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Mechanical characteristics of aged Hinoki (*Chamaecyparis obtusa* Endl.) wood from Japanese historical buildings

-Comparative analyses with accelerated aging wood-

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Abstract

Wood is present in many cultural heritage objects thanks to its capacity to resist advantage of the Japanese context where building traditions have been maintained for centuries. The paper deals with mechanical characteristics of aged hinoki (*Chamaecyparis obtusa* Endl.) wood of Japanese historical buildings especially their Young's modulus and rupture energy. Besides, the paper deals with mechanical properties of accelerated hinoki wood by thermally treated (90,120,150,180°C) for comparison of naturally aged hinoki wood.

3 points bending test were performed in longitudinal (L) and radial (R) directions on small clear wood specimens cut from 8 historical samples and one modern reference considered of high quality by craftsmen. Although aged wood appeared more rigid and stronger than recent wood, after density and humidity corrections were applied no significant variation of L and R rigidity or L strength was observed. The post-linear behaviour, however, was drastically influenced by wood age especially in R direction where the strength and rupture energy decreased markedly with the time elapsed since the wood was processed: aged wood can be considered as safe as long as it is not loaded perpendicular to grain.

Similar phenomena were observed on thermally treated wood, quantitative differences are likely to be observed between the various kinds of modifications induced by age.

1. INTRODUCTION

Wood is a material designed by nature to last, provided it is not attacked by biological agents. It can support trees for centuries, and as a technological material it can again sustain loads for considerable periods. It is, as a consequence, a major component of the cultural heritage of many civilisations and the assessment of wood properties from ancient objects and structures is a question of fundamental and practical interest [1].

One major difficulty for such research is the gathering of suitable samples, with well-defined origin, certified dating and permission of publication by conservation administration. The Japanese context, where traditional uses of wood have been maintained for more than 1600 years, offers a unique opportunity to address the question of wood aging. Wood has always played a major role in Japanese culture. More than 90% of buildings listed as a National property or a nationally important cultural property of Japan are constructed with wood. The most famous and the world's oldest wooden construction still standing is Horyu-ji temple from the latter half of the seventh century. Since 2004, a collection of wood samples from various temples and other historical building is being gathered by the Research institute for Sustainable Humanosphere of Kyoto University (Japan), expanding a collection gathered in the 1950s by Jiro Kohara [2].

The matching of specimens from different origins is another typical obstacle. Wood is a variable material due to genetic variations and dependency on growing conditions of the trees. To discuss property changes due to aging, a recent reference is required. However, it is difficult and sometimes impossible to obtain recent wood that closely matches a given old wood sample. To overcome the difficulty, well-established structure-properties relationships can be used to produce corrections, that will allow comparing data from slightly mismatched samples.

Kohara [2] reported that the bending strength and rigidity of aged Hinoki wood, used in temple structures for over 1300 years, initially increased for a few hundred years and then decreased with increase in elapsed time. This paper presents new results obtained on similar materials. Clear wood exempt of biological attacks, effect of weathering and visible damage has been selected, so that the properties measured reflect the intrinsic aging of the material, resulting from the long-term action of moderate mechanical stress, temperature and humidity fluctuation, and air oxidation [1]. The results of mechanical testing of specimens of increasing age will be presented and discussed in relation to the possibility to predict the consequence of natural aging on wood properties.

2. Materials and method

Sample origins

The aged samples were Hinoki (*Chamaecyparis obtusa*) wood from Japanese historical buildings, mostly Horyu-ji temple in Nara. The specimens used in this study were cut from aged wooden members provided from the restoration sites, which were not reused. The modern wood used for comparison was taken from a 360 years old tree from Kiso region, where the highest quality Hinoki has been grown for the last 3 centuries, and selected according to craftsman viewpoint. It was cut in 1988 and had been subjected to slow drying for 19 years before testing in 2006. Samples labelling, origin and basic structural information are given in Table 1. To avoid the effect of UV degradation and insects, the outer layer parts and the nails were removed, and the specimens for mechanical testing were taken from the central portion of the samples. No sapwood occurrence was detected, so that all the studied material consisted of heartwood.

