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Comparison of thermal expansion of wood and epoxy adhesives

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Epoxy-based adhesives are used both in the consolidation of decayed timbers and for new structural joints. Investigating the compatibility of wood and epoxy adhesives provides a better knowledge of the long-term behaviour of wood-epoxy joints. Besides mechanical compatibility, also temperature-related parameters are relevant in this context. The values of the thermal expansion coefficients (TEC) of two wood species (Spruce, *Picea abies* and Iroko, *Clorophora excelsa*) and four different epoxy resins have been compared using a modified test method that allows for nearly iso-hygric conditions of the wood specimens. Minor differences in TEC have been observed between wood in the transversal-to-the-grain direction and an experimental epoxy adhesive, which is considered highly compatible with wood on the basis of mechanical and ageing tests. Other commercial epoxy adhesives show greater differences in terms of TEC and a proportionally decreasing mechanical compatibility. According to these results, the evaluation of thermal properties can be considered very useful for a modelling approach to predict the long term behaviour of wood-epoxy adhesive interface.

Vergleich der thermischen Expansion von Holz und Epoxy-Harzen

Kleber auf Epoxy-Basis werden sowohl zum Festigen von geschädigtem oder abgebauten Schnittholz als auch für neue Holzkonstriktionen benutzt. Die Untersuchung der Verträglichkeit von Holz und Epoxyharzen führt zu einem besseren Verständnis des Langzeitverhaltens von Klebeverbindungen. Neben der mechanischen Kompatibilität sind dabei auch die von der Temperatur abhängigen Parameter von Bedeutung. Die Werte der thermischen Expansionskoeffizienten (TEC) von zwei Holzarten (Fichte, *Picea abies* und Iroko, *Clorophora excelsa*) wurden verglichen mit denen von vier Epoxyharzen. Die verwendete Prüfmethode ermöglicht nahezu iso-hygrische Bedingungen der Holzarten. Geringe Unterschiede ergaben sich zwischen der Werten für Holz, quer zur Faser gemessen, und einem experimentellen Harz, das aufgrund von mechanischen und Alterungstests als hochkompatibel mit Holz gilt. Andere kommerzielle Harze zeigten größere Unterschiede in den TOC-Werten und eine dementsprechend geringere Kompatibilität. Aufgrund der Ergebnisse erweist sich die Abschätzung der thermischen Eigenschaften als hilfreich für eine Modellierung des Langzeitverhaltens der Holz-Kleber-Verbindung.

1 Introduction

Epoxy resins are becoming increasingly used in the formulation of structural wood adhesives for the repair of old timber structures (Avent 1992; Mettem et al. 1995), of glued-laminated timber (Radovic and Goth 1992) and for new and highly-efficient framing solutions (Bengtsson et al. 2000). Nevertheless, the problem of compatibility between the two materials is still debated, and in some cases it hinders the acceptance of epoxy-based wood adhesives. Interface compatibility influences the durability of the bond and has therefore an important impact on safety.

This work is part of a research program, focused on the behaviour of wood-epoxy interface with the aim of providing quantitative data for defining the compatibility between such materials, modelling the durability of the wood-epoxy joints, and designing adhesives which are more compatible with wood than the currently available products. Mechanical aspects on the durability of the interface have been already analysed (Lavisci et al. 2001), but in the application field, as well as in experimental work, temperature is a crucial factor on which materials have to be evaluated. In order to focus on the thermal aspect of the problem, the thermal expansion coefficient (TEC) of the materials involved is considered in this work. Comparing the values of thermal expansion coefficients as critical parameters for evaluating the bond between two materials is common practice. Stumes (1975) applied this approach in the case of epoxy resins and steel used to repair ancient timbers. Measuring the TEC of synthetic polymeric materials is a standardised practice, but evaluating such coefficient for wood is not so simple. In order to take into account for moisture changes Hendershot (1924) suggests corrections of measured values, but this does not provide a direct and reliable measurement. Different methods have been proposed to prevent humidity variations of samples during the measurement of the wood TEC: Villari (1868)

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soaked samples in oil, Kubler (1959) and Schirp and Kubler (1968) wrapped specimens in aluminium foils and again Kubler et al. (1973) enclosed samples in brass, vapour-tight, tubes. All these configurations do not reproduce well the *in-situ* conditions. A different approach is to test water-saturated specimens (Salmén 1990) or ovendry specimens (Stevens 1960) or to work at temperatures at which the samples are moisture-free (Fusako 1973; Popper and Eberle 1992), but this clearly limits the field of application of the results.

