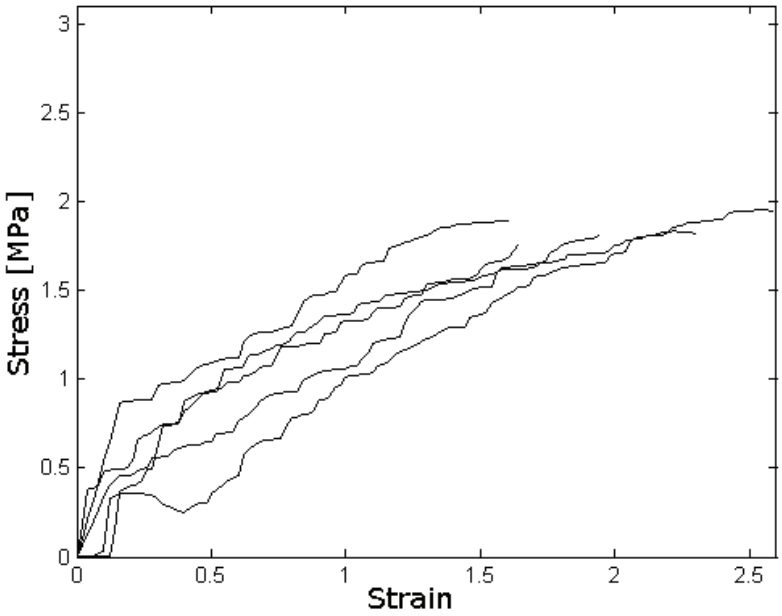

Figure 20: *Testing of tensile strength in the tangential direction in spruce. Testing was performed in an oven heated to 210°C.*

From results shown in figures 18–20 it is possible to see a tendency of the wood to become more plasticized at higher temperatures. One explanation for the increased plasticized behaviour is that the wood is approaching its glass transition temperature. The glass transition temperature in wood is highly dependent on MC. According to (Salmén 1982), the glass transition temperature for isolated lignin and hemicellulose when dry is between 180°C and 220°C. When wet, the transition temperature for lignin is lowered to 80–90°C, and for hemicellulose to room temperature. This indicates that the internal checking occurs before maximum temperature is reached, since during the heat-treatment regime, relaxation of stresses is more likely to occur. This is also supported by the slicing test performed on heat-treated boards in paper II, which did not indicate any stress whatsoever.

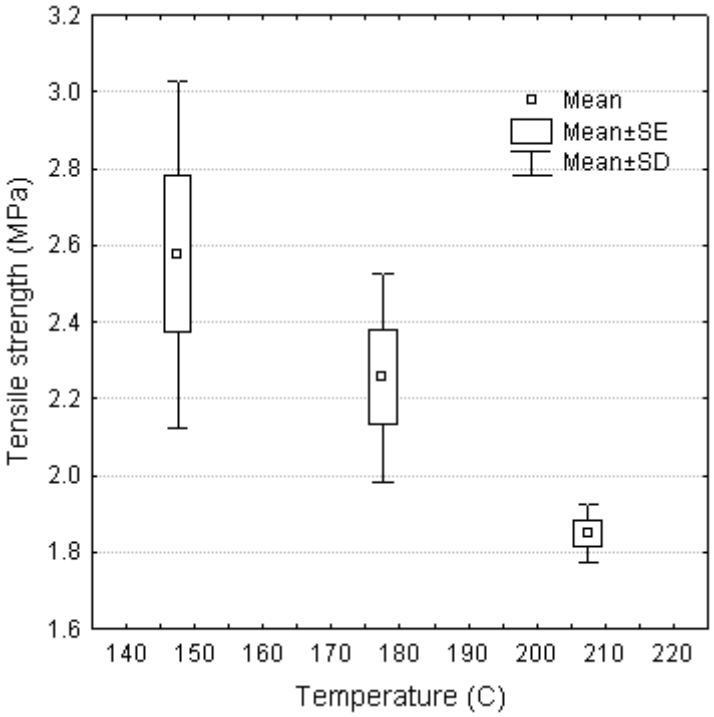

Figure 21: *Tensile strength in the tangential direction of spruce at 150°C, 180°C and 210°C.*

In figure 21, the tensile strength is plotted. The results show a 30% reduction in tensional strength from 150°C to 210°C that is statistically significant ($t = 3.264$ and $p = 0.0310$). But there is also a significant reduction of the standard deviation ($F = 9.919$ and $p = 0.0132$). This is probably due to a more even stress distribution when the wood is above the glass transition temperature. This also lends further support to the hypothesis of internal checking not occurring during the high temperature regime, because if internal checking were to occur above 200°C, the checking should be distributed more evenly between the boards, since the difference between them is reduced.

The creep behaviour is probably the explanation as to why results in both paper II and paper III demonstrate that a slower temperature increase in the 2nd drying regime results in less internal checking. Since the creep behaviour increases at high temperature, it might be possible to avoid internal checking by having very gentle drying down to the fibre saturation point and then quickly raising the temperature during 2nd drying regime. That is, if the temperature is increased fast enough, the surfaces of the board become softer and do not generate stress inside. Passard and Perré (2004) suggest that high-temperature drying should be performed at temperatures above the glass transition temperature to avoid defects.

Concluding remarks

Colour measurements are not suitable for quality control in industrial applications. However, there is a use for colour measurement for scientific studies. By measuring colour it is possible to get more information on what is happening inside the wood during the treatment process.

Internal checking is to some extent caused by thermal degradation that generates stress through shrinkage due to the loss of dry mass. But the main reason for internal checking of heat-treated wood is drying. This means it is possible to avoid internal checking by drying the wood slowly so that relaxation of drying stress through creep is possible.

Future work

Even though this method is used industrially, a lot of research is still required to gain an understanding of all of the different properties of this new material. Examples of areas in need of further research are:

- Moisture movement in heat-treated wood. The influence of treatment parameters and wood species in both capillary and diffusion flow.
- Strength properties of heat-treated wood. Detailed studies to determine which strength properties are affected at which temperature.
- Further research on the exothermic reactions that occur during the treatment process.
- Colour stability of heat-treated wood.

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Paper I

The potential of colour measurement for strength prediction of thermally treated wood

D. Johansson, T. Morén

Abstract

This paper investigates the effects of thermal treatment of birch with respect to colour and strength. Birch wood was treated at 175°C and 200°C for 0 h, 1 h, 3 h and 10 h. In bending-strength experiments, treatment was also performed at 185°C for 2 h. Both static bending strength and impact bending strength were investigated using multivariate statistics (PLS) for correlation to process parameters, density, EMC, position in board, modulus of elasticity (only in static bending), colour and dimensions of samples. In static bending, two PLS models were designed, one based on process parameters and the other based on colour and EMC. From these models it was concluded that colour is not a useful parameter for prediction of strength. In impacted bending, the correlation was too small to give useful results. One test of static bending strength with matched samples was performed, and it showed a strength reduction of 43% when treatment was conducted at 200°C for 3 h. Measurement of colour homogeneity of the treated boards showed that the colour is not homogeneous.

Introduction

Normally, wood is thermally treated in order to improve its durability, but sometimes thermal treatment is used to change the aesthetic properties of wood. In Finland, this is, according to Syrjänen (2001), the main reason for using thermally treated birch. The dark colour of the treated birch makes it possible to replace more expensive and exclusive wood materials with cheaper wood. Birch can be given a nice surface finish and is well suited for carpentry. This has opened a new market. A vision of the future is suppliers that will be able to supply wood within a specified range of the colour spectrum. Before this vision can be fulfilled, more research has to be done in the area of how process parameters affect these aesthetic properties.