Age determination

To evaluate wood age, radioactive carbon dating ^{14}C and dendrochronology were used. For each sample the wood was processed as a board containing more than 60 tree rings. Tree ring dating was performed by comparing these ring patterns with a standard pattern available until back to 912 BC for Hinoki [3]. Distinct ring patterns of Hinoki enabled dating with yearly precision. For precision dating, ^{14}C wiggle-matching method was applied [4]. As shown in Table 1, the agreement between both methods was good: the difference between ^{14}C and dendro-date ranged from -40 to 29 years. These methods can only provide information about the wood age, defined as the time elapsed since wood formation in the tree. The analysis of colour variations in the same samples suggested that most of the aging occurred after wood processing [5], so that the period of time separating wood formation and tree felling should be subtracted from the wood age for the analysis of aging processes. However, in most cases this information is not available, and the time elapsed since tree felling (t_T) cannot be calculated. For the subsequent analysis, an upper bound of t_T will be considered, based on the newest visible ring on the sample. For the most recent historical sample H and the reference I, this gives a direct estimate as the bark was included in the sample. For the older samples the relative error is likely to be small. In the following, this estimate of t_T will be designated as the “age” of the sample.

an accelerated aging :Thermally tratment

Aging is defined as a slow oxidation process caused by oxygen in the air. Therefore, based on the temperature-time conversion law an accelerated aging test was performed by thermally treated. Specimens were cut out with the dimensions of 120 mm (L) \times 20 mm (R) \times 4 mm (T) from near the outermost part of the heartwood. The specimens were dried at 60°C in an air-circulating oven for 12 hours and then at a room temperature in a desiccator with silica-gel and P_2O_5 until getting constant weight. Dried specimens were heated in an air-circulating oven at 4 temperatures levels from 90 to 180 °C for a duration ranging from 0.5 hour to approximately 2 years. Table 2 shows the treated time of the specimens at each treated temperature. They were planned by assuming that a 10 °C increase is equivalent to dividing the time by 2.

Table 1: Origin and dating of the samples

| | Collection | Origin | Block dimensions (R x T x L, cm) | RW (mm) | Dendro chronology* (AD) | ¹⁴ C interval dating* (AD) | <i>t_w</i> (yrs) | <i>t_T</i> (yrs) |
|---|-------------------|-------------|-------------------------------------|------------|-------------------------------|---|-------------------------------|-------------------------------|
| A | KYOw2701, RISH | HYJ | 11 x 3.4 x 10 | 0.8 | 343 / 434 | 367 / 458 | 1618 | 1583 |
| B | KYOw2738, RISH | HYJ | 7.0 x 4.2 x 10 | 0.5 | 458 / 612 | 418 / 572 | 1467 | 1405 |
| C | private | HYJ (leg) | 6.7x 11.5 x 47 | 0.9 | 400 /502 | 418 / 520 | 1548 | 1515 |
| D | private | HYJ (leg) | 7.5 x 11.5 x 55 | 0.8 | 431 /537 | 421 / 527 | 1530 | 1480 |
| E | private | HYJ (leg) | 9.5 x 13 x 42 | 0.7 | 584 / 792 | 587 / 795 | 1319 | 1225 |
| F | private | HYJ (leg) | 5 x 7.8 x 52 | 1.0 | 1029 / 1086 | 1000 /1059 | 899 | 931 |
| G | private | HYJ (leg) | 2.5 x 14 x 58 | 0.8 | 1106 / 1270 | 1098 /1262 | 822 | 747 |
| H | (temple donation) | SJJ | 1100 (∅) x 30 (L) | 0.8 | 1069 / 1438 | 1071 / 1438 | 753 | 569 |
| I | (workshop) | Kiso forest | | 1.0 | 1622 / 1988 | 1631 / 1973 | 200 | 19 |

HYJ = Horyuji temple, Nara ; (leg) = legendly ; SJJ = Senjuji temple, Mie ; RW =average width of annual rings ; * dates (A.D.) of first/last measured growth ring ; *t_w* = mean time elapsed since wood formation in the measured portion ; *t_T* = time elapsed since tree felling (estimated upper bound for samples A to G) ; *t_w* and *t_T* are estimated from dendro dating.

