

Thermophysical Properties of Beech Wood in the Range from Room Temperature to 900 °C

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Abstract

Wood is a structural material of biological origin that undergoes thermal degradation when exposed to high temperatures. Additionally, wood shows an anisotropic behavior in terms of thermal expansion and thermal conductivity along and across fiber direction. This work reports thermophysical measurements of beech wood from room temperature up to 900 °C. The wooden material was investigated in different states: moist, dry, charred and during pyrolysis. A push-rod dilatometer was used to measure thermal expansion, from which temperature dependent density was derived. Specific heat was determined by differential scanning calorimetry. A laser flash apparatus was applied to measure thermal diffusivity. Thermal conductivity was calculated from thermal diffusivity, specific heat, and density. The measurements of thermal expansion and thermal diffusivity were performed along and across fiber direction to consider the anisotropic behavior of wooden material. The results of the thermophysical properties are reported from room temperature to 200 °C for the beech wood, during pyrolysis, and up to 900 °C for the charred material. It was found that thermal expansion of beech wood across fiber direction is greater than along fiber direction in the order of a magnitude. In contrast, thermal expansion of charred material is rather independent on fiber direction. Thermal conductivity of beech wood along fiber direction was found to be approx. 2 to 3 times higher than across fiber direction. In the case of the charred material the relative difference is smaller.

Keywords Beech wood \cdot Charred wood \cdot High temperature \cdot Pyrolysis \cdot Thermal conductivity

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1 Introduction

Timber, as a natural material and renewable resource, is attracting growing attention in the construction industry within the last decades. The insulation behavior of wooden material is not only affecting the thermal comfort of living spaces [1], but also the energy efficiency of buildings [2]. Nowadays, structural materials should not only guarantee sufficient mechanical resistance and durability properties [3], but also energy efficiency, which has become one of the most important criteria [4]. The growing interest in "green construction" requires sustainable renewable materials [5] in the construction industry. The use of wooden materials can make a significant contribution in improving the overall energy efficiency of buildings. Therefore, wood is not only used in traditional areas such as detached houses, but also in high-rise buildings in urban areas, showing high potential regarding a sustainable densification of urban regions.

However, wooden material undergoes thermal degradation when exposed to high temperature. This process is a complex series of reactions, starting with the release of wood moisture, followed by the decomposition of the wood cell structure [5], forming a surrounding char layer. Nevertheless, depending on fire exposure and element dimensions, the core section of the wooden elements may remain stable for a certain amount of time [6]. Building regulations require a defined fire resistance for wooden structures to allow a safe evacuation of people in the case of fire [7]. It is important, therefore, to determine the fire resistance and the charring rate of wooden structures and furthermore the thickness of the residual core structure of wooden elements in the case of fire, as these parameters are crucial in fire safety design [6].

The fire resistance of timber structures is usually determined and verified by cost and time-consuming fire tests. In contrast, numerical models to predict the fire behavior of wooden structures are a promising approach in terms of introducing flexibility and cost-effectiveness in the development of wooden elements [7]. The study of the fire resistance of various wooden structures by computer models is therefore of great interest [6]. However, the development of numerical model requires thermomechanical and -physical properties of wooden materials, not only at room temperature, but also at elevated temperatures. Temperature dependent density, specific heat, and thermal conductivity are required as thermophysical properties.

Additionally, latent heat of evaporation and heat of pyrolysis have to be known. Room temperature data of various wood species and materials are often reported: e.g., Simpson and TenWolde [8] report that specific heat of wooden material is almost independent of wood species. Thermal conductivity of numerous hardwood and softwood species is reported in the dry and moist state; a summary of thermal conductivity measurements for wood using different measurement methods is given by Hu *et al.* [9]. At high temperatures, however, thermophysical properties data of wood are sparse in literature [10]. For example, Eurocode 5, part 1–2 [11] and Mehaffey *et al.* [12] do not distinguish between different wood species, density, moisture content, or the anisotropic behavior of wooden materials.

This work reports thermophysical measurements of beech wood over a wide temperature range, considering different states and the anisotropic behavior of this material. The beech is the most important broadleaved wood species in Europe and has a wide range of applications in the construction industry [13]. In contrast to soft wood species, beech wood has a higher load-bearing capacity to exceed the demands for low dimensioned structural timber products.

The beech wood samples were investigated in the moist, dry, and charred state, as well as during pyrolysis. A push-rod dilatometer was used to measure thermal expansion, from which temperature dependent density is derived. Specific heat was determined by differential scanning calorimetry. A laser flash apparatus was applied to measure thermal diffusivity. Thermal conductivity was calculated from thermal diffusivity, specific heat and density. The results of the thermophysical properties are reported in the temperature range from room temperature to 200 °C for the beech wood, during pyrolysis, and up to 900 °C for the charred material.

As wood is an anisotropic material, thermal diffusivity, thermal expansion and thermal conductivity depend on fiber direction. Therefore, the corresponding measurements were performed on specimens cut along and across the fiber direction, i.e., in longitudinal direction and transversal direction, respectively.

2 Experimental Methods and Data Reduction

2.1 Material

Two beech wood samples from the same origin were used in this work that are shown in Fig. 1. The sample dimensions were in the range of approx. $160 \times 50 \times 20$ mm³. From the beech wood samples, specimens were prepared for subsequent measurements in different wood fiber directions: (a) longitudinal direction, that is parallel to the fiber grain, (b) transversal direction that is across fiber direction, combining both, radial and tangential direction. After milling of the specimens and finishing



Fig. 1 Test samples of beech wood for measuring thermophysical properties in different directions

with 320 grid paper to achieve proper dimensions, the specimens were prepared for subsequent measurements in three different states: moist, dry and charred.