The TEC of wood in the transversal-to-grain direction is of the same order of magnitude as that of metals (10^{-5} $°C^{-1}$), even if in most applications the effect of shrinkage related to wood drying overshadows the effect of the expansion due to the increase of temperature. However, several circumstances exist in which the TEC of wood may become important: for example, deep inside structural timber with large cross-sections, where the moisture content cannot quickly follow the changes of the environmental relative humidity. In such cases, the time for reaching equilibrium moisture content is long and hence the stresses developed during the transitory state become important.

Preliminary tests were performed on wood samples where the natural shrinkage splits had been filled with epoxy resins. After an ageing treatment especially designed to maximise the thermal stresses at the interface, they showed that after repeated thermal cycles in nearly iso-hygric conditions a fracture is formed at the interface. This fracture propagates by continuing cycles, thus suggesting that the stresses induced by a thermal expansion gradient are large enough to cause delamination.

Thermal aspects of the interfacial compatibility of wood and epoxy adhesives are therefore analysed in this work, through the comparison of the TECs of four different epoxy products and two wood species. The comparison is based on a test method modified to measure the TEC of wood in nearly iso-hygric conditions, similarly to what happens *in-situ* within relatively large beams, where wood is within the hygroscopical range and exchanges moisture and heat very slowly with the surrounding air.

2
Materials and methods

Three commercial epoxy resins were used which were specifically sold for wood restoration, and an experimental one, all of them two-component and usually applied at room temperature. Further, two different wood species (Spruce, *Picea abies*, and Iroko, *Clorophora excelsa*) were selected. All the resins were mixed using the stoichiometric ratio indicated by the producers and the specimens were cured for 30 days in standard atmosphere (20 °C and 65% relative humidity) before measurements. This procedure was suggested by some preliminary kinetic tests.

The TEC was measured according to the Italian standard (UNI 1967) for plastic materials with a dilatometer (Ceast, Italy) consisting of two quartz tubes with different diameters and a micrometric transducer which measures the elongations, the specimen being positioned between the two tubes. According to the procedure, measurements are repeated for different temperatures and each time the specimens is returned to a cold reference bath to check the starting position. The standard was strictly followed for the epoxy resins, but for wood specimens the apparatus and the procedure were modified until they became more accurate and reliable (Pizzo 1999).

In the modified apparatus (Fig. 1), the dilatometric tube, with the specimen inside, is fixed and a jacket heat exchanger is moved externally. The jacket has an internal volume of 18 litres, which guarantees heat inertia. In the jacket the water laps the external wall of a vertical copper tube, located inside the jacket but not communicating with it. This inner tube is filled with still water and is open on the upper end so the quartz tubing of the dilatometer can be slowly positioned inside it. So, the still water transfers the heat whereas the specimen is shielded from rising water, which could produce vibrations or movements. The precision of the temperature control for the modified test apparatus was verified with some temperature measurements between 30 °C and 60 °C, with a step of 5 °C, using a mercury thermometer (0.1 °C precision) for checking the temperature in the bath, and a thermocouple (0.1 °C precision) located inside the still water of the jacket. A maximum error of 0.5 °C was observed.