One other important aspect of thermally treated wood is strength reduction. According to Viitaneimi (1997), effects of thermal treatment of wood at temperatures between 185˚C and 250˚C are a reduction of Equilibrium moisture content (EMC) by 43%–60%, shrinkage and swelling reduction of 30%–80% and a reduction of bending strength by 5%–25%. The wood species used were spruce, pine and birch. According to the Finnish Thermowood association (2003), ungraded spruce boards treated at 230˚C for 5 hours exhibit a 40% reduction in bending strength. Tests on clear wood of spruce showed a substantially smaller change. Results in fact indicated a small increase in bending strength for clear wood with treatment temperatures below 200˚C. Similar results are presented by Kobojima et al. (2000) with treatments performed in air and nitrogen atmospheres. One difference is that Kobojima et al. (2000) used a constant treatment temperature of 160˚C

and noted an increase in bending strength when the duration of treatment was less than 4 hours. According to Bengtsson et al. (2002), a reduction of approximately 50% in bending strength was found in tests of thermally treated spruce and pine.

In Bekhta (2003), the colour of thermally treated wood was examined to determine the effects of temperature, time and relative humidity during conditioning and whether there is a correlation between strength and colour. These experiments were performed without any shielding gas. The correlation between total colour change and the mechanical properties of the wood was found to be good. Patzelt et al. (2003) conducted thermal treatment inside a pressure vessel. They found good correlation between mass loss and the colour parameters lightness (L) and hue (h).

The aim of this work was to investigate the influence of varying process parameters on the mechanical properties of the board and on colour distribution through the board. The correlation between colour and strength was studied in order to examine the possibility of using colour measurement as a parameter for quality control of strength.

Materials and methods

Plant and process

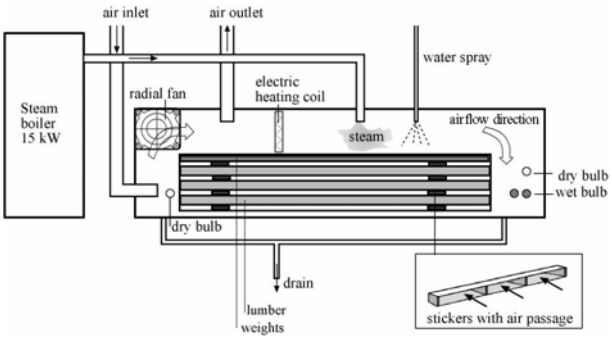

Figure 1: *Kiln for thermal treatment of wood by using superheated steam as shielding gas.*

A small-scale kiln designed for thermal treatment up to 230˚C was used in these experiments, as shown in Figure 1. The air flows along the boards and through specially designed metal stickers. In this work, the Finnish method for thermal treatment was used. Superheated steam at 130˚C and atmospheric pressure is used as a shielding gas, which prevents the wood from catching fire. Figure 2 shows the process divided into five regimes.

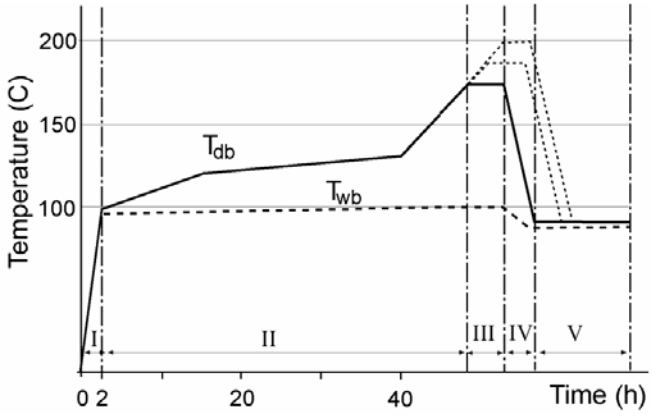

Figure 2: Treatement process divided into five regimes. I) Heateing with superheated steam. II) Drying regime – High temperature drying: the wood reaces almost 0% MC. III) Treatment regime –Wet bulb temperature kept at 99C by continuously injecting steam as shielding gas. IV) Cooling regime – Cooling rate is regulated with the electric heating coil, steam and water spray. V) Conditioning regime – The objective of this regime is cooling and remoistening.

Experimental design

Table 1: *The experimental design*

No.	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Temperature (ºC)	175	175	175	175	185	200	200	200	200
Treatment time (h)	0	1	3	10	2	0	1	3	10
Total time above 155˚C (h)	4.33	5.33	7.33	14.33	9	10	11	13	20

Nine batches of birch were thermally treated. All batches are presented with their process parameters in Table 1. *Zero hours* means that the batch was heated to the designated treatment temperature and then immediately cooled down. Except during the treatment regime, all batches had identical process schedules as shown in Figure 2. During the heating and cooling regimes, the wood undergoes some additional treatment due to the high temperatures therefore the parameter total treatment time is implemented. The total time above 155˚C will be referred to as *total treatment time*.

Specimens

Static bending and impact bending

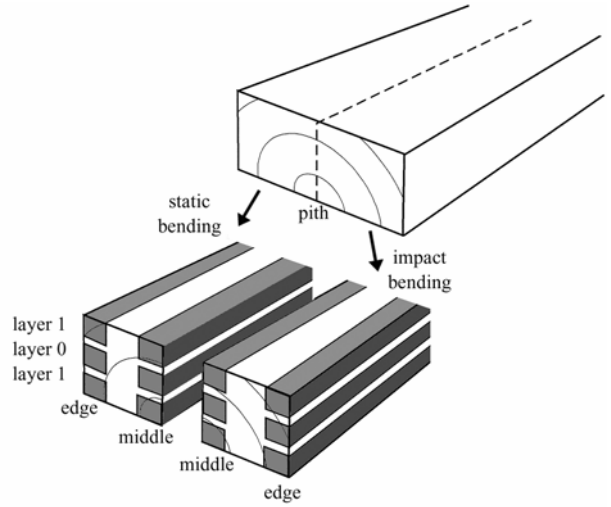

Figure 3: *Method for selecting test samples for static- end impact bending tests.*

Treatment was performed on 50 x 100 mm^2 birch boards of various lengths. Test pieces with clear wood were sawn from each board as noted in Figure 3. A total of 720 samples were used for the mechanical testing, 360 with the dimensions 12.5 x 12.5 x 200 mm^3 used for static three-point bending tests and 360 with the dimensions 12.5 x 12.5 x 120 mm^3 used for Charpy impact bending tests. Static three-point bending was performed on a Hounsfield H25KS with a span of 150 mm and a loading speed of 2.5 mm/min. The Charpy test was performed with span of 70 mm, and the energy at impact was 14.7 J. After mechanical testing, moisture content was adjusted by oven drying at 103˚C for 24 hours. After oven drying, density was measured with an X-ray CT scanner.

Matched pairs

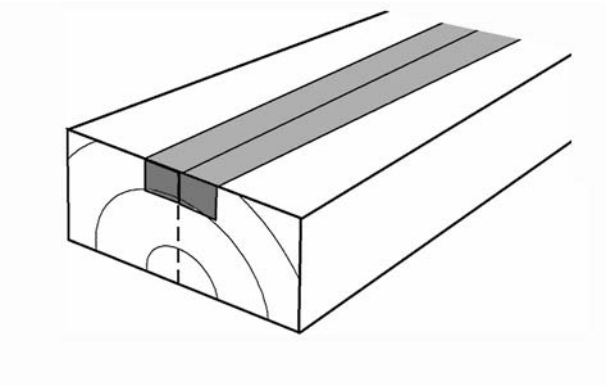

Figure 4: *Positions from which the samples for matched-pair testing were taken.*

This test set was designed to compare bending strength of untreated and treated birch. Bending tests where performed on 20 matched pairs cut directly adjacent to each other from the same board, as shown in Figure 4. A static three-point bending method was used. Samples were dimensioned to 140 x 140 x 250 mm. A span of 150 mm and a loading speed of 2.5 mm/min were used. One of each pair was used as an untreated reference, and the other one was treated at 200˚C for 3 hours. The specimen picked for treatment was not cut until after the treatment was performed. This was done to reduce the risk of samples becoming deformed as a result of the heat treatment.

Colour measurements

Colour homogeneity

In the study focusing on the colour homogeneity, 16 thermally treated boards were used, together with three untreated boards used as references. After treatment, the boards were planed to produce parallel surfaces and then sawn into slices. All surfaces were sanded to minimize the risk of variation of colour values caused by differences in surface structure. Measurements of the colour coordinates were performed with a Minolta Chroma Meter CR310 with a 50-mm measuring head. The CIE L*a*b* colour system was used according to the CIE standard (Hunt 1995). The light source used was Standard Illuminant D65 (average daylight including ultraviolet wavelength region). Measurements were made on both sides on each slice. The number of measurements on each surface varied due to differences in the lengths of the boards and the number of knots, since measurements were only performed on clear wood.

