COMPRESSION OF WOOD WITH SUPERIMPOSED SMALL SINUSOIDAL OSCILLATIONS. PART II: HIGH TEMPERATURE

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ABSTRACT

The changes occurring in wood when it is subjected to compression straining 7.0% parallel to the grain or 10.0% perpendicular to the grain in the temperature range 70–170 C have been studied. Small dynamic strain oscillations at a frequency of 9 Hz were superimposed during the straining.

The changes occurring in the wood have been determined by measurement of the resulting permanent strain of the wood specimens. Fiber damage has been evaluated by measuring the intrinsic viscosity of the pulp after a standardized sulfite delignification. Some microscopic examinations were also carried out. The energy consumption during compression treatment has also been considered.

Compression straining at temperatures of 150 C and above was found to be favorable with respect to wood disintegration yielding a high degree of wood deformation with low fiber damage and at low energy consumption.

Keywords: Compression, sinusoidal oscillations, fiber damage.

INTRODUCTION

The effects on wood of compression straining at room temperature were investigated in a previous study (Winter and Mjöberg 1984). However, since temperature has considerable influence on the behavior of wood, that study has now been extended to higher temperatures. With increasing temperature, viscoelastic and plastic flow during deformation increases. Thus, higher temperatures lead to an increase in the rates of creep and relaxation (Arima 1967; Davidson 1962; Kingston and Budgen 1972; Kitahara and Okabe 1959; Kunesh 1961; Youngs 1957) and to a decrease in the modulus of elasticity (Bach and Pentoney 1968; Bernier and Kline 1968; Dinwoodie 1975; Ganowicz et al. 1980; Norimoto and Yamada 1965; Suzuki and Nakato 1964).

The flow behavior is not uniform in wood and the softening temperatures are dependent on the water content in the wood (Goring 1963). In a water-saturated state, isolated amorphous carbohydrates (hemicellulose and amorphous cellulose) will be in a soft stage at room temperature, whereas at dry conditions the softening temperature is in the neighborhood of 200 C (Salmén 1982). Softening of water-saturated lignin in situ occurs at 65–90 C and at about 5 C higher temperature at 12% moisture content (Irvine 1980). The development of permanent deformations in wood during straining will therefore depend on the actual softening situation.

During straining of wood above the proportional limit at lignin-softening tem-

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peratures, permanent deformations will be more prominent in lignin rich regions, for instance the middle lamella. However, the lignin-softening temperature interval given above only indicates the level of the most drastic change; a complete softening of the lignin is not obtained until temperatures of 150–170 C are reached (Koran 1967). To promote flow in the middle lamella, temperatures as high as these should thus be chosen. This has also been verified from disintegration in refiners (Asplund 1953; Atack 1972; Winter et al. 1984) and from the breakdown of wood under tensile or shearing stresses (Atack et al. 1961; Carlsson and Lagergren 1957; Koran 1968; Ohsawa and Yoneda 1978; Suzuki et al. 1979).

The object of this study was to investigate the effect of temperature during the compression of wood with the intention of minimizing the extent of fiber damage obtained. To promote softening conditions, water-saturated specimens were used. A temperature interval from 70 C to 170 C was chosen for the studies.

The deformation of the wood structure (including both permanent deformations within the fibers and inter-fiber displacements) was evaluated by measuring the total permanent strain in the loading direction and in some cases by observation by polarized light microscopy. Fiber damage was assessed by viscosity determinations. Small strain oscillations were superimposed during the straining and the energy absorption during these cyclic stress-strain loops was also determined. The object was to find conditions yielding substantial permanent strain at preserved viscosity.

EXPERIMENTAL

The experimental procedures with respect to specimen preparation, compression straining, and analysis were the same as those used in the room temperature experiments described earlier (Winter and Mjöberg 1984).

For heating purposes, the compression plates and the specimen space were encapsulated in an autoclave around which an electric heating ribbon was wound. The temperature was measured with a resistance temperature detector placed near the specimen.

The strain of the specimen during compression was obtained from the displacement of the moving compression plate measured with a proximity sensor. Another proximity sensor was used to control and minimize the influence of vibrations of the static compression plate. During proper conditions, the amplitude of the vibration of the static plate was less than 5% of the amplitude setting of the movable plate.

Water-saturated conditions were obtained by saturating the specimens and by having the autoclave filled with water. In some cases the specimens were pretreated with sodium sulfite. Those treatments were performed using a 4% sodium sulfite solution instead of pure water during saturation of the specimens and in the autoclave.