The epoxy specimens for the TEC measurements were cylinders with a diameter of 9 mm, 37 to 40 mm in length. They were obtained by machining cured cylinders on a

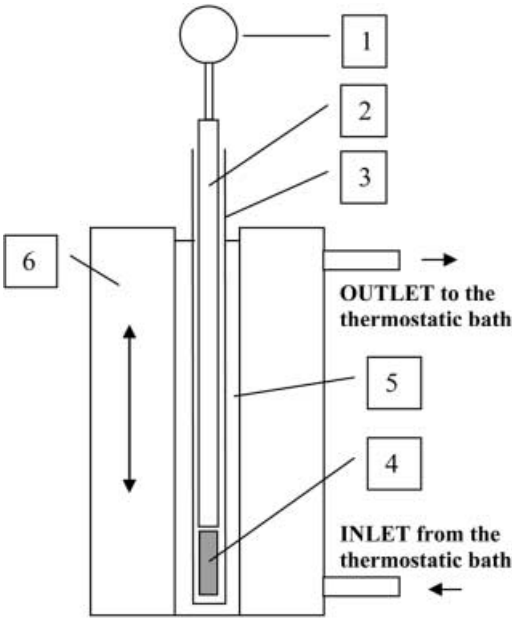

Fig. 1. Scheme of the modified dilatometer for measuring the Thermal Expansion Coefficient (TEC): 1) transducer, precision 1 μm; 2) internal quartz tube, external diameter: 1.28 cm; 3) external quartz tube, internal diameter: 1.43 cm – thickness: 0.15 cm; 4) specimen; 5) warm still water; 6) moving external jacket with circulating warm water from the thermostatic bath
Bild 1. Schema der Dehnungsmessung zum Messen des thermischen Expansionskoeffizienten (TEC): 1) Meßwertwandler mit einer Genauigkeit von 1 μm; 2) inneres Quarzrohr mit Außendurchmesser 1, 28 cm; 3) äußeres Quarzrohr mit Innendurchmesser 1,43 cm und 0,15 cm Wandstärke; 4) Probe; 5) warmes, nicht bewegtes Wasser; 6) beweglicher Mantel mit zirkulierendem thermostatisiertem Wasser

lathe. Measurements were repeated on two specimens, two times for each temperature, and in each case the dilatometer was brought back to the reference temperature of 0 °C, to check that the transducer settled again at the starting value of 0 μm. Tests not fulfilling this condition were not considered.

The wood specimens were oriented in the parallel-to-the-grain or in the transversal-to-the-grain direction. No distinction was made between radial and tangential directions. Parallelepiped specimens were cut with 8 × 8 mm cross-section and lengths as follows: 120 mm for longitudinal Spruce, 80 mm for transversal Spruce, 100 mm for longitudinal Iroko, 51 mm for transversal Iroko. In order to investigate the weight loss of wood during the test, Spruce wood specimens were heated within a thermo-balance (precision 1 mg) in the same configuration used in the TEC tests. Three types of characterisation have been carried out, as shown in Fig. 2.

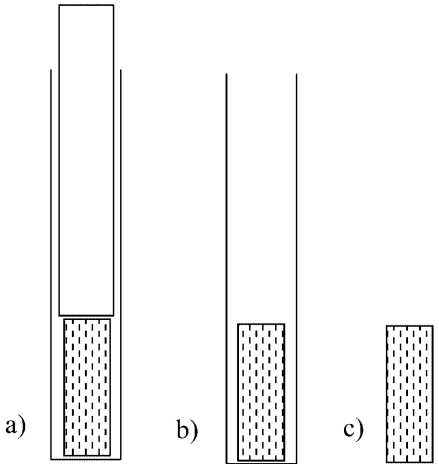

Fig. 2a–c. Representation of the "weight loss" test on probe spruce wood specimens (dotted rectangles): **a**) specimens placed into the bottom of a glass cylinder with a glass tube just over the upper surface of the specimen. This configuration reproduces very well that of the quartz tubing of the dilatometer. In this case, the weight of all the specimens has been measured just before and just after the exposure at 50 °C for two time intervals of 10 minutes and 15 minutes; **b**) specimens placed into the bottom of a glass cylinder without any other tubing inside, on the upper surface. Weight loss has been continuously measured during the exposure at 50 °C; **c**) wood probes as such, exposed for 10 minutes at 50 °C with the weight loss continuously measured. All wood specimens had an initial moisture content of 14.8% (±0.2%)
Bild 2a–c. Schema des "Gewichtsverlust"-Tests an einer Fichtenprobe (punktiertes Rechteck): **a**) Probe in einem Glaszylinder mit einer Glasröhre genau über der Oberfläche der Probe. Diese Konfiguration enspricht in sehr guter Annäherung der Quarzröhrenmessung mit dem Dilatometer. Die Proben wurden einer Temperatur von 50 °C ausgesetzt und ihr Gewicht unmittelbar danach in zwei Zeitintervallen von 10 und 15 Minuten gemessen; **b**) Proben in einem Glaszylinder ohne zusätzliches Glasrohr. Die Gewichtsverluste wurden kontinuierlich während der Erwärmungsperiode bei 50 °C gemessen; **c**) die Holzprobe allein wird 10 Minuten bei 50 °C gehalten und der Gewichtsverlust kontinuierlich gemessen. Alle Holzproben hatten eine Ausgangsfeuchte von 14,8% (±0,2%)