Table 2:Treated temperatures and time of accelerated aged wood samples.

| | 90°C | 120°C | 150°C | 180°C |
|--|---------|-------|-------|-------|
| | 256 | 32 | 4 | 0.5 |
| | 512 | 96 | 8 | 1 |
| | 1024 | | 16 | 2 |
| | 1536 | 192 | 24 | 3 |
| | 2560 | 320 | 40 | 5 |
| | | | 56 | 7 |
| | 5000 | | 80 | 10 |
| | 7296 | 768 | 96 | 12 |
| | 9216 | | 144 | 18 |
| | 12288 | | 192 | 24 |
| | 18432 | 2304 | 288 | 36 |
| | (24576) | 3072 | 384 | 48 |
| | (30720) | 3840 | 480 | 60 |
| | (36864) | 4608 | | 72 |
| | (43008) | | 672 | 84 |
| | (49152) | 6144 | 768 | 96 |
| | (61440) | 7680 | 960 | 120 |

The treated at 90°C is now in processing planed with treated time in parenthesis.

Bending test

Wood is a highly anisotropic material, much more rigid and strong along fibres (longitudinal direction, L) than across fibres (radial direction, R, or tangential, T). Although the loading of beams is dominantly applied in L direction, in the connections parts a complex stress state occurs and the response to transverse loading may become critical. For that reason, 3 points bending tests were performed not only in L, but also in R direction whenever enough material was available. Matched specimens of dimensions 60 mm (L) × 10 mm (R) × 2 mm (T) were cut for L tests, 60 mm (R) × 10 mm (T) × 2 mm (L) for R tests. The samples were initially dried at room temperature for 3 weeks in a desiccator with silica gel, then conditioned at 20°C and 60% relative humidity (R.H.). They were weighed before and after the tests performed in the air-dry condition, then oven dried at 60°C, 24 hours at atmospheric pressure and 24 hours in vacuum in presence of P₂O₅, and weighted again to calculate the moisture content during the test, as well as the oven-dry density and the air-dry density.

The tests in L and R directions were performed on 5 to 10 specimens per sample and loading direction, with span length 50 mm and crosshead speed 5 mm/min. For comparison of naturally aged hinoki wood, heat treated hinoki wood was tested as same way. Equivalent stress (σ) and strain (ε) were calculated from the load (F) and crosshead displacement (f) as follows:

$$(1) \quad \sigma = 3.Fl / (2t^2w)$$

$$(2) \quad \varepsilon = 6f / l^2$$

where t is the specimen thickness, w its width, l the span. These expressions correspond to the maximum value in the central part, assuming homogeneous mechanical response and linearity between stress and strain. The following parameters were used to describe each stress-strain curve: the Young's modulus or rigidity (E) defined as the initial slope; the elastic limit (ε^e) as the strain where the stress falls by 1% below the linear extrapolation from linear part; the strength (σ^m) as the peak stress; the breaking strain (ε^m) as the strain at peak stress; the rupture energy (W) as the area below the curve up to the complete rupture, divided by the cross-sectional area

$$W = \frac{1}{tw} \int F.df$$

where F is the applied force and f the displacement. From equations (1) and (2), W can be expressed as:

$$(3) \quad W = \frac{L}{9} \int \sigma.d\varepsilon$$

3. Results and discussion

3.1 Mechanical properties of aged wood

Stress-strain relationship

Fig. 1 shows typical stress-strain curves in L and R bending for each aged sample. In the following, the index L and R will be used to distinguish the value for each loading direction.

Our range of air dry density, 0.33~0.49 g/cm³ or of oven dry density, 0.32~0.46 g/cm³, almost covers that of modern hinoki [5, 6]. As a general trend, aged wood appeared stiffer (higher E) and stronger (higher σ^m), at least in L direction, than the modern wood tested. As will be discussed below, this can be partly explained by differences in density and moisture content. The post-linear behaviour of aged wood, on the other hand, was clearly more brittle than in modern wood: this increase in brittleness, apparent from the curves of Fig. 1. All aged R specimens exhibited a fragile response, so that the elastic limit ε^e could only be estimated for the recent wood (sample I).