Colour for mechanical testing

Colour measurement of test samples used in mechanical testing was performed with a high-sensitivity colour line-scan camera, Dalsa CL-T7-2048W. The line camera consists

of three light sensitive sensors, one each for red, green and blue. When a sensor is registering an image, it sweeps over the object pixel by pixel. After a line is registered, the measurement object is moved, and a new sweep will be performed. From each of the sensors, a number between 0 and 255 will be received for each pixel. All three sensors do individual sweeps along an individual line. The result will be three matrices, one for each colour, which together make up the colour image. The data collected for each specimen are average values and the standard deviation for each colour within the measured area. All the measurements were made with the same illumination.

Colour homogeneity

To analyze the measured colour, the statistical software Jmp was used. The L*, a* and b* coordinates were measured on all boards. L* is the lightness, positive a* is the red parameter, positive b* is the yellow parameter. The colour is defined as homogenous if it can be described with a linear function between the surfaces. The colour differences at the surfaces of the boards are assumed to be caused by differences in treatment conditions inside the kiln. Although there are colour differences caused by sapwood, heartwood, annual rings and other conditions, these differences are assumed to be smaller than the differences caused by the treatment.

A second-degree polynomial was fitted to the L*, a* and b* data. Since there are three colour coordinates, this will give three equations describing the change through the board. The equations were denoted L(x), a(x) and b(x). Then three new functions are calculated and denoted L_{new}, a_{new} and b_{new}. These new functions have the same curvature as the original ones, but are of a standard thickness of 44 mm and intersect the x-axis at 0 and 44. This thickness was chosen so it would correspond to a thickness of 50 mm with 3 mm planed on each side. From these new functions it is possible to calculate the colour difference caused by the curvatures. The colour difference between x = 0 and x = 22 are calculated as follows:

$$\Delta E_{new} = \sqrt{\Delta L_{new}^{\ 2} + \Delta a_{new}^{\ 2} + \Delta b_{new}^{\ 2}}$$

Data analysis

In both the static bending tests and the impact bending tests, a prediction model was designed with the multivariate statistics software Simca. Analysis was made with partial least squares (PLS) regression. PLS is a regression method that handles multiple Y's and multiple X's. The result of the PLS analysis is a number of coefficients (B). The relation can be expressed in matrix form as

$$Y_{nm} = X_{nk} B_{km} + F_{nm}$$

Here, F_{nm} is the residual. Often when working with PLS models, it is of interest to look at the coefficients when they are scaled and centred. This is because they then describe how the variation in a parameter relates to the prediction. More thorough information about PLS analysis can be found in Eriksson (2001).

From both the static bending test and the impact bending test, 60 samples were randomly selected as a prediction set. The prediction set is used to independently validate the model. Thickness and width of the sample, moisture content (MC), colour values from the line camera (red, green and blue), density from CT scanner, treatment time, total treatment time (>155˚C), treatment temperature, position in board (layer, middle/edge), MOE (only in static bending) were used as predictors (X).

Result

Table 2: *The coefficients of the PLS two models*

Variables	Model based on process parameters	Model based on colour
Constant	126	351E-3
Width	X	X
Thickness	5.60	8.86
MOE	9.37E-3	813E-6
MC	X	628
Treatment temperature	-1.07	X
Total treatment time	-1.30	X
Red	X	-180
Green	X	X
Blue	X	X
Density	X	X
Layer	X	X
Middle/Edge	X	X

PLS modelling resulted in two equivalent models for static bending, one with colour data and one without. The PLS model made without colour data had a goodness of fit (R2Y) of 0.67 and goodness of prediction (Q2) of 0.64. The model with colour parameters had slightly lower, but still comparable, R2Y and Q2 values of 0.63 and 0.60. Colour data can work together with the MC as a substitution for the parameters of the treatment process. The coefficients of the two models are presented in table 2. For impact bending, it was not possible to design a sufficient model with the data used.

Table 3: *percentage of the samples with shear fracture to the end surface*

Treatment temperature	Treatment time	%
175	0	0
175	1	5.5
175	3	4.4
175	10	5.5
185	2	0
200	0	0
200	1	13.9
200	3	27.8
200	10	44.4

When the impact bending tests were performed, some of the samples had a shearing breakage all the way to the end surface. The tested objects were divided into two groups, depending on failure type. Table 3 shows the distribution of shear fracture to end surface for different treatment times and temperatures. It is clear that an increase in temperature or time reduces the shear strength along the fibres.

A 43% reduction in bending strength was found for the matched pairs at a significance level of 0.01 The results for the MOE showed no statistically significant changes, but a tendency toward a decrease of the MOE.

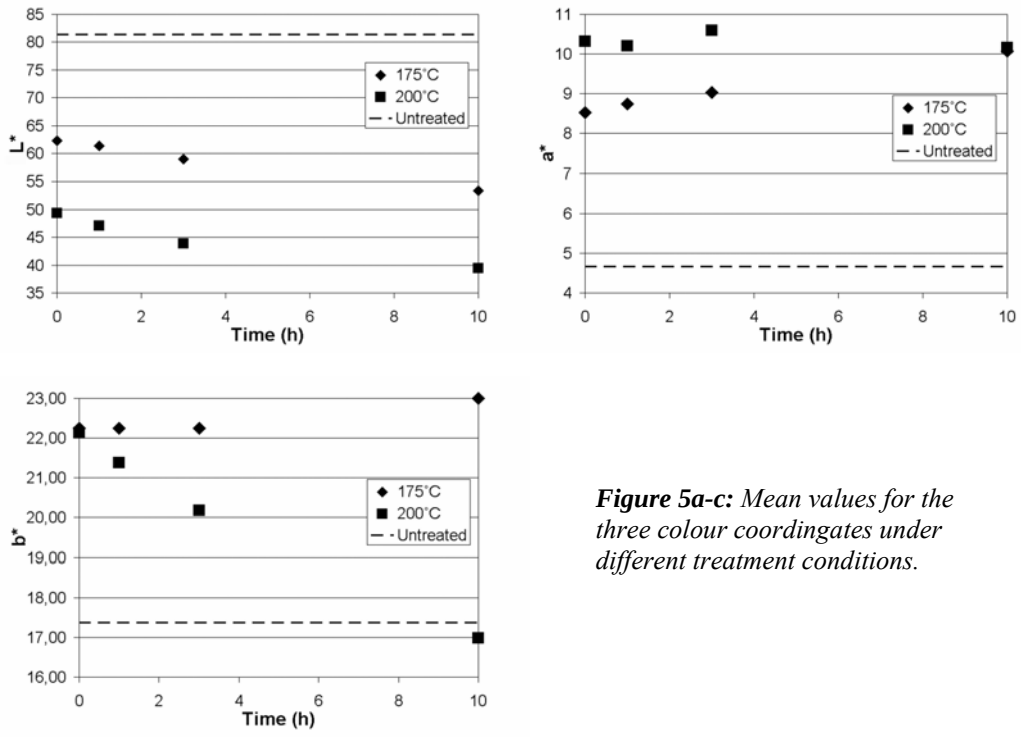

Figure 5a-c: *Mean values for the three colour coordingates under different treatment conditions.*

The average values of the colour data at different treatment times and temperatures can be seen in Figure 5 (a–c). A linear reduction of lightness was noted for both 175˚C and 200˚C. When boards were treated at 175˚C, the red a* coordinate was higher than in untreated boards and increased with treatment time; i.e., it became more reddish. Boards treated at 200˚C were at a fairly consistent level. The yellow b* coordinate was found to be higher than in untreated boards, but was fairly constant for the different treatment times at 175˚C. When treated at 200˚C, the b* decreased over time, and at ten hours of treatment was back to the level of the untreated boards.