The TEC tests were carried out as follows:

- only one measurement on each specimen at each selected temperature was performed, with the aim of avoiding any inconvenience related to the hygrometric hysteresis of wood;
- the reference temperature for all wood specimens was set at 10 °C, in order to prevent any inconvenience related to ice formation inside wood (Kubler et al. 1973). Stabilisation time at reference temperature was evaluated by regularly weighing some probe specimens kept into the same environment. These probes were also used for evaluating the initial moisture content;
- the values of maximum (instead of final) elongation reached during the tests were used for the evaluation of the thermal expansion coefficient, because of a slight shortening observed after a long duration of keeping the specimens at the test temperature, and in order to minimise the moisture exchanges of the specimens with the surroundings, based on results of preliminary tests.

Accelerated ageing and subsequent shear testing for checking bond durability were performed according to procedures described by Lavisci et al. (2001): two cycles of water soaking under air pressure of 5 bar and subsequent drying at 60 °C in a ventilated oven. Specimens were then tested in compressive shear after an additional wetting step.

3
Results and discussion

3.1
Weight loss

According to some preliminary controls of weight loss, after 10 minutes wood specimens in the thermo-balance (Fig. 2c) already lost weight in appreciable manner, showing slight difference between longitudinal and transversal specimens (Fig. 3). This difference is essentially related to the effective area of moisture exchange, which is higher for transversal specimens, because of the greater vapour permeability in the transversal direction in comparison with the longitudinal one. When wood specimens are inserted in the bottom of the glass cylinder (Fig. 2b), weight losses are appreciably reduced, both in transversal and longitudinal directions. This is certainly related to the quick increase of the relative humidity of the environment air as already observed by Hedlin (1969) in a similar case, and to the decreasing of the driving force that reduces the rate of moisture evaporation from the wood. This effect obviously increases when also the inner glass tube is inserted in the cylinder (Fig. 2a). In this latter case the change of wood moisture content in the specimens is in the range of 0.14%–0.15%. By assuming a linear variation of the dimensions form 0% up to the fiber saturation point of 30%, the observed values would give for spruce wood a reduction in length of about 1.3 μm for longitudinal specimens (referred to a 120 mm initial length) and about 17 μm for the transversal ones (referred to a 80 mm initial length) when the temperature is kept at 50 °C for 10 minutes. Correcting the measured TEC values for drying shortening would therefore be possible, although it is not desirable for the scope of this work.