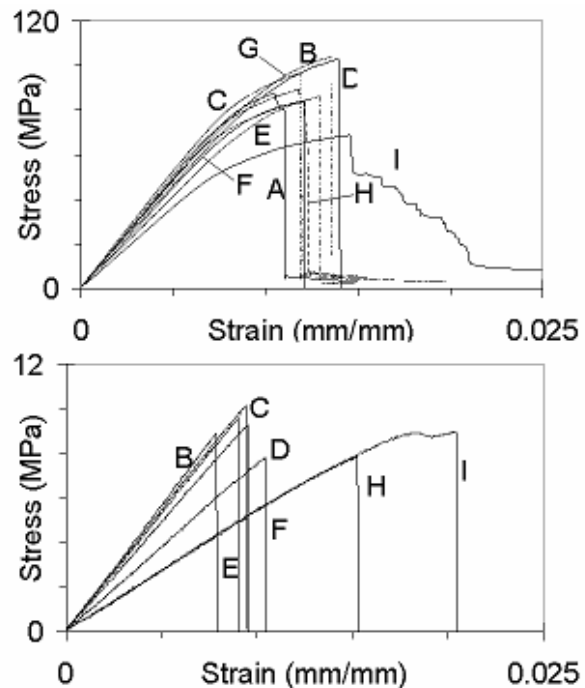


Fig.1 Typical stress-strain curves
Upper: L direction / Lower: R direction

Corrections for sample matching

Fig. 2 shows the relationships between sample age and air dry density (d_a): the older the specimens observed in our study, the denser the wood. In contrast to the density, no systematic variation of the MFA was observed: the value of the modern reference was close to the average

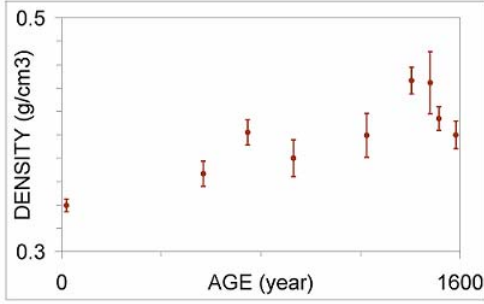


Fig.2 Relationship between age and density

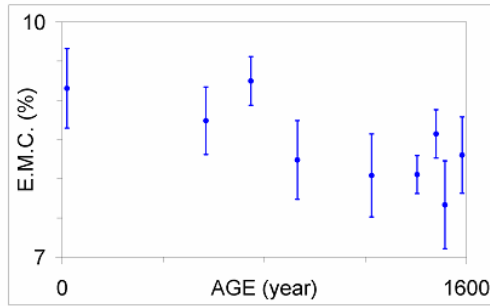


Fig.3 Relationship between EMC and age.

of all aged samples, and the range of variation was small anyway. Fig.3 shows the relationships between equilibrium moisture content (h) and age: h tends to decrease with age according to a linear regression:

$$(4) \quad h = -0.00075 t_T + 9.2 \quad (R^2=0.57)$$

According to equation (4), 1500 years reduce h by about 1.1%, which is close to the decrease of 1.0% for 1200 years for the equilibrium moisture content at 60%RH reported by Kohara [2]. These two effects of the age, on d and h , bear different meanings: while h decrease indicates a modification of the material properties with time, the d increase reflects a possible evolution, over the centuries, of the quality of hinoki wood considered as suitable for structural uses. Since h and d influence the mechanical properties of wood, both effects need to be understood and taken into account in the analysis of the effect of elapsed time on wood properties.

Generally speaking, quantities describing the rigidity and strength of wood tend to increase with d and decrease with h . The situation is a little more complex for the effect of h on rupture energy because of the higher breaking strain in wetter wood that compensates the lower stress.