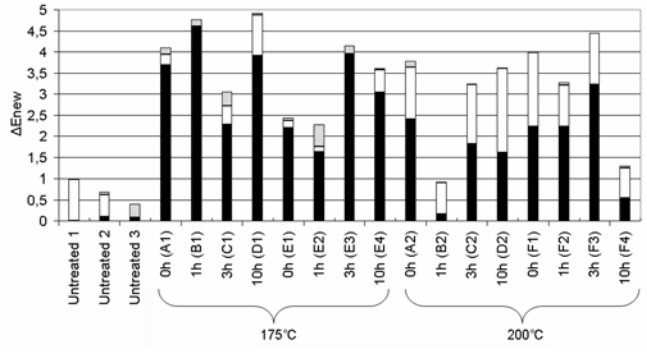

Figure 6: *The values of $\Delta E_{new,}$ for each board divided onto contribution of $\Delta L_{new,}$ Δa_{new} and $\Delta b_{new.}$*

The results show that colour is not homogeneously distributed through the board. The average value of ΔE_{new} for the treated boards was 3.4 and for the untreated boards 0.68.

There was a reduction of ΔE_{new} for boards treated at 200˚C. An examination of the extent to which the colour difference is made up by each colour coordinate (Figure 6) reveals that difference in lightness contributed almost nothing to ΔE_{new} in the untreated boards, while it was the main contributor in the treated boards. The tendency noted was that the contributions from ΔL_{new} and Δa_{new} are reduced when the treatment is performed at 200˚C. At the same time, the contribution of Δb_{new} to ΔE_{new} increases significantly when treatment is performed at 200˚C.

Discussion

Static bending

From the static bending models, it is clear that variation in MOE is the parameter that best describes the variation in bending strength. According to the prediction model based on the process parameters, a decrease of 1˚C in temperature can be balanced by an increase of approximately 50 minutes in total treatment time. This is certainly not entirely true, because there is a correlation between total treatment time and treatment temperature. That is, when treatment is performed at higher temperatures, the total treatment time will also increase. In the model based on the colour data, it is only the red colour that shows signs of describing strength. The model has to include the MC as a predicting parameter. But since all samples have been kept in the same climate the MC at the time of testing will actually be a measurement of the EMC.

According to the results presented in this work, colour lacks potential as a predictor of strength. This is mainly because predictions made by the process parameters give better results. There is a correlation between colour and process parameters and also between strength and process parameters. But since both strength and colour responses are dependent on additional parameters such as sapwood, heartwood, annual rings, juvenile wood, etc., a board can acquire a darker colour without additional reduction in strength.

Impact bending

The only valuable information from the impact bending test is the fracture due to shearing. This probably occurs because the lignin is degraded. This supports the results in the Thermowood handbook. The results show that shearing strength is reduced with longer process times and that the effect will be accelerated by an increase in temperature. It is important to point out that it is not a shear-strength test that has been performed, only an impact bending test. But, the results indicate significant reduction in shear strength in thermally treated birch.

Colour homogeneity

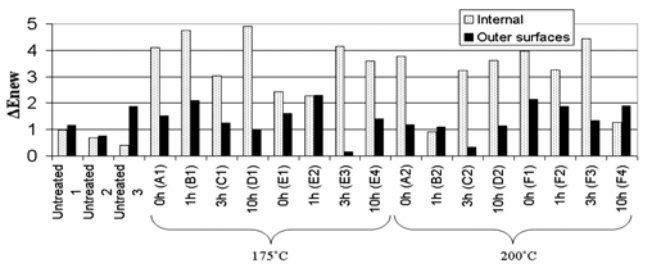

Figure 7: *The internal colour difference ΔE_{new} and the colour difference between the outer surfaces of the board.*

Figure 7 compares the value of ΔE_{new} with the average colour difference between the two outer surfaces for each board. The colour difference between the surfaces is smaller than ΔE_{new} in 13 of 16 treated boards, while it is the opposite for all untreated boards. This lends strength to the assumption that colour differences mainly depend on the treatment process. The reason for the lower value of ΔE_{new} at 200˚C was the reduction of the contribution of ΔL_{new}. Δa_{new} contributed somewhat to ΔE_{new} for both the untreated boards and those treated at 175˚C, but almost not at all for boards treated at the higher temperature. This was interpreted to be an effect of the value of a* stagnating at a value of about 10.5. This gives the interior time to catch up with the outer parts of the board. An effect of Δb_{new} was noted in all groups, but the contribution increases at 200˚C as shown in Figure 8. In the test at 200˚C for 3 hours, two holes were drilled, and thermocouples were inserted into the board. One of the thermocouples was inserted near the surface of the board and the other in the middle of it. In Figure 8, the temperature of the outer thermocouple is shown together with the temperature difference between the two.

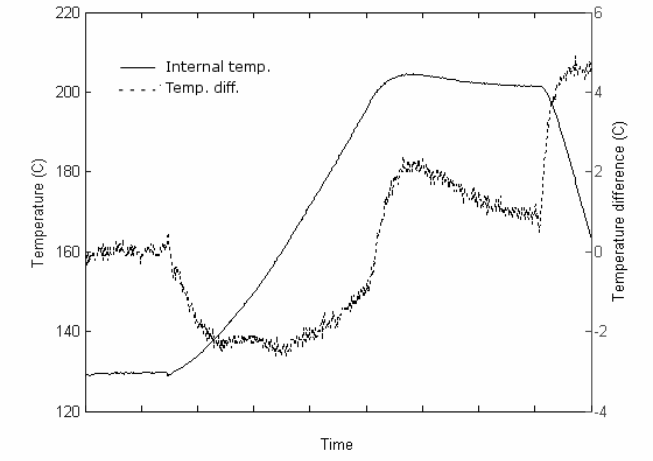

Figure 8: *Internal temperature and its difference to the temperature at the surface af the board (internal temperature – surface temperature).*

From Figure 8 it can be seen that in the initial stage of the treatment regime, results were as could be expected: the internal part of the board is not as warm as the outer parts. But when the temperature reaches temperatures above 160˚C, the interior of the board begins to catch up to the outer parts. At about 180˚C, the rate of reduction of the temperature difference is further increased. When the temperature reaches the targeted temperature of 200°C, and the heating stops, the interior becomes warmer than the exterior. This was seen as an indication of an exothermal reaction being present. A hypothesis is that the reduction of ΔL_{new} is due to heat generated from the exothermal reaction, and the changes in Δb_{new} are caused by chemical aspects of the reaction. Further investigation should be done on this subject.

Conclusion

This study shows a 43% reduction in bending strength of clear wood in comparisons between birch thermally treated at 200˚C for 3 hours and untreated birch. No indications have been found of a strength increase of clear wood when thermally treated at temperatures between 175˚C and 200˚C. From the multivariate prediction model of bending strength, it is concluded that temperature is the most crucial process parameter and that colour is not suitable as a predictor of strength. If quality control of the treatment processes is to be done with respect to strength, the best way of doing so is to have good control over the climate inside the kiln together with measurement of the MOE. An apparent reduction of shear strength is indicated by a study of the nature of fractures occurring during the impact bending test.

Colour distribution through the thermally treated boards was found not to be homogeneous. Lightness is the main contributor to the uneven colour distributions; When treatment is performed at 200˚C, the contribution from lightness decreases, and the contribution of the yellow b* coordinate starts to have a significant effect on colour distribution.

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Paper II

DRYING AND HEAT TREATMENT OF WOOD: INFLUENCES ON INTERNAL CHECKING.

Dennis Johansson[1]
1. Luleå University of Technology, SE-931 78 Skellefteå, Sweden
email: Dennis@ltu.se

Keywords: Wood, Heat treatment, Internal checking

ABSTRACT

Heat treatment and drying of wood is a way to improve durability and stability of wood. The treatment method used in this research is based on steam treatment. A special kiln is used to perform the treatment. The kiln is basically a drying kiln with capacity up to temperatures of 230°C. During the heat treatment the wood has a moisture content of about zero per cent. Therefore the regime up to the treatment can be seen as a high temperature drying in an atmosphere of superheated steam. After this drying regime, when the temperature has reached the heat treatment temperature, it is kept constant for 2-4 hours. Internal checking is a problem when thick boards (>50mm) are heat treated. The objective of this work is to investigate what it is that is causing these cracks. The main question to answer is whether stress from the drying regime is affecting internal checking.