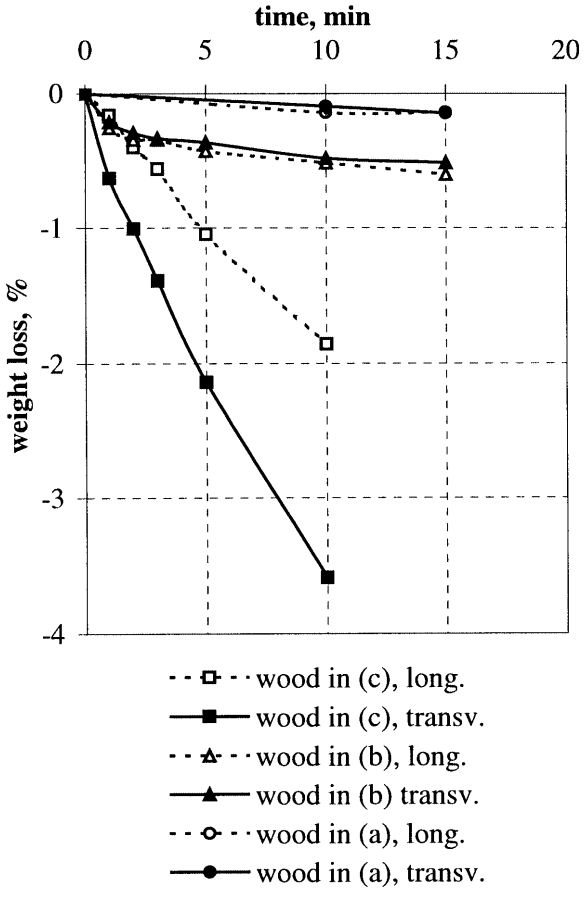

Fig. 3. Weight loss (WL) of spruce wood specimens tested according to the configurations reported in Fig. 2. The WL was calculated as follows: $WL = \frac{(P_o - P_f)}{P_o}$ where P_o and P_f are initial and final weight, measured in mg

Bild 3. Gewichtsverlust (WL) einer Fichtenprobe (Messung wie in Blid 2). In der Gleichung bedeuten P_0 und P_f das Anfangs- bzw. Endgewicht in mg

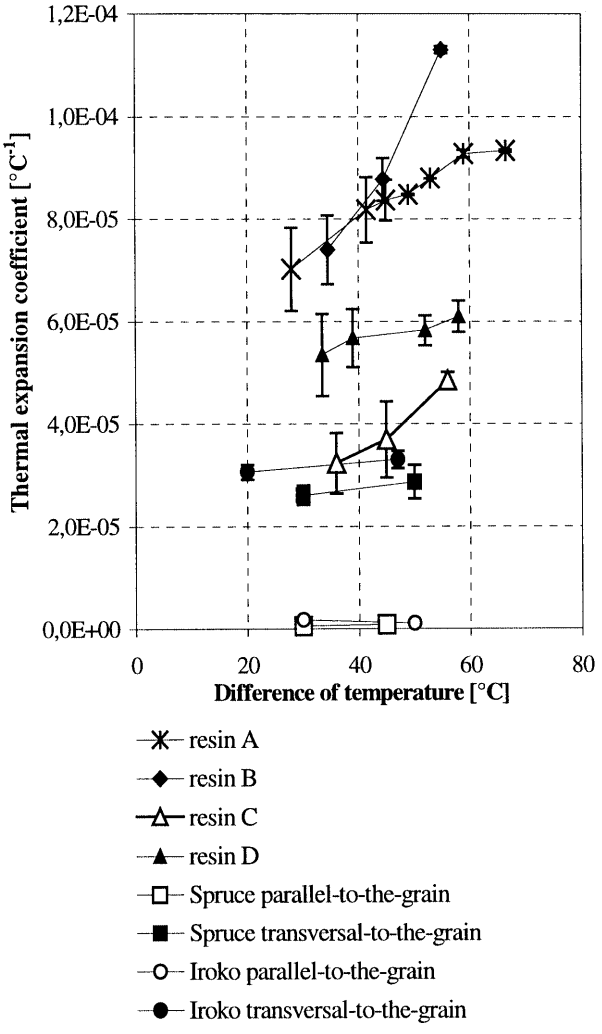

Fig. 4. Plot of the Thermal Expansion Coefficient (TEC) vs. the difference of temperature for epoxy adhesives and wood specimens. Vertical bars show standard deviations

Bild 4. Thermischer Expansionskoeffizient in Abhängigkeit vom Temperaturunterschied für Holz- und Harzproben. Standardabweichungen als vertikale Linien

Based on these results, it may be concluded that both the test procedure and the modified apparatus produce low moisture transfer rate from the specimen to the surrounding air, that is the *in-situ* condition of wood within a roof or a floor beam.

3.2
Thermal expansion coefficients

The values of TEC of both epoxy adhesives and wood species are reported in Fig. 4. In all cases a positive TEC is observed, i.e. specimens elongate by increasing temperature. Measurements on the transversal specimens were highly reproducible. Tests were carried on for about 30 minutes, though after 10 minutes the samples did not elongate any more and were dimensionally stable. In the longitudinal specimens maximum elongation was reached after less than 5 minutes, whereas a very low shortening was observed after 15 minutes.