An equilibrium wood moisture content (MC) of 10.5 ± 0.5 % was chosen for the measurements in the moist state, that corresponds to a water content of approx. 9.1 wt-% to 9.9 wt-%. A MC of approx. 10.5 % is achieved by storing wood at a relative humidity of approx. 60 % and corresponds to an average wood moisture used in construction industry. Larger samples for density measurements at room temperature were stored in a conditioning cabinet at 20 °C and a relative humidity of 60 ± 1 % for approx. one week, smaller samples for thermogravimetric analysis, DSC, and measurement of thermal expansion and thermal diffusivity were stored for at least 72 h.

Specimens to be investigated in the dry state were dried in a drying cabinet according to the Darr method at 103 $^{\circ}$ C for at least 72 h; larger samples for approx. one week.

The measurements in the charred state were performed on pyrolyzed samples. Shrinkage due to pyrolysis along and across fiber direction was found to be approx. 22 % and 37 %, respectively. Considering the shrinkage of wood during pyrolysis in the different directions, samples with greater dimension than described in Sects. 2.4 to 2.6 were prepared and submitted to the heat treatment process. The charring of the samples was performed in a furnace with an Al₂O₃ sample holder that was evacuated and flushed with argon gas 3 times in order to minimize residual oxygen in the furnace chamber. The treatment of the samples implied a heating from room temperature to approx. 1030 °C at a heating rate of 10 K ·min⁻¹ under protective atmosphere (argon with a flow rate of 100 ml·min⁻¹). After the heat treatment, the charred samples had to be finished with 800–2500 grid paper due to slight distortion of the samples depending on the wood fiber to achieve samples with proper dimensions. Furthermore, charred samples were stored and pre-dried in a drying cabinet at 103 °C prior to the measurements to avoid excessive moisture pick-up due to the porous character of the charred wood material.

2.2 Measurement of Density at Room Temperature and Control of the Wood MC

Density of beech wood at room temperature, in the moist, dry and charred state was determined from dimensions and mass. From each of the two beech wood samples a specimen of smaller dimensions was taken for measurement of density at room temperature. Sample dimensions were in the range of approx. $50 \times 30 \times 20$ mm³ and were stored according to the aforementioned procedure to perform measurements in the moist and dry state. In the charred state specimen dimensions were in the range of approx. $23 \times 7 \times 5$ mm³.

Wood MC of the density samples was determined according to [14]:

$$Moisture\ content(MC)[\%] = 100 \times \frac{Weight\ of\ moist\ wood\ -\ Ovendry\ weight}{Ovendry\ weight}$$
(1)

2.3 Thermogravimetric Analysis

Mass loss data during the heating of the beech wood samples were obtained using a simultaneous thermal analyzer of NETZSCH Gerätebau, Selb, Germany, type STA 449 F3 Jupiter, equipped with a DTA sample holder and alumina crucibles with a volume of 0.3 ml. Sample dimension for measuring of the mass loss from room temperature to 900 °C were in the range of approx. 5 mm in height and 5 mm in diameter. The sample weight of moist beech wood samples was approx. 70 mg to 80 mg. After conditioning, samples were placed in the alumina crucible. The balance chamber was slightly flushed with argon gas with a flow rate of 20 ml·min⁻¹ to protect the balance. A dwell time of approx. 10 min was applied prior to the measurement run to stabilize the balance. The measurements were performed on each sample during two heating cycles.

The first run: There was no evacuation of the furnace chamber prior to the first measurement run to avoid significant loss of moisture content. Subsequently, the individual samples were heated from room temperature to approx. 150 °C (ambient air with a relative humidity of approx. 30 % to 40 % and a slight argon protective gas stream coming from the balance chamber). Cooling from 150 °C to room temperature under same conditions. The second run: The furnace and balance chambers were evacuated and flushed with argon 3 times to minimize residual oxygen. Subsequent heating from room temperature to 900 °C under protective atmosphere. Two heating rates were applied, 2 K·min⁻¹ and 5 K·min⁻¹ to account for the kinetics of moisture release and pyrolysis reaction.

2.4 Thermal Expansion and Temperature Dependent Density

Thermal expansion of moist, dry and charred beech wood material was measured by a single push-rod-dilatometer of NETZSCH-Gerätebau, type DIL 402 Expedis Select. Sample holder and push-rod were made of alumina. Sample dimensions were nominally 25 mm in length and 5 mm in width and height in the moist and dry state. In the charred state, smaller specimens with approx. 12 mm in length were prepared due to distortion of larger samples depending on the grain structure. Samples were nominally 5 mm in width and height. In each state, moist, dry, and charred, 6 individual samples were measured, 3 samples in longitudinal and 3 samples in transversal direction.

Moist samples were placed in the sample holder and the measurement was immediately started without prior evacuation of the furnace chamber to avoid significant loss of wood moisture. During measurement, the specimen was heated from room temperature to 200 °C at a heating rate of 2 K·min⁻¹ in ambient air (approx. 30 % to 40 % relative humidity).

The individual dry samples were placed in the sample holder and two consecutive measurement runs were performed: (a) heating from room temperature to 110 °C at a heating rate of 2 K·min⁻¹ to remove potential residual moisture within the wood cell structure. Subsequently, the sample was cooled down to room temperature under protective atmosphere (helium gas); (b) heating from room temperature to 200 °C. Both measurement runs were performed under protective atmosphere (a mixture of helium and argon gas) with prior evacuation and purging of the furnace chamber 3 times.