INTRODUCTION

Wood is a biological material that can be degraded by insects, fungi and bacteria attack. But if the wood is dried to moisture content less than approximately 22% there is not enough water for fungi to grow. Another reason for drying wood besides to avoid the biological degradation is to improve mechanical properties as well as to adopt the wood to end user products. It is also important to reach stress-free conditions with a homogeneous moisture distribution. In many situations were wood is used; it will be subjected to water in different forms with an increased moisture content as a result. To prevent rewetting of the wood it is possible to use one of numerous protective methods e.g. impregnation and coating. These protective methods are usually based on adding different chemicals to the wood that will protect it, however with thermal treatment this is not the case.

Heat treatment changes the properties of the wood resulting in a more dimensional stable and less hygroscopic material. The basic idea of the method is heating the wood to temperatures above approximately 150°C where chemical reactions are accelerated. The well known fact that charcoal is very decay resistant has been used for centuries e.g. by burning ends of wooden poles. Stamm and Hansen (1937) studied the possibilities to making wood more dimensional stable by using heat treatment. Stamm, Burr et al. (1946) suggest it would be feasible to commercialize

heat treatment but it was not until beginning of the 90's that the technology developed. Around Europe different methods have been developed. Due to the high temperatures of the process, shielding gas is needed to prevent the wood from catching fire.

In this work the Finnish so called Thermowood method is used where the superheated steam works as a shielding gas. Typically, for heat treatment of softwood, temperatures between 190°C and 220°C are used. This means that the moisture content during the treatment is close to zero percent and that the process before that includes more or less drying (heat treatment can be performed from green wood as well as from pre-dried).

According to Viitaniemi (1997) the most important property changes are:

- The equilibrium moisture content decreases 43-60%
- The shrinkage and swelling decreases 30 – 80%
- The biological resistance (decay resistance) has been improved depending on the treatment circumstances and the product is in the best case equivalent to naturally resistance wood.
- The strength decrease 5 - 25%
- The colour darkens variably

Viitanen, Jämsä et al. (1994) studied the decay resistance of heat treated spruce. They concluded that the decay resistance was improved by 25 – 100% and that the best results are comparable to some CCA-impregnation methods under certain conditions. According to Edlund (2003) heat treatment is a good method for protection against rot and decolorizing fungi, but should not be used in ground contact. Kamden, Pizzi et al. (1999) shows an improved decay resistance, "from non-resistant to moderate/resistant species". In all cases it is observed that the decay resistance is associated with some strength reduction. According to Bengtsson, Jermer et al. (2002) the bending strength of spruce and pine after heat treatment at 120°C during 5 hours is approximately 50% compared to the untreated for full length boards containing knots. This means that heat treated wood is not very suitable for load bearing constructions. Applications where the decay resistance and dimensional stability are important are for instance decking, cladding and window frames.

This study is focused on the development of internal checking during heat treatment and drying. The cracks are oriented in the radial direction mainly along the wood rays, as shown in figure 1. Internal checking is common in high temperature drying of certain hardwoods but is then often closely associated with cell collapse and is initiated during the capillary drying regime. This is not the case for internal checking during the heat treatment process. In this work spruce was used and the question is whether the internal checking is the result of thermal degradation or caused by stress from the drying.

Figure 1. Internal checking in spruce caused by heat treatment.

MATERIALS AND METHODS

The kiln

In this work an experimental kiln for heat treatment of wood has been used, figure 2. The maximum size of the lumber batch including stickers was 500x370x2500mm^3 and due to the small size of the kiln the air was flowing along the boards. For that reason specially designed hollow metal stickers that allow air passage were used.

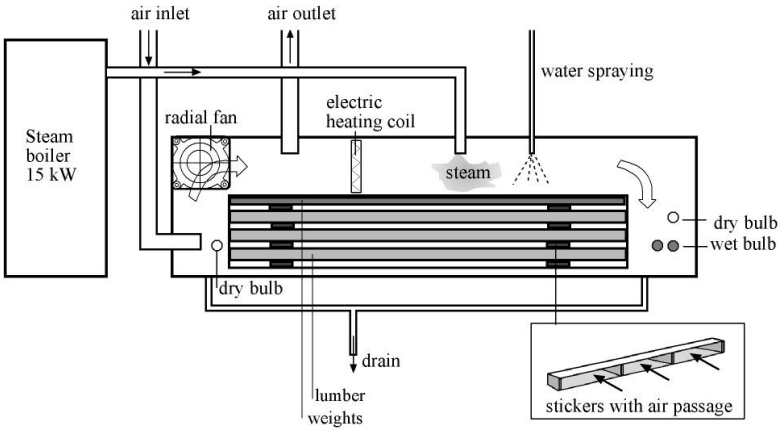

Figure 2. Experimental kiln for heat treatment of wood.

The process

The experiments were divided into three groups. A total of four different process schedules have been used in the three different experiments. In experiment 1, two treatments were performed with the process schedule in figure 3. The processes used were constituted of five regimes as shown in figure 3.

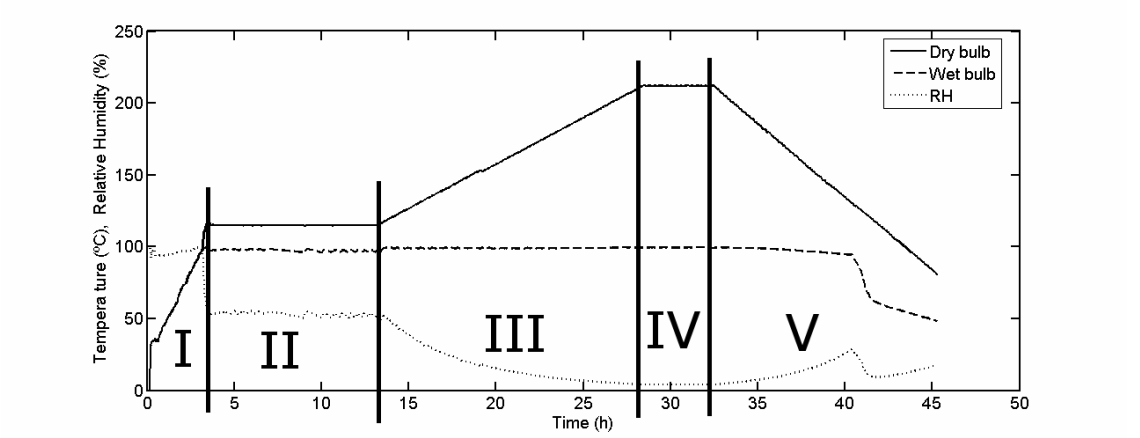

Figure 3. Heat treatment schedule used in this work. The process can be divided into five regimes: **I** Heating, **II** 1st drying, **III** 2nd drying, **IV** Treatment, **V** Cooling regime.

Heating regime: Heating with saturated steam generated at 130°C injected into the kiln at atmospheric pressure.

1st drying regime: This regime is a drying step which can either be high or low temperature drying.

2nd drying regime: This is the regime where the final drying takes place. It is also during this regime that the chemical reactions accelerate.

Treatment regime: The temperature is kept at a constant value normally for 2-4 hours. This time and temperature is normally what defines the heat treatment class.

Cooling regime: Cooling the wood first with steam at 130°C and then with water spraying until the kiln is opened. For industrial practice it is common also to include a conditioning regime for remoistening. In these experiments that has not been necessary.

Experiment 1

In the first experiment two batches of spruce board was heat treated. The first batch of spruce board had the dimension 50x125mm^2 and in the second batch two different dimensions were used, ten 50x100 mm^2 and eight 50x125 mm^2 boards. All boards were weighed and samples were taken for measuring the moisture content. To establish the moisture content of the samples the oven-dry method was used. The dried moisture content samples were then scanned in a CT-scanner to establish the density.

Table 1. Properties of the spruce boards before treatment.