Transversal wood specimens show TEC values of the order of magnitude of 10^{-5} °C^{-1} (as for steel), whereas for the longitudinal wood specimens values are about one order of magnitude lower (10^{-6} °C^{-1}). In regards to the transversal values, they are very close to those obtained by Popper et al. (1992) with Spruce and by Kubler et al. (1973) with other species at similar specific gravities. Instead, for longitudinal specimens some effects of the reduction of the elongation related to drying during tests were observed. It is known that longitudinal TEC for ovendry wood is not dependent on specific gravity or wood species (Wood Handbook 1999), so the differences between Spruce and Iroko observed in our case are probably due to the way by which the water interacts with the two species. The values reported for Spruce by Popper et al. (1992) in the range of 100 °C–120 °C (TEC=1.6·10^{-6} °C^{-1}) are higher than ours, also considering corrections for drying, but in that case the TEC was calculated in a dynamic manner and the values are possibly not directly comparable.

TEC values are significantly affected by the anisotropy of wood properties with respect to the longitudinal and the transversal directions. The differences observed can be

related to the much higher vibration capability of relatively weak hydrogen bonds between water and molecular chains of cellulose and between molecules of cellulose themselves that are, conversely, preferentially oriented along the longitudinal direction.

A slight increase of TEC was observed on increasing the test temperature, as already obtained in the hygroscopic moisture range by Kubler et al. (1973), even if in our case such difference is within the experimental accuracy.

The TEC values measured for the epoxy specimens are very different. In all cases tests were easy to conduct and measurements were reproducible. The average value obtained for adhesive A ($9.3 \cdot 10^{-5}$ °C^{-1} from 0 °C to 60 °C) agrees very well with that reported in the literature for a very similar product (Stumes 1975). Also adhesive B has similar TEC values but shows a higher slope of the TEC/Temperature curve, thus indicating an even higher elongation on increasing temperature. On the other hand, adhesive C has a TEC profile which is remarkably similar to that of wood specimens in transversal-to-the-grain direction, at least at the lower temperatures. Adhesive D has significantly lower TEC values than adhesives A and B, but still very different from wood specimens. The major difference between epoxy resins and wood is in the behaviour of thermal expansion versus temperature: all the resins examined increase their TEC on increasing temperature, whereas the influence of the temperature on wood's TEC is quite low for both species and directions. Since adhesives like A and B expand more easily than wood (in every anatomical direction), when temperature is rising in a joint, e.g. in epoxy-filled fissures and splits, already in the transitory state some stresses are developed at the interface, and may even increase when the wood starts to shrink. Cyclic variations of such interfacial stresses can reduce the durability of the bond between wood and adhesive.

Therefore, increasing the temperature should affect the durability of the interface between wood and resins to a different extent, depending on the different measured values of TEC. In particular major effects are expected on the durability for products like A and B, whereas only a moderate effect for product D and a negligible or slight effect for a product like C.

3.3
Bond durability

In order to verify these assumptions, accelerated ageing and subsequent shear testing have been performed on wood specimens glued with products B, C and D. Specimens were tested in compressive shear in wet conditions, according to Lavisci et al. (2001). Results shown in Fig. 5 indicate that adhesive C produces higher durability (high value of wet shear strength), whereas adhesive B is the weakest. In fact, the product C is an experimental epoxy-based adhesive which was specifically formulated to optimise the mechanical compatibility with wood. The fact that product C is the resin which shows a thermal expansion behaviour closer to wood if compared to other tested products seems to confirm the major role that TEC plays in affecting the durability of wood-resin joints. Additionally, an interesting relationship seems evident