In the charred state, samples were measured from room temperature to 900 °C at a heating rate of 3 K·min⁻¹ under protective atmosphere (a mixture of helium and argon gas) after evacuation of the chamber for 3 times. Two consecutive measurement runs were performed after pre-drying of the samples.

Temperature dependent density in the dry $\rho(T)_{dry}$ and charred state $\rho(T)_{charred}$ was calculated from density at room temperature $\rho(T)_{0,dry,charred}$ and the thermal expansion $\Delta l(T)/l_{0_{dry,charred}}$, considering both, thermal expansion in longitudinal and transversal direction according to a modified version of the equation given in [15]:

$$\rho(T)_{dry,charred} = \frac{\rho_{0,dry,charred}}{\left(1 + \frac{\Delta l(T)_{longitudinal}}{l_{0,longitudinal}} dry,charred\right) \left(1 + \frac{\Delta l(T)_{transversal}}{l_{0,transversal}} dry,charred\right)^2}$$
(2)

In the temperature range of moisture release (room temperature to 200 °C) the density evolution $\rho(T)_{moist}$ was calculated from mass loss derived from thermogravimetric analysis and the volumetric contraction of moist wood obtained from dilatometric measurements, considering the both, contraction along and across the wood grain fiber. The mass loss and the thermal contraction were derived at a heating rate of 2 K·min⁻¹ from:

$$\rho(T)_{moist} = \frac{m(T)}{m_0} \frac{\rho_{0,moist}}{\left(1 + \frac{\Delta l(T)_{longitudinal}}{l_{0,longitudinal} moist}\right) \left(1 + \frac{\Delta l(T)_{transversal}}{l_{0,transversal} moist}\right)^2},$$
(3)

where $\rho_{0,moist}$ is the density of moist wood at room temperature, m(T) is the actual mass as a function of temperature, m_0 is the initial mass, and $\Delta l(T)/l_{0_{moist}}$ is the thermal contraction.

In the temperature range of pyrolysis from approx. 200 °C to 900 °C the density evolution $\rho(T)_{pyro}$ was approximated from the extrapolated dry wood density $\rho(T)_{dry}$, the charred density $\rho(T)_{charred}$, and the mass change from thermogravimetric analysis using a dimensionless sample conversion:

$$X(T) = \frac{m_{200 \,\,^{\circ}\text{C}} - m(T)}{m_{200 \,\,^{\circ}\text{C}} - m_{900 \,\,^{\circ}\text{C}}} \tag{4}$$

$$\rho_{pyro}(T) = \rho_{dry,extrapolated}(T)(1 - X(T)) + \rho_{charred}(T)X(T)$$
(5)

2.5 Specific Heat

The specific heat of the beech wood specimens in the different states was determined by heat-flow differential scanning calorimeters of NETZSCH-Gerätebau, type DSC 404 F1 Pegasus (Setup 1) and type DSC 204 F1 Phoenix (Setup 2). Furthermore, the aforementioned simultaneous thermal analyzer of NETZSCH Gerätebau, type STA 449 F3 Jupiter was used, equipped with a DSC sample holder and platinum crucibles with a volume of 85 μ l (Setup 3). The sample dimensions in the different states were nominally 6 mm in diameter and 1.5 mm in height with a sample mass of approx. 30 mg in the case of wooden material and approx. 20 mg in the case of charred beech wood. For the measurements in the moist and dry state 4 individual samples were used. Apparent specific heat of the selected moist wood specimens was determined, consecutively specific heat of dry wood was measured using the same individual specimens. In the charred state, again 4 individual samples were measured.

The mass of the sample in the moist state was determined by a balance immediately after taking the individual samples from the conditioning cabinet. Subsequently, the individual specimens were transferred into the measurement chamber of the DSC (Setup 2) and placed in aluminum crucibles (85 µl) with a lid with a small hole. The samples were rapidly cooled to -10 °C, followed by two consecutive measurement runs in nitrogen atmosphere with a heating rate of 5 K \cdot min⁻¹: (a) heating from - 10 °C to 150 °C to determine the apparent specific heat of moist wood. Subsequently, the samples were cooled down to -10 °C; (b) heating from - 10 °C to 150 °C to determine the specific heat of dry beech wood. The individual specimens were then transferred directly to a DSC (Setup 1) and placed in platinum crucibles with lid where the temperature range of the specific heat of dry beech wood was extended to 200 °C. The measurements were performed in argon atmosphere at a heating rate of 10 K·min⁻¹. Prior to the measurement run, the furnace chamber was evacuated and purged with argon 2 times to avoid residual moisture content. In the case that the samples could not be transferred directly, the samples were stored in a drying cabinet at 103 °C to avoid absorption of moisture. A first measurement run was performed from room temperature to 160 °C, followed by a second run up to 200 °C on each sample.

The specific heat of pre-dried samples in the charred state were measured in a DSC (Setup 1) in platinum crucibles in argon atmosphere. The heating rate was 20 K \cdot min⁻¹ and two measurement runs were performed from room temperature to 900 °C.

Four individual samples were measured using a STA (Setup 3) with platinum crucibles with a lid that has a small hole for pressure equalization to determine the apparent specific heat in the temperature range of pyrolysis above 200 °C. Sample dimensions were 6 mm in diameter and approx. 0.5 mm in height with a sample weight of approx. 20 mg. Samples were pre-dried to avoid excessive moisture pick-up. After placing the individual samples in the platinum crucible the furnace chamber was evacuated and purged with argon 2 times to avoid residual oxygen. The individual samples were heated up to 150 °C, cooled down to 100 °C and stabilized for 30 min. Subsequently, the samples were heated up to 700 °C at a heating rate of 10 K·min⁻¹. The purge gas was argon with a flow rate of 150 ml·min⁻¹, additionally to argon protective gas with a flow rate of 60 ml·min⁻¹ to protect the balance.