Experiment	Board dimensions [mmxmm]	Moisture content [%]	Density [kg/m^3]	Number of samples
Batch 1	50x125	18.6	413	18
Batch 2	50x125 / 50x100	6.2	385	8/10

After heat treatment the boards were weighed and cut into 5 cm thick slices parallel to the end surfaces. The surface of every cross section was then visually examined and the number of internal cracks was documented. Two slices from each board were used to determine the density. Slices were also taken for prong and slicing tests to determine if there was internal stress in the wood after the treatment.

Experiment 2

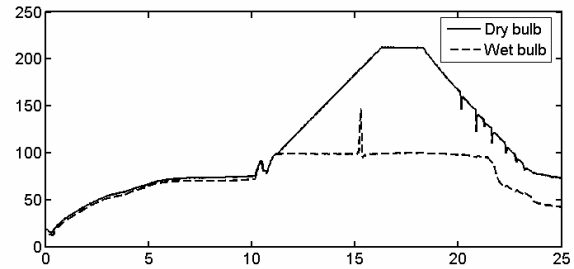

Figure 4. Process schedule used in batch 3

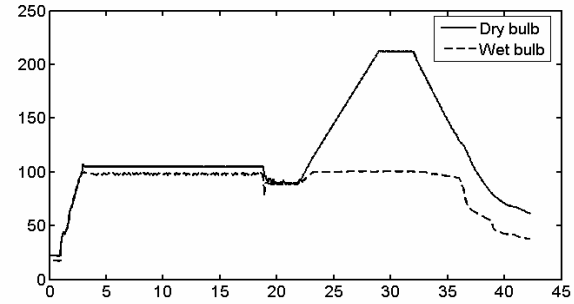

Figure 5. Process schedule used in batch 4

In this experiment two batches of 50x125mm^2 spruce boards were heat treated. In both experiments three groups with different initial moisture content levels were used. The different groups with their corresponding moisture content level are presented in table 2. The heat treatments have been performed according to the process schedules in figure 4 & 5. After treatment the boards were tested for internal checking analogously with experiment 1.

Table 2: Initial moisture content

Batch 3	Batch 4
7.1%	6.4%
8.4%	10.7%
9.7%	14.3%

Experiment 3

In this last experiment twelve boards 50x125mm^2 spruce boards were heat treated. They all had an approximate initial moisture content of 15%. After treatment the boards were also tested for internal checking analogously with experiment 1.

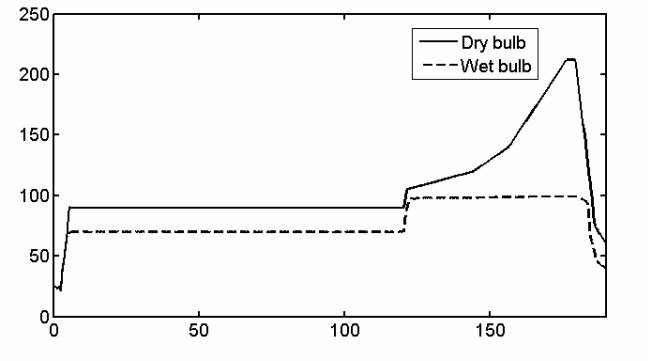

Figure 6. Process schedule used in batch 5

Results

Experiment 1

Table 3: Batch wise presentation of the results of experiment 1

Batch	Initial moisture content [%]	Dimension [mmxmm]	Mass loss [%]	Density loss [%]	Boards with internal checking [%]	Cross sections with internal checking [%]
1	18.6	50x125	8.1	10	67	25
2	6.3	50x125	7.6	6.0	38	10
	6.6	50x100	7.7	7.0	50	26

The results from the treatments are presented in table 3. The objective of the experiment was to study the influence of the initial moisture content and board size on the development of internal checking. But in this first experiment there was no statistically significant effect due to either moisture content or dimension.

All three groups of boards were then divided into two groups, failed and accepted. All the boards with internal checking were classified as failed and all without as accepted. If however the mass loss for individual boards with compensation for moisture was determined, the distribution of mass loss for the two different groups was significant different ($t = 2.312$ and $p=0.0269$).

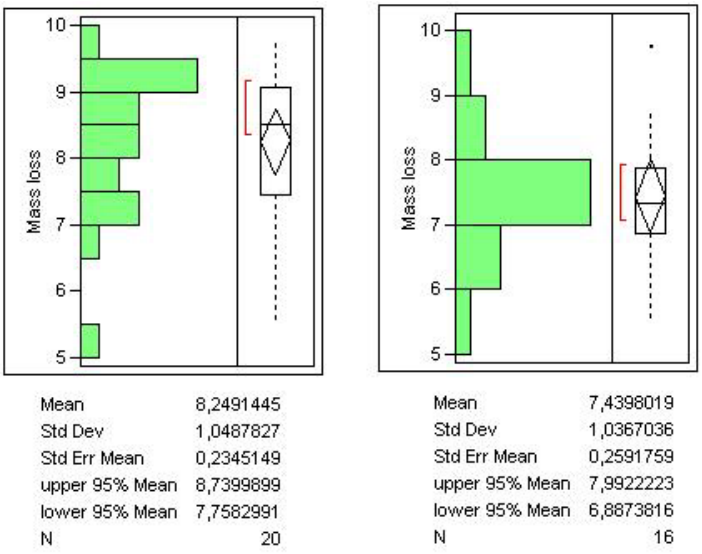

Figure 7. Distribution of mass loss in heat treated boards. Left side is the distribution of mass loss of boards with internal checking. Right side is the distribution of mass loss of boards without internal checking.

The prong and slicing tests did not show any sign of stress. This means there was no or very little stress in the wood after the treatment. Therefore the cracks should not continue to propagate after the treatment.

Experiment 2

Table 3: Results from experiment 2

	Initial moisture content [%]	Cross sections with internal checking [%]
Batch 1	7.1	34
	8.4	46
	9.7	70
Batch 2	6.4	28
	10.7	58
	14.3	90

The results from both batches are presented in table 3. All together there were only two individual boards that were completely free from internal checking, that are why the percentage of the cross sections with checking is presented. Both experiments show the same behavior. The tendency is that groups with higher initial moisture content yield more internal checking.

Experiment 3

In this last experiment internal checking was found in 5 out of 12 boards and in 2.5% of the cross sections from these boards. When checks occurred they were small and originated from the pith.

Discussion & Conclusion

The results show that there was a significant difference in mass loss between boards with and those without internal checking. This indicates that the internal checking in heat treated wood is caused by more than just drying stress. Since there is a mass loss there should be shrinkage of the wood which also might generate stress on a cellular level.

There is a similar pattern in both treatments of experiment 2. The boards with low initial moisture content have less internal checking than the ones with higher initial moisture content.

Apparently it is quite obvious that internal checking is related to initial moisture content. The basic mechanism for internal checking is however not completely understood, at least not based on these experiments.

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Paper III

Possible causes of internal checking of spruce treated above 200°C

Keywords: heat treatment, thermal modification, internal checking, spruce, drying

Author: Dennis Johansson
Address: Luleå University of Technology
Division of Wood Science and Technology
Skeria 3
931 87 SKELLEFTEÅ
SWEDEN
E-mail: dennis.johansson@ltu.se

Abstract

The influence of drying on internal checking of heat treated spruce was studied. Six batches of 50x125mm^2 spruce boards were treated. The boards originated from two groups with different initial moisture content. Three different time spans were used for increasing the temperature from 120°C to 212°C. In addition to the heat treatment, there were two reference groups, one from each initial moisture content group. Both reference groups were treated with the same process as the heat treated ones except that the process was stopped before the temperature increase from 120°C. The result indicates a strong connection between fast drying and increased internal checking.

Introduction

It has long been known that heat can be used to improve durability and dimensional stability of wood (Stamm and Hansen 1937; Thunell and Elken 1948). Stamm, Burr and Kline (1946) suggested that it would be feasible to develop heat treatment as a commercial method for improving durability and stability of wood but it was not until the beginning of the 1990s that the method was commercialized in response to the increased environmental awareness that drives the industry towards reducing the use of chemicals. In this work the heat treatment process used was based on the Finnish Thermowood method (Syrjänen, Jämsä and Viitaniemi 2000) in which steam is used as shielding gas during treatment at elevated temperatures up to 230°C. This method is applied industrially. But there have been problems concerning internal checking of boards with dimensions thicker than 50mm, (figure 1).