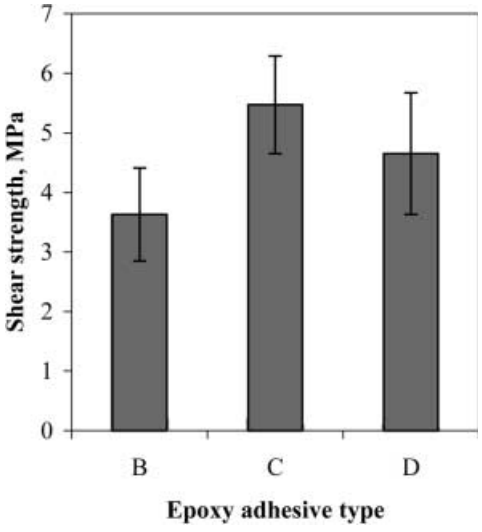

Fig. 5. Compressive shear strength of epoxy adhesives, tested wet after the accelerated ageing procedure described in Lavisci et al. (2001)
Bild 5. Druck-Scherfestigkeit von Epoxyharzen, feucht getestet nach beschleunigter Alterung nach Lavisci et al. (2001)

between TEC and the wet shear strength (Fig. 6), but further investigations are required to verify this observation.

4
Conclusions

The thermal aspects of the interfacial compatibility of wood and epoxy adhesives have been analysed by comparing the TECs of four epoxy products and two species of wood. A method has been proposed for measuring the

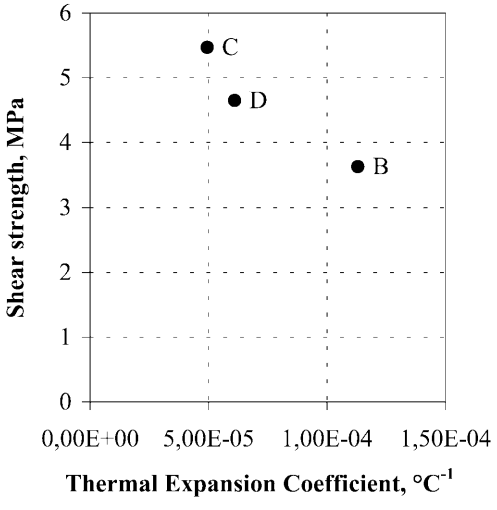

Fig. 6. Plot of the wet shear strength of the epoxy adhesives (see Fig. 5) vs. the Thermal Expansion Coefficient of the same products (adhesives B, C, D). The TEC is measured from 0 °C to 55 °C, which is very close to the temperature of the drying step in the ageing procedure (60 °C)
Bild 6. Scherfestigkeit in feuchtem Zustand für Epoxyharze (Vgl. Bild 5) in Abhängigkeit vom TEC der selben Produkte (Harze B, C, D). Der TEC wurde im Bereich zwischen 0 und 55 °C gemessen, d.h. nahe an der Temperatur des Trocknungsschrittes bei der beschleunigten Alterun

TEC of wood, keeping the sample in conditions that are quite similar to the *in-situ* conditions within timber structures. The test is easy and fast to conduct, and provides reliable and reproducible results if some precautions are taken:

a) to avoid any accidental movement of the sample, mainly during the zero-setting phase, by using a properly designed test equipment;
b) to take measurements at the maximum of the time curve and not at the stationary state, in order to minimise moisture loss of the specimens.

By comparing values for wood and epoxy resins, the following conclusions can be drawn:

1) there is a significant difference in the values of the TEC as measured in the parallel-to-grain direction of both wood species considered (Spruce and Iroko) with respect to that of the epoxy adhesives, whereas values in the transversal-to-grain direction are of the same order of magnitude;
2) the tested epoxy adhesives examined are substantially different from each other in terms of absolute TEC values;
3) only an experimental epoxy adhesive showed a TEC value quite close to wood in transversal-to-the-grain direction. It shows the best mechanical compatibility with wood in shear tests performed after an accelerated ageing procedure, whereas the other commercial adhesives with greater differences in terms of TEC have a proportionally decreasing mechanical compatibility (lower durability of the joint);
4) TEC is a useful parameter to better understand and evaluate the interfacial compatibility between wood and epoxy resins. In this perspective, modelling the behaviour of a wood-epoxy interface in order to predict its durability may benefit from taking into account the thermal properties as parameters.

The results also seem to indicate that a relationship exists between TEC and the wet shear strength of bonds after the ageing cycles, although this aspect requires further investigation.

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