2.6 Thermal Diffusivity

A laser flash apparatus of NETZSCH-Gerätebau, type LFA 427 was used to determine thermal diffusivity of the beech wood samples in three different states, moist, dry and charred. The samples were nominally 12.5 mm in diameter and 3 mm in height. Charred sample dimensions were approx. 10 mm in diameter and 3 mm to 4 mm in height. Four individual samples were measured in each direction in the case of wooden material, 3 individual samples in each direction in the case of charred material.

Moist and dry beech wood samples were coated both, at the bottom and top surface. At the bottom surface a conductive acrylic adhesive copper tape of 3 M, type 1181 was used. The nominal thickness of both, the copper foil and the acrylic adhesive was 66 μ m. The coating at the bottom surface was used to protect the samples from the laser beam to avoid local charring of the sample surface. At the top surface a thin layer of a conductive heat sink compound of DOW CORNING, type 340 was applied. The thickness of the layer was approx. 80 μ m. The coating at the top surface was required due to the porous character of the wooden material to prevent an early detection of the signal coming from the porous structure. Furthermore, the layer system (sample + bottom and top coating) was additionally covered by a thin layer of graphite (approx. 5 μ m on each side).

The thickness of moist wood samples was measured by a micrometer caliper immediately after conditioning according to the aforementioned procedure, following by rapidly coating the samples and measurement at room temperature in ambient air (approx. 30 % to 40 % relative humidity). Higher temperatures were not included in the measurement of moist wood samples, as long times to equilibrate the samples at elevated temperature would lead to an undefined moisture content. In contrast, dry wood samples were measured from room temperature to 200 °C in steps of 20 °C, again in ambient air. Reference measurements were carried out using black Bakelite (Struers, phenolic hot mounting resin with glass filler) and a fibrous refractory board of Pyrotek, type N-17, to take into account the influence of the layer system on thermal diffusivity of moist and dry wood. These materials can be measured both, with and without layer system using the laser flash technique. It was found that the true thermal diffusivity of the wooden sample can be calculated by making a small correction of the measured thermal diffusivity (Cape-Lehman's model [16] with pulse length correction was used for evaluating thermal diffusivity) for the total thickness of the sample (wooden sample and layer system). However, an additional uncertainty contribution in the order of a few percent must be taken into account in the case of estimating expanded measurement uncertainty.

Charred beech wood samples were measured without an additional coating system. However, due to the porous character of charred material, a model for evaluating thermal diffusivity was used that takes into account the penetration of the laser pulse into the material. The Cape-Lehman's model [16] with pulse length correction assumes that the pulse energy is totally absorbed on the bottom surface of the sample. The penetration model [17], based on the work of McMaster *et al.* [18], takes in account that the absorption of the pulse energy extends over a thin layer of the sample thickness. The charred material was measured in both directions from 100 °C to 900 °C in steps of 100 °C in argon atmosphere. Prior to the measurement run, the furnace chamber was evacuated and flushed with argon.

3 Results

3.1 Thermogravimetric Analysis

The mass loss of the beech wood samples with increasing temperature from room temperature to 900 °C is shown in Fig. 2. The first-order derivative at a heating rate of 5 K·min⁻¹ is also plotted. Two significant mass changes occur: in the range of approx. 100 °C and between 200 °C and approx. 350 °C. From room temperature to approx. 150 °C the mass of moist beech wood samples decreases approx. 9.5 wt%, corresponding to a MC of 10.5 %. The total mass loss due to the release of moisture is independent of the heating rate. However, a slight kinetic effect can be observed between room temperature and 100 °C. A plateau can be seen in the temperature range from approx. 150 °C to 200 °C with a mass change rate close to zero at about 150 °C and only very little mass loss up to 200 °C. However, a significant mass loss can be observed above 200 °C due to the pyrolysis reaction and the transformation of wood into charcoal. Therefore, 200 °C is specified as the stability temperature limit of wooden material. Above 500 °C, the pyrolysis reaction seems to be rather finished, with little mass loss up to 900 °C under protective atmosphere. The residual mass of the pyrolyzed samples is roughly 20 % of the raw material.



Fig. 2 Mass loss of the beech wood samples with increasing temperature from room temperature to 900 °C at heating rates of 2 and 5 K·min⁻¹. First-order derivative (mass change rate) at a heating rate of 5 K·min⁻¹ (short dashed curve)



Fig. 3 Thermal expansion of the dry beech wood samples in longitudinal and transversal direction



Fig. 4 Thermal expansion of the moist beech wood samples in longitudinal and transversal direction

3.2 Thermal Expansion and Temperature Dependent Density

Figures 3 and 4 show the results of measurement of thermal expansion of the dry and moist beech wood samples in longitudinal and transversal direction. Thermal expansion in transversal direction is significantly higher than that in parallel to the wood fiber for dry beech wood in the order of a magnitude. The same applies to the thermal contraction of moist wood. The density was derived from the results of thermal expansion as described in Sect. 2.4. As depicted



Fig. 5 Density of the moist beech wood samples at room temperature (MC = 10.5 ± 0.5 %), temperature dependent density from room temperature to 200 °C of dry wood and calculated density evolution due to the release of wood moisture

in Fig. 5, the difference between density at room temperature of moist wood $(MC = 10.5 \pm 0.5 \%)$ and dry wood is approx. 20 kg·m⁻³. Temperature dependent density of dry beech wood decreases from room temperature to 200 °C in the range of approx. 1 %. Furthermore, the density evolution due to the release of moisture is also plotted, calculated according to Eq. 3. The difference between the temperature dependent density derived from Eqs. 2 and 3 is small, lower than 1 %. The expanded measurement uncertainty for the density is mainly determined by the uncertainty of the room temperature value and is estimated to be approx. ± 1 % at room temperature to 200 °C in the dry state. All estimated uncertainties of measurement reported in Sect. 3 of this work are stated as the standard uncertainty of measurement multiplied by the coverage factor k = 2, which for a normal distribution corresponds to a coverage probability of approx. 95 %.