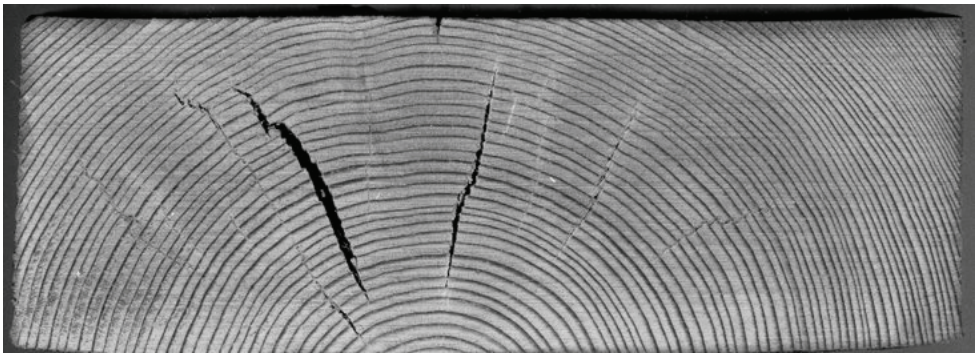

Figure 1: *Example of internal checking of heat treated spruce.*

Johansson (2005) studied internal checking that sometimes occurs when spruce boards of thicknesses greater than 50 mm are heat treated. Internal checking is often a bigger problem than surface checks since internal checking are not visible, which makes quality control more complicated.

Internal checking of boards during drying has mainly been a problem in drying of wood species that easily suffer cell collapse during drying. According to Booker (1994), the initiation of internal checking and collapse during drying are due to the same phenomenon i.e. negative relative pressure during the capillary regime of drying. Later stress reversal leads to tension at the core of boards, which in turn leads to the propagation of internal checks. No signs of correlation between collapse and the internal checking of heat treated boards have been found. Therefore it is assumed that the mechanism of internal checking of heat treated wood is not the same as that involved in high temperature drying.

According to Johansson (2005), both mass loss and drying influences the development of internal checking of heat treated wood, and no stress was detected when slicing tests were performed after the heat treatment. According to Chang and Keith (1978), heat treatment of wood causes anisotropic shrinkage through thermal degradation. One theory to explain

this is that the anisotropic shrinkage caused by thermal degradation generates stress, and that this contributes to internal checking during heat treatment.
To be able to physically explain the phenomenon of internal checking during heat treatment more clearly, it is necessary to know which process parameters influence check development. In this article the focus will be on how drying prior to the actual heat treatment regime affects the development of internal checking.

Materials and methods

Process

The treatments were performed in a small experimental kiln described earlier by Johansson (2005). The treatment process was divided into five regimes as shown in figure 2.

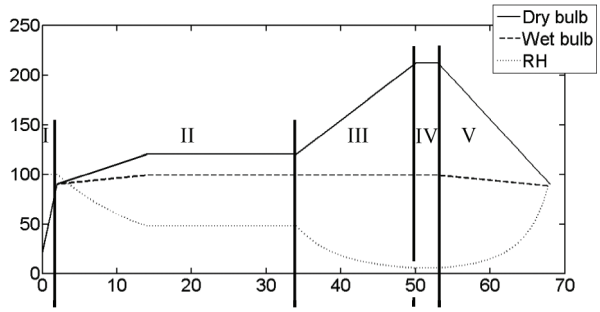

Figure 2: *Process schedules used in the experiment. The dotted line shows relative humidity. Process also divided into five regime: I) Heating regime II) 1st Drying regime III) 2nd drying regime IV) Treatment regime V) Cooling regime*

I) Heating regime: Heating with saturated steam generated at 130°C that is injected into the kiln, which is at atmospheric pressure.

II) 1st drying regime: Drying step that can be either high or low temperature drying (above or below 100°C).

III) 2nd drying regime: The regime during which the final drying takes place. During this regime the chemical reactions accelerate with rising temperature.

IV) Treatment regime: The temperature is kept constant, typically for 2-4 hours. Time and temperature during regime IV normally define the heat treatment class.

V) Cooling regime: Cooling the wood with steam at 130°C followed by water spraying until the kiln is opened. In industrial practice it is common to include a remoistening regime for remoistening. In these experiments, that has not been necessary, since this part of the process does not influence the development of internal checking.

In this study, three different treatment processes at 212°C with different time lengths of the 2nd drying regime were used, 12, 18 and 24 hours respectively, as shown in figure 3.

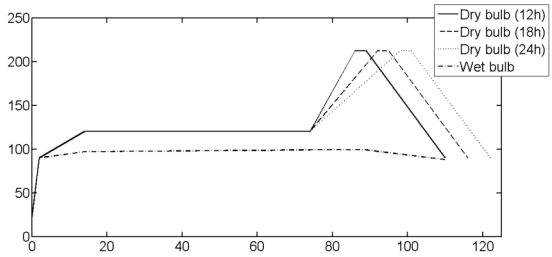

Figure 3: *The different schedules used in this work.*

Boards

All boards used in this experiment originated from the same batch of 50x125mm^2 predried spruce (*Picea abies*) boards with a moisture content MC≈18%. The batch was divided into two sets. One of the sets was additionally dried down to MC≈6% in a 90°C/70°C (dry bulb/wet bulb) drying climate. From each set, three groups of six boards each were taken and cut to about 2 m. The initial moisture content before the heat treatment process was determined through oven dry method (24 h at 103°C) and is shown in table 1.

Table 1: *Average initial moisture content of the 6 batches.*

Group	Treatment	MC (%)	Number of samples	Std dev (%)	Number of boards
1	18% 12 h	18.3 (S. 0.3)	12	0.3	6
2	18% 18 h	17.7 (S. 0.4)	12	0.4	6
3	18% 24 h	17.0 (S. 1.0)	12	1.0	6
4	6% 12 h	5.8 (S. 0.3)	12	0.3	6
5	6% 18 h	6.0 (S. 0.5)	12	0.5	6
6	6% 24 h	5.6 (S. 0.1)	12	0.1	6

After heat treatment, all boards were crosscut into 5 cm thick slices. One end surface of each slice was visually examined, and the number of checks was documented. Besides internal checks, the presence of knots and pith within each cross section was documented. Checks appearing in knots were not graded as internal checking.

Reference boards

In addition to the six groups, two reference groups were used. The first group contained six of the boards with 18% MC (Ref. 1) and the second group consisted of five boards from the batch of predried boards with 6% MC (Ref. 2). Both reference groups were treated according to the process schedule in figure 3 but the process was stopped after the 1st drying regime. After this process, the moisture content, split gaps and internal checking were measured. Two different slicing tests were performed. The first test set was cut immediately after treatment and then kept in plastic bags for 48 hours so the moisture had time to equalise. The second test was performed about five months after treatment. The test samples were cut from the 5 cm thick samples used for inspection of internal checking. The split gaps in the second slicing test were measured directly after cutting.

Results

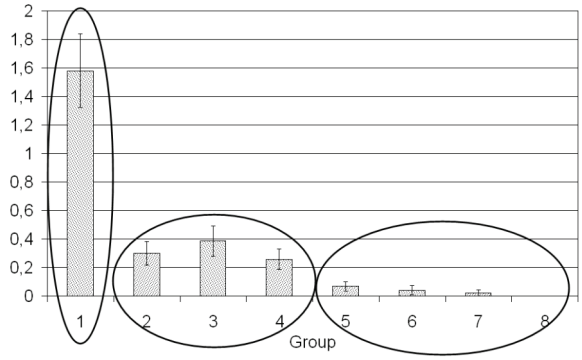

Figure 4: *Mean number of internal checks per cross section together with bars showing the 95% confidence interval. Circles show groups with statistically significant different number of internal checks with 95% confidence.*

The results in figure 4 show the eight groups divided into three levels with a significantly different number of internal checks. The tendency was for a low initial moisture content and long duration of the 2nd drying regime to reduce the number of internal checks.
The second reference group did not have a single internal check whilst the first reference group had 0.02 checks per surface. However, the checks were associated with the presence of pith within the cross section. The same tendency was found in the test as a whole when the test pieces were divided into cross sections with and without pith (see table 2). These results showed a significant ($t = 7.98$ and $p < 0.001$) difference in the number of checks.