The following expression can be used to describe the influence of d and h in the case of modern softwood:

$$(5) \quad X(d, h) = (d/d_0)^n [1 - \alpha \times (h - h_0)] X(d_0, h_0)$$

where X stands for Young's modulus (E_L, E_R), strength (σ^m_L, σ^m_R), fracture strain ($\varepsilon^m_L, \varepsilon^m_R$) or breaking energy (W_L, W_R) in L and R directions, respectively, d_0 and h_0 are reference density and humidity, respectively, and α and n are constants depending on the type of quantity X . The chosen set of values for α and n is shown in Table 3.

Table 3 Parameters used for density and humidity corrections

| | L | | | | R | | | |
|----------|-----|------------|--------------------------------|---|-----|----------|-----------------|-----|
| | E | σ^m | $\varepsilon^c, \varepsilon^m$ | W | E | σ | ε^m | W |
| n | 1 | 1 | 0 | 1 | 1.5 | 1.5 | 0 | 1.5 |
| α | 1.5 | 6 | 0 | 6 | 3 | 4 | 0 | 4 |

It is taken from usual interspecies relationships for softwoods [7-11], although some authors proposed linear regression for density [8]. In L direction, the value of $n=1$ can be justified by the quasi parallel disposition of cell walls and cell cavities [7, 10, 12]. It can differ from 1 within a given species, when MFA and density variations are correlated. Indeed, the observed variations of E_L and σ^m_L within modern Hinoki wood [13] could have suggested higher values of n for L direction. However, based on the observation of similar MFA among the samples, the value $n=1$ seemed more appropriate in the present situation. Transversally to the fibres, the dominance of cell-wall bending could increase n up to the theoretical value of 3 [14], but in R direction due to the radial alignment of the cellular structure this level can never be reached and a range $n=1.0\sim 1.8$ is generally observed [7, 9]. No effect on the strain at peak stress ($\varepsilon^m_L, \varepsilon^m_R$) was assumed.

Expression (5) can be reversed in order to produce a correction to the measured data $X(h, d)$ relative to d_0 and h_0 :

$$(6) \quad X(d_0, h_0) = \frac{X(d, h)}{(d/d_0)^n [1 - \alpha \times (h - h_0)]}$$

Since the humidity change is itself a part of the aging process, the humidity correction may be avoided, leading to the correction:

$$(7) \quad X'(d_0, h_0) = \frac{X(d, h)}{(d/d_0)^n}$$

The values for recent wood: $d_0 = 0.34 \text{ g/cm}^3$ and $h=9\%$, will be taken as reference. The underlying viewpoint with equation (7) is that the data obtained on old wood are used to predict the long-term properties of the present material, while equation (6) highlights the property changes not merely due to the drop of moisture content. Due to the small effect of age on equilibrium moisture content, the difference between corrections (6) and (7) was small. In the next graphs, the correction according to formula (6) will be used.

Fig. 4a, b shows the evolution of rigidity with time. When no correction was applied, both E_L and E_R seemed to increase. However, after correction the trend vanishes, and both values fluctuate around the reference value.

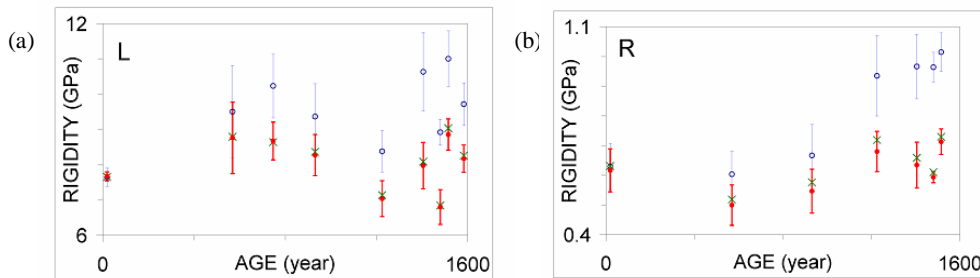


Fig.4 Effect of specimen age on Young's modulus.(a) L direction (b) Rdirection

Error bars indicate the standard deviation. Empty symbol and thin bars: uncorrected values; filled symbol and thick bars: corrected values. X-marks: specific values.