The thermal expansion of the charred material is plotted in Fig. 6 from room temperature to 900 °C. In contrast to thermal expansion of dry and moist beech wood, only a very small dependency on fiber direction can be found for charred material. The density as a function of temperature was derived according to Eq. 2, indicating an almost negligible variation of density with increasing temperature that is shown in Fig. 7. The density approximation calculated according to Eq. 5 is also shown and gives an indication of the density evolution in the temperature range of pyrolysis. In the charred state an expanded uncertainty of ± 5 % is estimated from room temperature to 900 °C.



Fig. 6 Thermal expansion of the charred beech wood samples along and across fiber direction from room temperature to 900 $^{\circ}\mathrm{C}$



Fig. 7 Density of the beech wood samples, temperature dependent density of charred material and interpolated density evolution due to pyrolysis reaction

3.3 Specific Heat

The specific heat of dry beech wood samples is shown in Fig. 8. The obtained data were fitted by a least squares fit and shows an almost linear behavior from room temperature to 200 °C. This is for practical reasons in the temperature interval from room temperature to 200 °C to calculate thermal conductivity, assuming no residual



Fig.8 Specific heat of the dry beech wood samples from room temperature to 200 °C (fit), showing measurements from two DSC apparatus: from 20 °C to 150 °C (triangle up) and from 100 °C to 200 °C (triangle down)

moisture absorbed to the cell walls. The specific heat of dry beech wood is shown again in Fig. 9 together with that of moist wood. Using the mass loss data from the STA measurements at a heating rate of 5 K·min⁻¹, the apparent specific heat related to initial mass was corrected for the actual mass. The measurement uncertainty for the specific heat is influenced by the repeatability between individual specimens. In the moist state the expanded measurement uncertainty for the specific heat is



Fig. 9 Apparent specific heat of the selected moist wood specimens related to the actual mass and compared to the specific heat of the dry wood samples

estimated to be ± 3 % at room temperature and up to ± 5 % at elevated temperatures. In the dry state an expanded measurement uncertainty of ± 3 % is estimated in the temperature range from room temperature to 200 °C.

The specific heat of the charred material is shown in Fig. 10. The measurement results from 130 °C to 800 °C are included in the plot (individual measurements of 2 times heating and cooling). The obtained data were fitted by a cubic polynomial. The first heating run performed on each sample is not included due to potential moisture pick-up. Additionally, the data of graphite [19] are also shown, indicating a negligible difference between charred material and graphite. In the charred state the expanded measurement uncertainty is estimated to be up to ± 5 % in the temperature range from 130 °C to 800 °C.

The apparent specific heat of the selected specimens in the temperature range of pyrolysis is shown in Fig. 11, combined with the specific heat of dry beech wood and charred material. Due to the change in sample dimensions in diameter and height, furthermore due to potential distortion of the sample, a good contact required for specific heat measurements cannot be guaranteed. Therefore, the specific heat in the temperature range of pyrolysis is only shown from 200 °C to 500 °C. The short dashed curve represents an interpolated specific heat without reaction, derived from a mixture rule of extrapolated specific heat of dry wood and of charred material. Comparing specific heat in the temperature range of pyrolysis to the interpolated curve, a sequence of only exothermic reactions is found for beech wood, which is in contrast to other wooden material, such as spruce [20].

3.4 Thermal Diffusivity and Thermal Conductivity

Thermal diffusivity of the moist and dry beech wood samples in longitudinal and transversal direction is shown in Fig. 12. In the case of the dry wood thermal diffusivity



Fig. 10 Specific heat of the charred beech wood samples compared to the data of carbon [19]



Fig. 11 Apparent specific heat of the selected beech wood specimens in the temperature range of pyrolysis. Data of spruce wood [20] is also included



Fig. 12 Thermal diffusivity of the moist and dry beech wood samples along and across fiber direction

decreases with temperature from room temperature to 200 °C. Moist wood with a MC of 10.5 ± 0.5 % shows a somewhat lower thermal diffusivity at room temperature than that of the dry beech wood. Furthermore, it is clearly indicated that thermal diffusivity parallel to the fiber grain is approx. two to three times higher than across fiber direction. The expanded uncertainty for the thermal diffusivity of wood in the moist and dry state is mainly determined by the repeatability between individual specimens and the influence of the layer system. In longitudinal direction an expanded uncertainty of ± 10 % in the dry state (from room temperature to 200 °C) and ± 11 % in the moist state (room temperature) is estimated; ± 11 % in the transversal direction in the moist state at room temperature and up to ± 13 % in the dry state from room temperature to 200 °C.

Thermal diffusivity of the charred beech wood samples is shown in Fig. 13. Again, thermal diffusivity along the fiber direction is approx. two times higher than that across the fiber direction. The expanded measurement uncertainty for the thermal diffusivity in the charred state is estimated to be up to ± 7 % in longitudinal direction; up to ± 15 % in transversal direction and is mainly determined by the repeatability between individual specimens.