After the system was heated to the desired temperature, the mechanical treatment was carried out. The treatment consisted of a compression period at a constant rate of displacement (1.0 mm/min) followed by a stress relaxation period at the chosen strain level. During the experiments, small sinusoidal strain oscillations were superimposed (see Fig. 2 in Winter and Mjöberg 1984). In compression parallel and perpendicular to the grain, the chosen strain levels were 7.0% and 10.0%, respectively. The time of stress relaxation was 5 min, the amplitude

in the sinusoidal strain oscillations was 0.26%, and the frequency was 9 Hz in all experiments. Immediately after the mechanical treatment, the autoclave was opened and the specimen was removed. The total time of the mechanical treatment was about 6 min. The time at the chosen temperature was about 8 min reached after 0 min at 70 C, 2 min at 90 C, 5 min at 110 C, 10 min at 130 C, 16 min at 150 C, and 23 min at 170 C.

Stress and strain values were recorded during the experiments. An average stress-strain loop area was determined each 5 sec from ten consecutive loops by use of memory oscilloscope. A mean loop area, representing the whole experiment, was obtained from the mean of these average loops.

In the linear viscoelastic range, the loop area (hysteresis loss) W can be expressed as (Findley et al. 1976):

$$W = \pi\sigma_0\epsilon_0 \sin \alpha \tag{1}$$

where σ_0 is the stress amplitude, ϵ_0 the strain amplitude, and α the phase angle between stress and strain. The dynamic elastic modulus E and the internal friction Λ can then be expressed as

$$E = \frac{\sigma_0}{\epsilon_0} \cos \alpha \tag{2}$$

and

$$\Lambda = \pi \tan \alpha \tag{3}$$

With the small strain amplitude chosen, loops almost in accordance with linear viscoelasticity were obtained (as could be judged from the elliptical shape of the stress-strain loops) and Eqs. (1) through (3) were used to determine softening (E) and loss effects (W). Stress and strain amplitudes (σ_0 and ϵ_0) were obtained from the average loop. The softening was expressed as a stiffness ratio, calculated from Eq. (2) as the actual average elastic modulus in relation to the average modulus during the compression period at 70 C.

Permanent strain of the specimens (from the relative difference in length of the specimen before and after the mechanical treatment) and pulp viscosity ratios of the delignified specimens (ratio of viscosity for mechanically treated specimen and reference specimen) were determined as in the first study (Winter and Mjöberg 1984). The pulp viscosity could not, however, be determined on all specimens that had been treated at 170 C. Because of thermal degradation, specimens treated in pure water were dark in color and impossible to delignify during acid cooking conditions. However, in the experiments where a sulfite pretreatment was used at that temperature, no thermal degradation occurred and the viscosity analysis could be carried out.

In the figures, each point is the result from one measurement only, i.e., from one specimen only.

RESULTS

Softening effects

With increasing temperature the softness of wood increases. This is seen in Fig. 1, where the average stiffness ratio during both the compression period and the

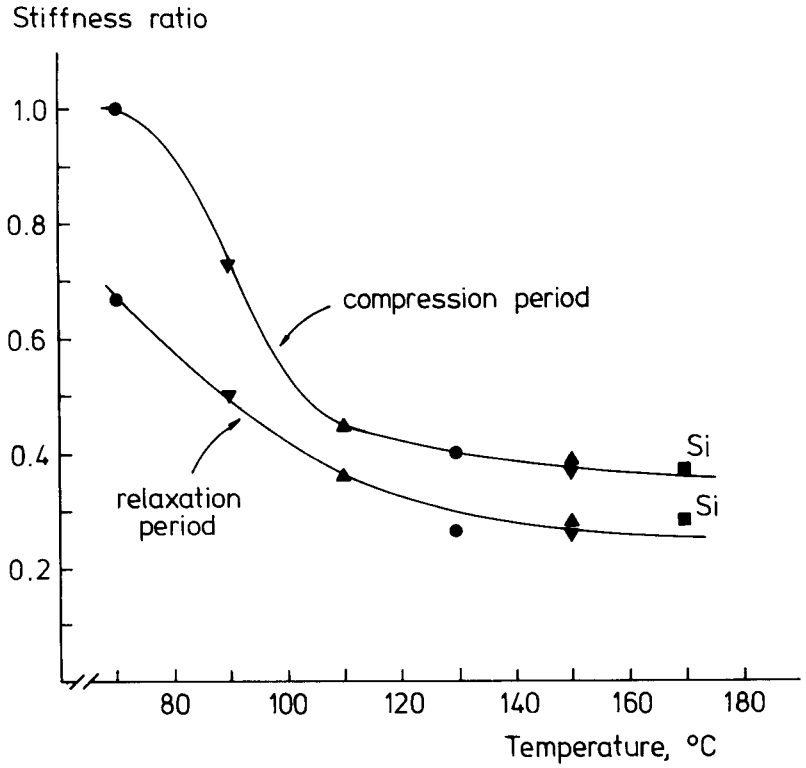

FIG. 1. Stiffness ratio (average) after compression 7.0% parallel to the grain versus temperature. Different symbols are used for specimens originating from different wood blocks (specimens from different places in the circumference of the tree). The specimen compressed at 170 C has been pretreated with a sulfite solution.

