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CREEP PROPERTIES OF HEAT TREATED WOOD IN RADIAL DIRECTION

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Abstract.
This paper discusses the ageing-like process of heating wood in completely dry conditions and the effects on creep properties for poplar wood (Populus alba). Creep tests have been performed in the radial direction, for heat treated and untreated samples over 10 hours. Beforehand, some samples were heat treated at 150°C during a few days. Differences in creep phenomenon between new and aged samples are discussed with the help of graphical methods such as the approximated complex plane.

Key words: heat treatment, poplar, creep tests, radial direction.

1. Introduction
Recently, the author and coworkers have been interested in the mechanical behavior of wood panel paintings [1,2]. Collaborations were established to perform and analyze the monitoring of dimensional changes on 3 to 6 centuries old wooden boards. A large amount of data on long term period were obtained, which raised also many questions on the properties of ancient wood. Because of the real difficulty of analyzing the material of panel paintings, we are now facing the lack of data to understand the behavior of ancient wood.

It has already been shown that ancient wood exhibits different mechanical properties than fresh wood [3]. Partly due to the variation in chemical composition, several properties like stiffness, axial strength, hardness and dimensional stability slightly improve within a few centuries, while color is getting darker. In order to reproduce partly the effects of time, heat treatment was used in many studies as an ‘accelerated ageing’ and showed so far good results on different softwood with respect to the darker color, fragile behavior and the improved stability against humidity changes [4,10]. In the cultural heritage context, this technique was used for instance in the restoration of wooden statues [5,6].

Further applications in cultural heritage, possibly in structures, would imply to get interested more about the mechanical behavior of heat treated wood. It has already been shown that heat treatment in dry conditions tends to reduce the elastic modulus in longitudinal direction [7], while the ancient wood get naturally stronger, probably due to the crystallization of cellulose in the earlier centuries [8]. But so far very few attempts have been made to show the long term behavior or water-related mechanical behavior of artificially heat treated wood.

This study was conducted on hardwood for several reasons. So far most of the studies were conducted on softwood. Moreover, European poplar is one of the most common species for panel paintings and thus was chosen for our experiments. The creep experiments were all conducted in radial direction; so far, most of the literature on the subject was done in the longitudinal direction.

2. Material and method
This study was conducted on poplar wood (Populus alba) from Italy. Creep tests were all performed on radial samples, with dimensions 10mm (T), 1.6mm (L) and 60mm (R); for practical reasons, adsorption isotherms were checked by weighting the same size of samples. All samples were first air-dried for 24 hours at 60°C, and then vacuum-dried over P2O5 at the same temperature and duration to reach the oven dry state.

Heat treatment was performed on dried samples at 150°C. Two different durations (24 hours and 48 hours) were tested on two different groups of samples. Durations were decided in accordance with previous results on Hinoki (Chamaecyparis obtusa) [4], in order to obtain significant decrease in hygroscopicity.
Static bending tests were performed until rupture on 5 samples at least for each group (non-treated, treated for 24h and 48h) at 65% relative humidity.

In order to obtain both isotherm curves and creep at different moisture contents, samples were conditioned in 9 different relative humidity (RH%) at 24°C, obtained with saturated solutions, at 25°C for nearly 11 days. Adsorption isotherms were measured at each humidity conditions while creep tests were only performed at 22, 62, 84, 92 and 97% relative humidity, for both treated and non-treated samples.

Constant humidity during creep tests was obtained by wrapping samples in polyethylene bags. Room temperature regulation was considered to be satisfactory for this study. Moisture stability of the wrapped samples was checked by weighting before covering and after experiments, with no significant deviation.

Creep tests were performed on a three points bending apparatus. Constant load was applied, and maximum strain applied to the material was estimated to be 0.20%, most of the samples being deformed at around 0.15%.

Each sample was at least loaded for 10 hours, and data points were collected at a 0.1 interval, given in natural logarithm of the time in second. To go through the variability, all data were interpreted in terms of compliance.

3. Results and discussion

As expected, hygroscopicity of poplar wood was reduced by heat treatment. Static bending test showed that elastic modulus at 65% is also decreasing with the treatment duration. Hygroscopicity of poplar treated during 24 and 48 hours were almost the same. If sufficient time was dedicated to this study, it would have been interesting to investigate regularly spaced durations on the log(time) axes. Fig 1a and Fig 1b show the adsorption isotherms with experimental data and fitted equation as used by Hailwood and Horrobin [11]:

$$A + B \cdot rh + C \cdot rh^2 = \frac{rh}{mc}$$

Fig. 1: adsorption isotherms plotted in (a) relative humidity divided by moisture content versus relative humidity, and (b) relative humidity versus moisture content. ● non-treated samples ▲ treated samples.

In this paper, two different approaches are proposed to analyze creep data: first the material properties are described as functions of ln(time sec ) and moisture content, and the second approach will be to plot data in the approximated complex plane to find a suitable rheological model. All results are presented in terms of creep compliance in order to avoid dispersions in the samples properties.