Thermal conductivity λ in longitudinal and transversal direction in the dry and charred state was calculated from the results derived, according to following equations:

$$\lambda_{longitudinal}(T) = \rho(T)c_p(T)a_{0,longitudinal}(T)\left(1 + \frac{\Delta l_{longitudinal,dry,charred}(T)}{l_0}\right)^2 \quad (6)$$

$$\lambda_{transversal}(T) = \rho(T)c_p(T)a_{0,transversal}(T)\left(1 + \frac{\Delta l_{transversal,dry,charred}(T)}{l_0}\right)^2$$
(7)



Fig. 13 Thermal diffusivity of the charred beech wood samples along and across fiber direction

In the case of the moist beech wood at room temperature, thermal conductivity was also calculated from thermal diffusivity, density and specific heat of the moist wood. However, in the case of specific heat $c_{p,moist}$, a following mixture rule for dry wood (90.5 wt-%) and water (9.5 wt-%) was applied:

$$c_{p,moist} = c_{p,dry} \frac{\text{wt} - \%_{dry}}{100} + c_{p,water} \cdot \frac{\text{wt} - \%_{water}}{100},$$
(8)

where $c_{p,water}$ is the specific heat of water (4183 J·kg⁻¹·K⁻¹ [21]), and $c_{p,dry}$ is the specific heat of dry wood at room temperature. As indicated in Fig. 14, thermal conductivity of dry beech wood increases with temperature. A wood MC of 10.5 ± 0.5 % (9.5 wt-%) increases the thermal conductivity in the range of approx. 20 %. The uncertainty for thermal conductivity is estimated to be up to ± 11 % for wood in longitudinal direction in the dry state in the temperature range up to 200 °C; up to ± 13 % in transversal direction. No uncertainty estimation is given for wood in the moist state, as thermal conductivity is calculated from specific heat of moist wood, using a mixture rule for dry wood and water.

Thermal conductivity of charred material (Fig. 15) is higher than that of the wooden material in the temperature range between 100 °C and 200 °C and increases with temperature. In the charred state the expanded measurement uncertainty for thermal conductivity is estimated to be up to ± 10 % in longitudinal direction from 200 °C to 800 °C; up to ± 17 % in transversal direction that is mainly influenced by the repeatability between individual specimens measuring thermal diffusivity.



Fig. 14 Thermal conductivity of the moist and dry beech wood samples along and across fiber direction



Fig. 15 Thermal conductivity of the charred beech wood samples along and across fiber direction. Thermal conductivity at 100 °C and 900 °C was calculated from the extrapolated specific heat data

4 Discussion

The results of thermogravimetric analysis are in good agreement with the work of other authors, Poletto [22], Liu *et al.* [23], Zang *et al.* [24], Rath [25], and Rinta-Paavola [26], revealing the characteristic stopping point in the first-order derivative of mass loss of beech wood. This is related to the decomposition of hemicelluloses and cellulose, as the cellulose of hardwood tends to decompose faster than that of softwood [22, 27]. Care must be taken with regard to the initial MC, as the final mass loss is strongly depending on the initial wood MC. Furthermore, the measurement procedure influences the wood MC, as evaporation of the furnace chamber reduces the MC to a few percent. In this work a two-step procedure was applied to overcome this task.

The thermal contraction for beech wood across fiber direction in this work was found to be greater than in fiber direction by the order of a magnitude. The same case was found for dry beech wood. A thermal expansion, significantly greater across fiber direction than in fiber direction is also reported by Kubler *et al.* [28], Espinoza-Herrera *et al.* [29] and the works reviewed by Goli *et al.* [30] for different wood species.

The resulting density variation from room temperature to 200 °C of dry beech wood is lower than 2 % and shows an almost linear behavior. Starting from moist wood density, a characteristic shoulder close to 60 °C is found. This is assumed to be an effect of the expansion of water prior to evaporation. A different behavior is found for charred beech wood. Thermal expansion is rather independent on fiber direction. Furthermore, thermal expansion in both directions is small compared to thermal expansion of dry wood across fiber direction, even at high temperatures of several hundred degree Celsius. This is assumed to be due to the decomposition of

the wooden material. The charring of the wooden material results in a degradation of the cell structure, a distortion of the tracheid morphology and a disappearance of intercellular layers [31].

Specific heat of beech wood in the dry state was found to be practically linear from room temperature to 200 °C. A slight deviation from the least squares fit was found in the temperature range from 160 °C to 190 °C. This might be an effect due to residual moisture adsorbed to the wood cell walls, as the first measurement run was performed up to 160 °C only. As stated by Simpson and TenWolde [8], specific heat is depending on temperature and the wood moisture but shows no significant difference between various wood species. This is confirmed by the work of Koch [32]. A linear relationship with temperature of pine wood specific heat in the dry state results in approx. 1.19 kJ·kg⁻¹· K^{-1} at room temperature. A linear increase of 21 biomass types in the temperature range from 40 °C to 80 °C was also found by Dupont et al. [33] (Calvet calorimeter, type C80). Czajkowski et al. [34] report specific heat of beech wood, determined by a water calorimeter (initial temperature of the samples: 90 °C, equilibrium temperature of the calorimetric system: approx. 20 °C). A mean specific heat of 1.38 kJ·kg⁻¹·K⁻¹ is reported, that is approx. 5 % higher than the mean found in this work (approx. 1.31 kJ·kg⁻¹·K⁻¹). Potential residual moisture of kiln-dried samples may have influenced measurement results.

The specific heat of charred beech wood can be described by a polynomial function, that was also observed by Gupta [35] investigating a mixture of 3 softwood chars. Furthermore, the results of this work are in close agreement with the data of graphite [19]. This indicates that charcoal consists mainly of carbon.