relaxation period for specimens compressed parallel to the grain is presented as a function of temperature. The stiffness is lower during relaxation than during the preceding compression period because of relaxation phenomena and possibly also because of mechanical degradation in the wood. In the temperature range from 70 C to 110 C, there is a substantial softening effect.

The softening temperatures of the wood constituents are frequency dependent. At the same frequency level as used in this work, a lignin softening temperature of 100 C has been reported (Atack 1972; Salmén 1982). Thus, the softening in the range from 70 C to 110 C probably reflects the softening of the lignin. However, the softening is not a distinct transition that occurs at a well-defined temperature. The lignin gradually changes from a hard and glassy to a soft and rubberlike state with increasing temperature. Thus, the softening effects obtained in the range 110–150 C probably reflect the successive transition into the total softening of lignin. This would also be in accordance with the temperature range 150–170 C for complete lignin softening given by Koran (1967). Atack (1972) and Salmén (1982) defined the softening temperature as the temperature giving maximum internal friction. By determining the internal friction from Eq. (3), a maximum was obtained at a temperature of 110 C in this work.

Figure 2 shows the permanent strain ratio after compression 7.0% parallel to or 10.0% perpendicular to the grain as a function of temperature. Obviously,

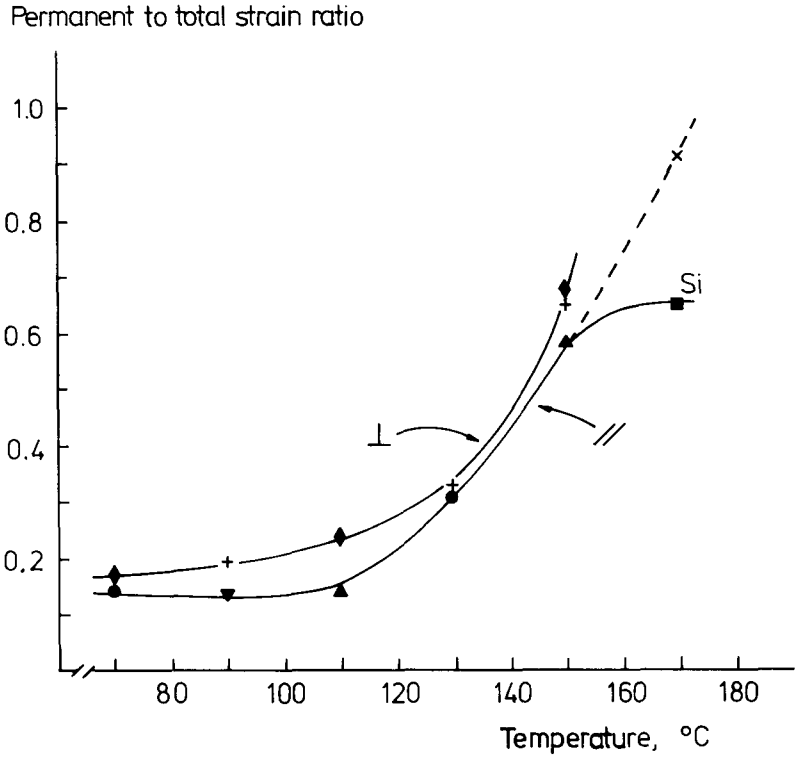

FIG. 2. The ratio of permanent to total strain after compression 7.0% parallel (//) or 10.0% perpendicular (⊥) to the grain versus temperature. Different symbols are used for specimens originating from different wood blocks. The lower point at 170 C has been pretreated with a sulfite solution.

increased temperature promotes further permanent deformation in the wood structure upon loading. In particular, the permanent strain increases above 110 C. This is the case both for compression parallel to and for compression perpendicular to the grain. Reference to Fig. 1 shows that the main increase in permanent strain occurs at temperatures above the main softening temperature interval. It thus seems that in order to utilize the lignin softening to favor a large permanent deformation, a nearly total softening of lignin should first be attained.