3.1. Influence of moisture content on elastic properties

Fig. 2 shows the relative variations of elastic modulus, estimated during creep tests at t=5sec, depending on the moisture content. For treated and non-treated samples, all data are divided by modulus at 64%. Radial modulus (Er) at 64% (Er64%) in static bending was around 1.1GPa for non-treated wood and 0.97GPa for treated wood. Values found in creep tests were close to these values. Non-treated wood shows clear linear dependency between ER and mc%, and strong reduction with increasing humidity.
In former studies [4], static bending tests have suggested that heat treatment increases the elastic compliance at standard ambient conditions. For practical applications, it would be useful to know if the same effect can be expected for creep compliance, as well as for higher levels of relative humidity.

3.2. Analysis in terms of compliance

The compliance at t=10 seconds and at t=9 hours are both plotted on Fig. a, as a function of moisture content. Instantaneous compliance ($J_{10sec}$) shows a regular increasing with moisture content (see dotted line on Fig. 3a) while the curves have particular shape depending on treatment. The difference between the two curves ($J_{9hours} - J_{10sec}$) i.e. the contribution of delayed behavior of wood, is called creep compliance and is plotted in Fig. 3b. This curve points out the differences in creep phenomenon between treated and non-treated samples.

After heat treatment, if the dependency between creep compliance and moisture content is the same, the two curves of Fig. 3b might be superimposed. In other words, if heat treatment results only in a reduction of moisture content, experimental points representing heat treated wood should be on the untreated samples curve, with only a shift due to the reduced moisture content.

However it is not the case: the delayed deflection of heat treated samples is sometimes higher, sometimes lower, depending on moisture content. This observation supposes that the influence of heat treatment should not be considered systematic, that the effect might depend on moisture content during loading.

The difference in shape of the two curves also implies to explain what happen at 6% moisture content in the new wood, where a clear minimum is shown in creep compliance (see Fig. 3). The same effect does not occur in heat treated wood.

The differences in creep behavior can also be shown by plotting the time spectrum of treated and untreated samples, as shown on Fig. 4. Time spectrum consists in plotting characteristic time $\tau$ versus moisture content, where $\tau$ is the time to reach a given creep rate in the compliance curve. The faster the deformation increases, the lower the characteristic time $\tau$. Thus the curves in Fig. 4 show that modification of delayed behavior of heat treated wood depends on moisture content.

The previous observations suppose that the chemical composition of wood changed, and that the components that were modified play an important role around 6% moisture content (this is shown by the reduction of minimum pick after treatment).
Fig. 3: (a) compliance versus moisture content, (plain line) after 9 hours, (dotted line) instantaneous, (b) creep compliance after 9 hours. ● non-treated samples ▲ treated samples.

Fig. 4: time response spectrum, ln(τ in seconds) versus moisture content: τ is the time for which the rate of creep compliance is equal to a given value (dJ/dln(t)=10^{-4}, J given in MPa and t in seconds).

3.3. Analysis by approximate complex plane (ACP)

Based on an approximation by Alfrey (1948) of the complex compliance usually considered for dynamical studies, we can interpret our data in a different way. With this method, on X axis we plot the creep compliance as calculated from the data, and Y axis is proportional to the compliance rate:

\[ X = J(\ln t) \quad Y = \frac{\pi}{2} \cdot \frac{dJ}{d\ln t} \]

On the graphic method proposed by Huet [9] we identify a rheological model that can fit to our data. First step is to determine the type of plot in the approximate complex plane. On the basis of pattern in Fig. 6, we assume that the phenomenon is described by a linear curve in the ACP, this for all moisture content and both treated and untreated wood. A slope \(< 1\), like observed here, means that the suitable model would have a large spectrum of Kelvin models.

Then we analyze and discuss the effect of moisture content on the parameters of the linear regression used as model. Fig. 7 gives the slope of the linear regression and the intersection with X axis, respectively the spreading of Kelvin spectrum and the elastic compliance given by the model fitting. Experimental measurements of instantaneous compliance are also re-plotted in dotted line to show the difference between fitted model and initial data.
Fig. 5: (a) Kelvin body, (b) representation of a single Kelvin body in the ACP, (c) spectrum of several Kelvin bodies in the ACP.

Fig. 6: an example of creep curves in complex plane for non-treated wood.

Fig. 7: parameters given by model in complex plane: (a) slope of model (b) instantaneous compliance as represented by model; dotted line correspond to measured compliance at 10 seconds, as shown in Fig. 3.

4. Conclusion

New data have been obtained on a hardwood (poplar) and in radial direction; while studies conducted so far and available in the literature concern usually softwood in longitudinal direction. Mechanical behavior in transverse plane (R,T) is of great interest, from the panel paintings to the structural applications.

As expected, hygroscopicity of poplar wood was reduced by heat treatment at 150°C for 1 day. Results suggest that the effect of heat treatment on creep deflection is depending on moisture content under service conditions. The discussion has to be detailed on several points: the shape of creep curve on natural wood, with appearance of a minimum around six percent moisture content, and the modification of this shape due to heat treatment.

With further experiments, it would be interesting to compare these results with other directions (longitudinal and tangential). It would be also interesting to draw experiments with a large range of treatment times and temperatures.

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