The apparent specific heat of beech wood in the temperature range of pyrolysis from 200 °C to approx. 500 °C shows a sequence of only exothermic reactions. This is in agreement with the detailed analysis of heat of pyrolysis of beech wood of Rath *et al.* [25], but contrary to the behavior of spruce as a softwood species [20, 25], showing both, endothermic and exothermic reactions. As the thermal degradation of wood is determined by the decomposition of the three main components, the proportion of the components, hemicelluloses, cellulose and lignin influences the behavior of wood species during pyrolysis. The proportion of the three components differs among wood species; hardwood species have higher content of hemicelluloses and less lignin [27]. This contradictory result between different wood species is substantiated by the reported heat of pyrolysis that ranges from strong exothermic values to strong endothermic values, as reviewed by Rath et al. [25] and Sinha et al. [36]. As reported in the review of Bartlett et al. [37], pyrolysis of wood is a complex process, as the chemical processes occurring are numerous. The degradation of the wood main components results in the formation of inert and combustible gases, tars, organic acids, ketones, esters and char [36, 37]. With subsequent heating, pyrolysis products may undergo further pyrolysis themselves. According to Kubler [38], it is generally believed that pyrolysis is endothermic up to 280 $^{\circ}$ C. At higher temperatures the process may become exothermic and can cause self-heating. That is assumed to be highly dependent on the residence time of pyrolysis gases in the solid matrix [36], as this leads to secondary pyrolysis reactions. These reactions are believed to be exothermic, affecting the overall heat of wood pyrolysis. As a result, the values of heat of wood pyrolysis do not only vary with wood species, but are strongly dependent on the measurement procedure, as shown by Rath *et al.* [25]. Initial sample weight, sample dimensions, heating rate, crucible size (and lid) and gas flow rate may influence the flow of primary volatiles from the vicinity of the sample. Furthermore, wood fiber orientation [36] of the sample may influence the measurement results, as heating across fiber direction results in a higher resistance of the flow of the volatiles from the sample towards the bulk gas in contrast to the flow in fiber direction. In this work, the samples were cut across fiber direction, resulting in the heat flow parallel to the wood fiber. However, a lid was used that is assumed to be the major influence in affecting the flow of the volatiles from the vicinity of the sample towards the bulk gas. This is confirmed by the work of Rath *et al.* [25] showing a strong variation of the overall heat of wood pyrolysis, whether a lid is used or not.

In contrast to room temperature measurements of thermal conductivity, reported measurements at high temperature are sparse. In particular, the laser flash method to determine thermal diffusivity at higher temperatures is hardly mentioned; Harada *et al.* [39] used the laser flash apparatus to investigate 13 Japanese softwood and hardwood species from room temperature to approx. 250 °C in air and under vacuum, highlighting the effect of the atmosphere on measurement results of porous materials. In this work, the measurements on beech wood were performed in ambient air that is assumed to come closest to the conditions of wood in fire. In the case of charred beech wood however, argon was used as purging gas to protect the charred samples from combustion at elevated temperatures. As thermal conductivity of argon is different to air atmosphere, the measurement results are supposed to be slightly influenced compared to the behavior of charred material in fire. However, as pyrolyzing gas (inert gases, combustible gases) influences gas atmosphere under real fire conditions, the composition of the surrounding atmosphere is very complex and not well defined.

At room temperature a thermal conductivity of approx. 0.13 W·m⁻¹·K⁻¹ across fiber direction and 0.35 W·m⁻¹·K⁻¹ in fiber direction was found for beech wood with an oven-dry density of approx. 710 kg·m⁻³ in this work. Sonderegger et al. [40] investigated European beech wood using a guarded hot plate apparatus (oven-dry density: approx. 673 kg·m⁻³). In the dry state, approx. 0.34 W·m⁻¹·K⁻¹ is reported in fiber direction, approx. 0.10 $W \cdot m^{-1} \cdot K^{-1}$ in tangential direction and 0.14 W·m⁻¹·K⁻¹ in radial direction. Assuming an average thermal conductivity of approx. 0.12 W·m⁻¹·K⁻¹ in transversal direction, the measurement results are in very good agreement to the findings of this work, considering the effect of density on thermal conductivity. Vay et al. [2] investigated beech wood in the moist state (MC = approx. 11 %) using a guarded hot plate apparatus (average density: approx. 744 kg·m⁻³). Approx. 0.42 W·m⁻¹·K⁻¹ is reported in fiber direction, approx. 0.14 $W \cdot m^{-1} \cdot K^{-1}$ in tangential direction and 0.17 $W \cdot m^{-1} \cdot K^{-1}$ in radial direction. In this work, 0.42 W·m⁻¹·K⁻¹ and 0.16 W·m⁻¹·K⁻¹ are found in longitudinal and transversal direction for beech wood with an average density of approx. 730 kg·m⁻³ in the moist state. Dried European beech wood was also investigated by Czajkowski et al. [34] by heat transfer experiments and inverse finite element analysis to evaluate thermal conductivity. Significantly higher values were found along fiber direction compared to this work. Additionally, a significant difference between radial and

tangential direction is reported. This is in contrast to the results of this work and to the work of Sonderegger *et al.* [40] and Vay *et al.* [2]. However, contradictory results point out the challenges in measuring wood species. Nevertheless, the good agreement with the results of Sonderegger *et al.* [40] and Vay *et al.* [2] indicates that the laser flash apparatus seems to be an appropriate method to determine thermal diffusivity and furthermore thermal conductivity in different directions and in different states. The advantage of the laser flash apparatus is that measurements can be performed at temperatures up to several hundred degree Celsius and at relatively short time scales. The disadvantages are, that measurements on multiple samples are required, as measurements are performed rather localized, and that the samples have to be coated in an appropriate way to protect the specimens from the laser pulse. However, the aforementioned comparison to literature results [2, 40] proves that the procedure given in this work is highly appropriate.