Table 2: *Average number of checks in each cross section. Divided on whether there was pith in the cross section or not.*

Group	Average	Number of samples
No pith within the cross section	0.22	1164
Pith within the cross section	0,61	684

MC and split gap in reference boards

The results of moisture content from the reference boards are shown in table 3 and the difference between the two groups is statistically significant ($t = 2.49$ and $p = 0.019$).

Table 3: *Moisture content in the high temperature dried reference boards.*

Group of initial moisture content	Number of samples	Average moisture content	Standard error
6%	10	3.8	0.06
18%	12	4.0	0.06

Slicing tests also revealed a difference between the two reference groups. In slicing tests, all samples from Ref. 1 had split gaps. In the slicing tests performed directly after the 1st drying regime, in which the samples were kept in plastic bags for 48 hours, the gaps in samples from Ref. 1 approximately 1 mm (see figure 5), whereas the ones from Ref. 2 did not have any split gaps. The second slicing that was done after five months storage immediately showed gaps of approximately 0.4 mm in Ref. 1 and 0 mm in Ref. 2.

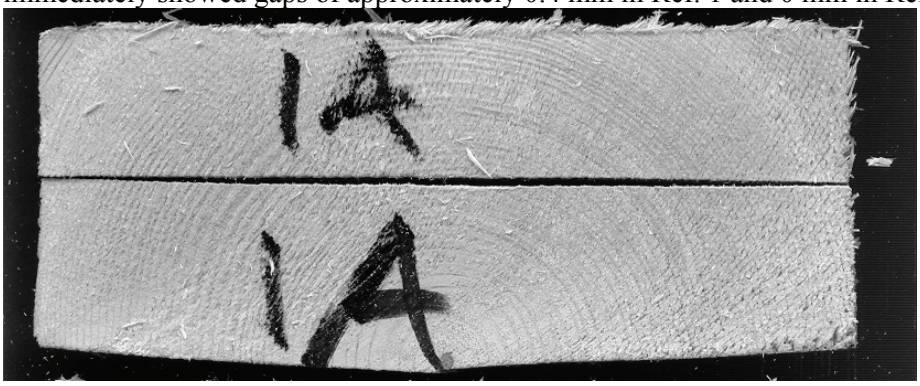

Figure 5: *Slicing test from high temperature dried reference boards. Gap due to approximately 1 mm. Samples were cut after the 1st drying regime.*

Discussion

From the results in this study it is clear that drying procedures influenced the development of internal checking. Also, the results showed that when pith was present within the cross section the average number of internal checks was three times higher. The connection between pith and internal checking was expected. The juvenile wood surrounding the pith is sensitive and the effects of the anisotropic shrinkage are large because of the short distance to the centre.

There was a significant difference in split gap between Ref. 1 and Ref. 2. The difference in number of internal checks between the two groups was probably a result of drying stress due to the fact that more stress is likely to be introduced by high temperature drying above 100°C. Hanhijärvi (2000) showed an increased mechanosorptive creep during high temperature drying of pine and spruce, a factor that could cause increased stress in Ref. 1. It is also possible that the difference in stress was an effect of relaxation in Ref. 2 due to high temperature during the 1st drying regime. The slicing test showing a split gap of 1mm was an indication of internal stress together with the influence of the moisture gradient, whereas the split gaps revealed in the slicing test performed after five months storage were only due to stress. A gap of 0.4 mm in the second slicing test was used to get an approximation of the level of internal stress.

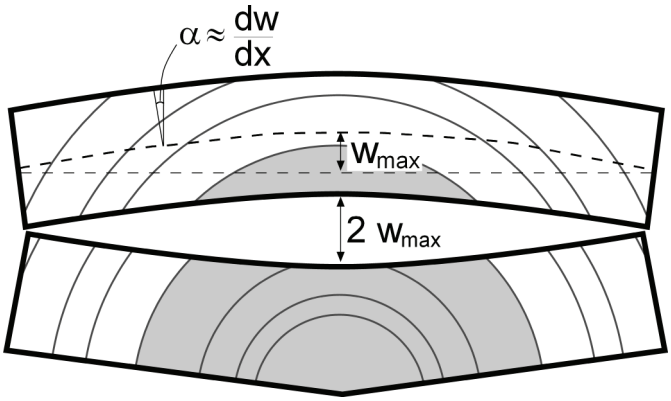

Figure 6: *The measured gap in the slicing test is assumed to be twice the deflection of each part. It is also assumed that the centre lines of the two parts are sinus function along the dotted line.*

The gap in the second slicing test revealed an average gap of 0.4 mm. To do this estimation of the stress in the cross section the deflection was assumed to be a sinus function as shown in figure 1 and equation 1.

$$w = 0.2 \cdot \sin\left(\frac{x}{125}\pi\right) \quad (1)$$

From figure 1 it can also be seen that the displacement (u) for small deformation can be expressed as equation 2.

$$u = -\frac{dw}{dx}z \quad (2)$$

which then gives equation 3

$$\varepsilon = \frac{du}{dx} = -\frac{d^2w}{dx^2}z \quad (3)$$

The cross section was assumed to be cut into two equally thick slices. Hence z = 12.5 mm. This results in an extension of 0.16%. Then, if Hooke's law, equation 4, is applied, it is possible to get an estimation of the stress inside the wood.

$$\sigma = E \cdot \varepsilon \quad (4)$$

Modulus of elasticity was set to 245 MPa based on Siimes (1967) data for spruce with 4% MC, 0.4 g cm^{-3} and 80°C. This gives an average stress level of 0.4 MPa. High temperature reduces the modulus of elasticity but the calculation is just done to show level of stress and not the exact value.

The stress calculated from the slicing tests is not likely to cause internal checking on its own but stress is not negligible. However, it should be remembered that the final stress state does not reflect the stress that the interior of the sample has been subjected to during the full treatment cycle. During the 2nd drying regime, additional stress is likely to be generated and then the stress of 0.4 MPa can be the difference between internal checking occurring or not. A reduction of the tensile strength in the tangential direction due to heat could make the wood more sensitive to stress and more prone to crack. This reduction could be an effect of both strength loss due to thermal degradation (Stamm 1956; Rusche 1973; Hanhijärvi 1999a), or just a reduction due to wood being subjected to high temperatures.

Faster temperature increase during the 2nd drying regime results in faster shrinkage due to both drying and thermal degradation which would mean the wood had less time to creep and adapt to stress development, thus leading to more internal checking. On the other hand, Hanhijärvi (1999b) has shown that high temperatures should result in more visco-elastic creep, which means that stress is reduced. The difference is that this experiment was performed at lower moisture contents than Hanhijärvi 1999b. This might be an important difference because of the change in glass transition temperature of lignin and hemicelluloses. According to Salmén (1982), the glass transition temperature for lignin and hemicelluloses is between 180°C and 220°C. When wet, the lignin is lowered to 80°C–90°C and the hemicellulose to room temperature. This means the wood will be stiffer and generate higher stress with the same strain when it is dry while at the same time being less inclined to creep. This is also supported by Passard and Perré (2004), who suggest that high-temperature drying could benefit from using higher temperatures to keep the wood soft to prevent cracking. This would also explain the results in Johansson (2005), where no stress was found in slicing tests performed after heat treatment, *i.e*. the relaxation of stress through softening of the wood.

Another contributing factor to explain why the different MC groups yielded different amounts of internal checks could be that there was a development of microcracks of the type discussed in Terziev and Daniel during the 1st drying regime. That would mean that the internal checking would be initiated by the drying previous to the 2nd drying regime. But if internal checking were to be caused only by thermal degradation, then a longer time in the 2nd drying regime should result in more internal checking, since it is then subjected to high temperature for a longer time. Microcracks could also have been caused by drying stress, not thermal degradation. That would also help to explain the difference in internal checking between the two levels of initial MC. This means there might be stress on a microlevel that does not show in a slicing test but which still damages the cells.

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