The same can be said for σ^m_L in Fig. 5a, but Fig. 5b shows a marked decrease of σ^m_R , especially after application of the correction.

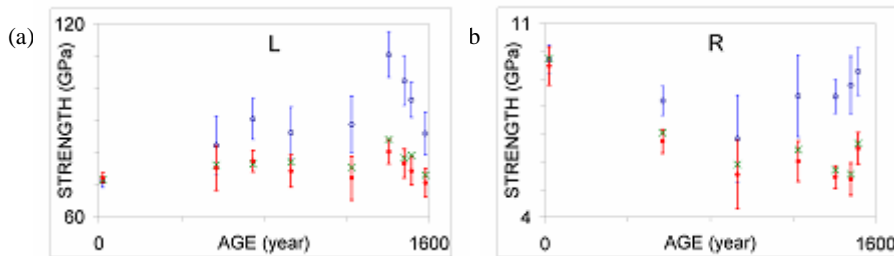


Fig.5 Effect of specimen age on strength. (a) L direction (b) Rdirection

Fig. 6a shows also a clear decrease of W_L thanks to the correction, while in Fig. 6b the fall of W_R is drastic and would have been observed even without correction. In these figures, the error bar indicates the standard deviation. In all cases, the correction resulted in a lower dispersion of the results.

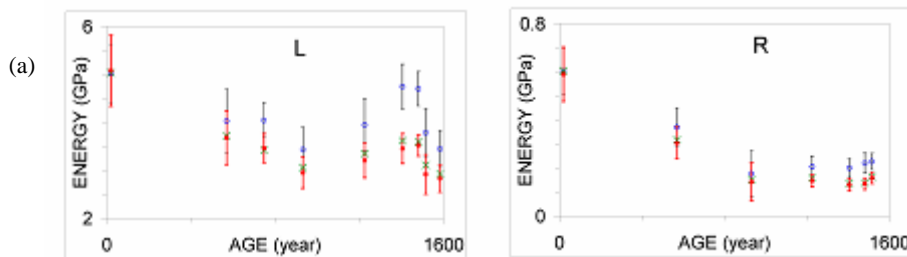


Fig.6 Effect of specimen age on rupture energy (W).(a) L direction (b) Rdirection

Fig.7 shows the effect on strain parameters, for which no correction was tried: the elastic limit (ϵ^e) in L direction and the breaking strain (ϵ^m) in L and R directions. A slight increase of ϵ^e_L , a slight decrease of ϵ^m_L and a drastic decrease of ϵ^m_R were observed.

The effect of aging on mechanical properties

The difference of behaviour between L and R directions can be explained by the structural organisation of wood fibres. In a softwood like hinoki, most of the wood is composed of tubular tracheid cells, typically 3.5 mm long, 40 μm diameter and with a cell-wall thickness varying between 2 μm in earlywood and 4 μm in latewood [15]. The cell-walls are multilayered, with a dominant S_2 layer made of axially oriented crystalline cellulose [16]. There is no storied structure in hinoki; tracheids overlap randomly so that under loading in L direction, the stress is efficiently transferred from one cell to the next and most of the load is supported by the cell walls. Under R loading, a much larger proportion of the stress is supported by the middle lamella. L and R also differ at the meso-scale: the response of thin-walled tracheids in earlywood contributes dominantly under R load while the denser latewood bears a significant part of the L load. R rupture occurs usually by the failure of an alignment of earlywood cells, whereas in L direction a complex rupture path involving much slippage between fibres is generally observed. This explains, for the recent wood, the more developed post-linear response in L direction as compared to R [17].

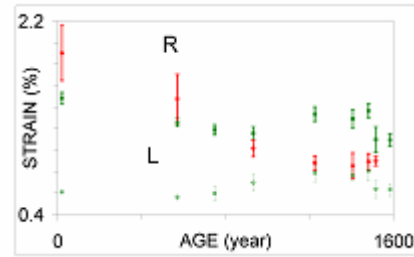


Fig.7 Relationships between strain and age.