In contrast to room temperature data of thermal conductivity of wooden materials, charred material is less frequently investigated. Data found at elevated temperatures of several hundred degree Celsius have to be taken with care, as thermal conductivity is either extrapolated from room temperature data [41] or determined by calibration to the results of fire tests [42]. Hankalin et al. [43] report thermal conductivity of pine and birch char in different directions, ranging from approx. 0.08 W·m⁻¹·K⁻¹ to 0.13 W·m⁻¹·K⁻¹ with char densities ranging from approx. 150 kg·m⁻³ to 350 kg·m⁻³. Da Silva al. [44] report thermal conductivity of eucalyptus wood charcoal, charred in a brick kiln, in the range of approx. 0.03 W·m⁻¹·K⁻¹ at a charcoal density of approx. 350 kg·m⁻³. Luke Williams *et al.* [45] report an average thermal conductivity in fiber direction of approx. 0.22 W·m⁻¹·K⁻¹ and 0.05 $W \cdot m^{-1} \cdot K^{-1}$ across fiber direction for 6 softwood species, that have been pyrolyzed at 453 °C for 8 h. The densities were in the range of approx. 225 kg·m⁻³. These values are in contrast to the results obtained in this work, with higher values of thermal conductivity in the charred state in the temperature range of 100 °C to 200 °C than of virgin material in the same temperature range.

As stated by da Silva et al. [44], the thermal conductivity of charcoal is depending on the structural changes during pyrolysis. Crack formation, pore size distribution may significantly influence the measurement results. In the case of small sample sizes, distortion due to the grain structure and resulting crack formation is limited. In this work rather small samples were prepared that do not show any macrocracks, that is also confirmed by the relatively high density of the charred samples. Furthermore, the work of Brown [46] shows results of measurements of pine wood, performed with an individual measurement technique on samples during the heating cycle, potentially without crack formation. An increase of thermal conductivity is shown from virgin material to charred material, that is similar to the results of this work. This highlights the influence of sample preparation, measurement method and the resulting crack formation, when it comes to charred material. In this work, a potential microcrack formation in the inner section of the samples can be assumed from the scattering between individual measurements, found for thermal diffusivity of charred material across fiber direction. This is strongly influenced by the fiber structure, resulting in a relatively high estimated measurement uncertainty of thermal conductivity. Furthermore, it is of interest that thermal conductivity in fiber direction remains higher than across fiber direction, despite the degradation of the cell structure. However, the difference is lower in comparison to wooden material that is also reported by Hankalin *et al.* [43].

5 Conclusion

In this study, mass loss, thermal expansion, specific heat and thermal diffusivity of moist, dry and charred beech wood were measured in a wide temperature range, considering the anisotropic behavior of wooden material. The following conclusions can be drawn from the present work:

- Mass loss of moist beech wood shows a series of reactions, from evaporation of wood moisture to the decomposition of the wooden cell structure. The two-step procedure applied in measuring mass loss of beech wood with a STA enables both, the consideration of the release of wood moisture and the wood pyrolysis under inert atmosphere.
- Density of beech wood was derived from room temperature density and measurement of thermal expansion in different directions. It was found that thermal expansion is higher across fiber direction than along fiber direction in the order of a magnitude. Furthermore, it was found that temperature dependent density of moist wood can be determined by combining the measurements of thermal contraction using a push-rod dilatometer and of mass loss using a STA. The measurement results at elevated temperatures are in very good agreement to the results of temperature dependent density of dry wood derived from room temperature density and measurement of thermal expansion in both directions.
- In contrast to wooden material, thermal expansion of charred material is rather independent on fiber direction due to the supposed degradation of the wooden cell structure. Furthermore, it was found that thermal expansion is small, even at a temperature of 900 °C, resulting in an almost negligible temperature dependency of charred beech wood density.
- Specific heat of beech wood shows an almost linear dependence on temperature from room temperature to 200 °C. Slight deviations from the linear relation at elevated temperature might indicate potential residual moisture adsorbed at the cell wall even at temperatures far beyond 100 °C. Specific heat of charred beech wood is very close to that of graphite [19], indicating that charred wooden material consists mainly of carbon.
- The investigation of specific heat of beech wood in the temperature range of pyrolysis shows the contrary behavior of different wood species when it comes to pyrolysis reaction. Beech wood as a hardwood species shows results that are in clear contrast to spruce wood as a softwood species [20, 25], with no endothermic reactions noticeable.
- The laser flash method was found to be a highly suitable method to determine thermal diffusivity of both, wooden material and charred material, considering the porous character of charred wooden material and the anisotropic behavior of wood in general. The only significant disadvantage of the laser flash method is

the necessity of the coating of the samples that is required to protect the wooden material from the heat of the laser pulse. However, the measurement procedure used for this work is confirmed by an excellent agreement of the results of measurements performed on reference materials and of thermal conductivity of beech wood reported by other authors [2, 40].

• It was found that thermal conductivity of beech wood in longitudinal is approx. 2 to 3 times higher than in transversal direction. The same applies to charred beech wood. However, the relative difference is smaller due to a supposed defragmentation of the wooden cell structure. However, thermal conductivity reported in this work for charred material differs significantly form the work of other authors [44, 45], showing only an agreement with the work of Brown [46]. It is supposed that sample size and sample preparation significantly influence the porous character and potential crack formation of the charred material. This is also supported by the relatively high expanded measurement uncertainty estimated for the thermal conductivity of charred beech wood, which is mainly influenced by the scattering of the individual samples.

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Data Availability No datasets were generated or analysed during the current study.

Declarations

Competing interests The authors declare no competing interests.

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