Although aged wood appeared more rigid and stronger than the recent wood, after density and humidity corrections no clear trend was anymore observed for L and R rigidity, as well as for L strength. The post-linear behaviour, however, was drastically influenced by wood age. Aged wood was brittle, especially in R direction where the linear elastic limit was not reached during the tests; the higher brittleness was also made evident in L direction by the decreasing distance between ϵ^m and ϵ^e . The cellulose content and crystallinity ratio, on the other hand, do not vary significantly within modern hinoki [18] as well as between aged and modern hinoki [1]. Chemical analysis and thermomechanical testing performed in parallel to the present study evidenced a decrease of hemicellulose content [19], as well as an increase of lignin crosslinking [20]. Similar observations were previously made on other aged wood by using FTIR [21]. Hemicelluloses ensure the transverse cohesion between cellulosic microfibrils and lignin matrix, so that their degradation, especially in S_2 , is also more detrimental to R than to L direction. The increase of crosslinking would increase the brittleness of lignified parts, especially in the middle lamella, thus accounting for the drastic decrease of R toughness without inducing any drop in rigidity.

3.1 Mechanical properties of thermally treated wood

Fig.8 shows the changes of rigidity, young's modulus and rupture energy of dry thermally treated woods and aged woods with no correction as a function of temperature and time. Kohara observed that all these features are similar to the effect of a thermally treatment [18, 22]. The general trends might be similar, quantitative differences are likely to be observed between the various kinds of modifications induced by age. The relation of the kinetics of identified phenomena with physical and chemical changes remains to be investigated. Analysis on the time- temperature equivalency in these woods is under progress concerning these mechanical properties.

Another common effect of aging and thermally treatment is the colour change. In an another parallel study, lightness L^* systematically decreased with time or temperature, variations of redness a^* and yellowness b^* exhibited complex patterns, indicating the combined action of several processes subject to different thermally activation. The extrapolation to the colour of ancient wood was only possible by taking into account the accelerating effect of the ambient conditions, taken as equivalent to higher temperature levels.

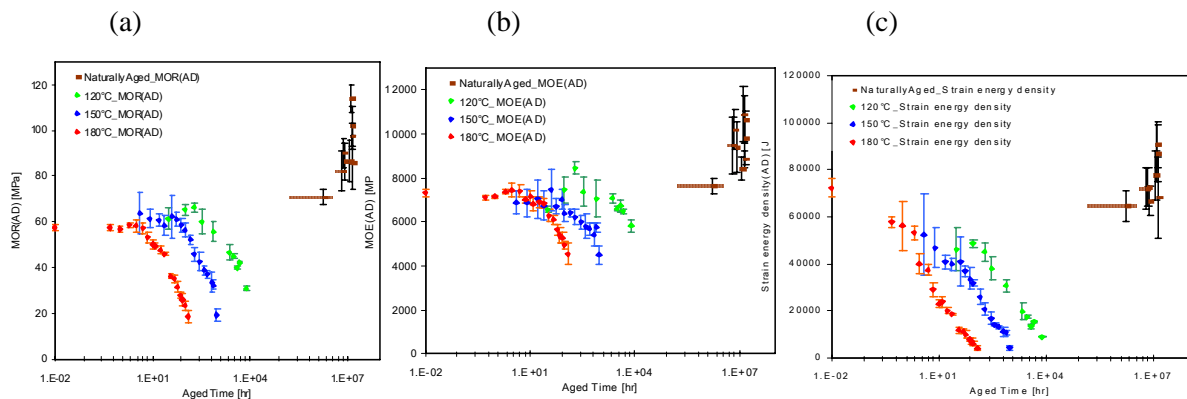


Fig.8 changes of rigidity, Young's modulus and rupture energy age
 (a) Rigidity, (b) Young's modulus, (b) rupture energy

4. Conclusions

As a practical consequence, the results obtained suggest that ancient wood can be considered as safe as long as it is not subject to unusual action perpendicular to the grain. The existence of large wooden structures dating back more than 1200 years from now is the clearest confirmation of that statement. The trend of mechanical properties of aged wood are similar to the effect of a thermally treatment were observed. But the mechanism should be discuss in aged woods and dry thermally treatedwood in detail on the time- temperature equivalency.

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