At 170 C, a permanent strain close to the total strain is obtained (upper point). However, at this temperature the wood was found to suffer from thermal degradation. Breakdown of the cellulose structure might therefore also have contributed to the permanent strain. Using a weak alkaline sulfite solution, such effects were avoided (lower point). The presence of sulfite at moderate temperatures is known to yield additional softening effects (Atack et al. 1978). However, the present study seems to indicate no additional softening effect due to the presence of sulfite at such high temperatures as 170 C. This is indicated in Fig. 1 and is also in agreement with other studies (Atack and Heitner 1979).

Fiber damaging effects

The viscosity ratio of sulfite delignified specimens after compression straining at different temperatures is presented in Fig. 3. The viscosity ratios of both the whole specimen and of the end region of the specimen are included.

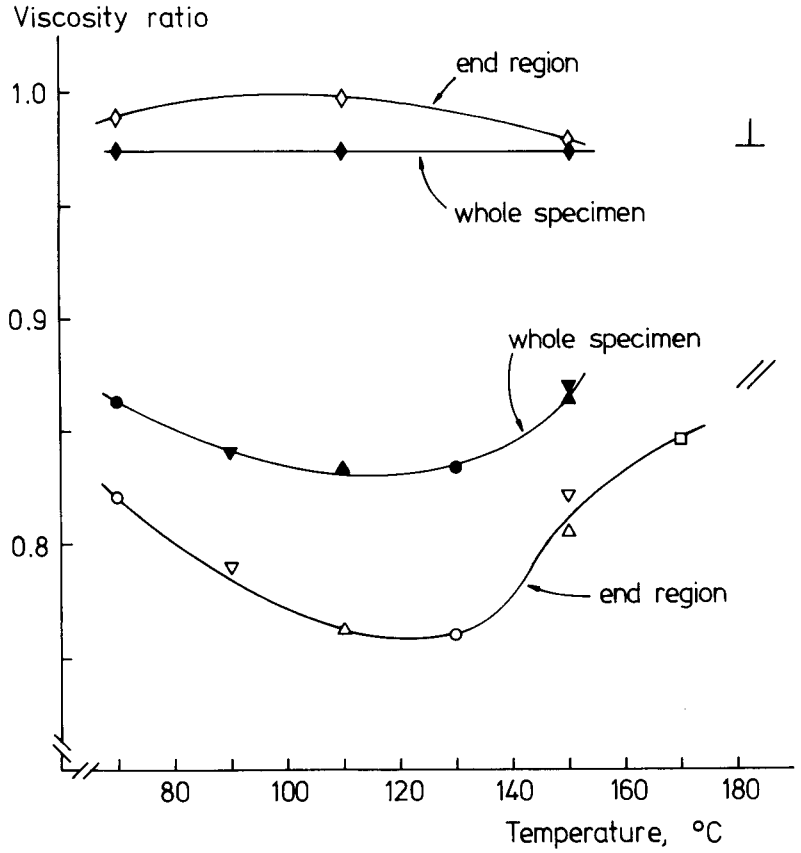

FIG. 3. Viscosity ratio of whole specimen (filled symbols) and of end regions (open symbols) after compression 7.0% parallel (//) or 10.0% perpendicular (⊥) to the grain versus temperature. Symbols according to Fig. 2.

In the perpendicular-to-grain compression experiments, there is little effect of temperature on the viscosity although the total compression strain is 10%. Surprisingly, the viscosity ratio of the whole specimen was slightly lower than that of the end regions which suggests that the fibers in the middle part of the specimen have suffered more damage. In the room temperature experiments (Winter and Mjöberg 1984) there was a concentration of the damage to the ends of the specimen. In the radial direction of wood the strength is actually determined by the weakest early-wood layers (Bodig 1963, 1965; Higgins and Griffin 1947). It thus appears that in the particular specimens used in the perpendicular studies of Fig. 3, these layers were to be found in the middle part of the specimen.

In compression parallel to the grain, considerable fiber damage, as indicated by a substantial viscosity reduction, occurred. A total compression straining of 7.0% resulted in a reduction in viscosity of more than 12%. The temperature during straining also influenced the result. In the range from 70 C up to 130 C, the extent of fiber damage increased with increasing temperature, but above 130 C the fiber damage decreased. Maximum fiber damage seems to occur at about 130 C. The effect is more significant in the end regions of the specimens. Thus,

there is an increase both in permanent strain (Fig. 2) and in viscosity after delignification (Fig. 3) at temperature above about 130 C. If the straining effects at 70 C and at 150 C are compared, the viscosities are at the same level, whereas the permanent strain is considerably higher at 150 C. This demonstrates that an increase in permanent strain at temperatures where lignin has become well softened is not related to fiber damaging deformations. At high temperatures, deformations in middle lamella regions are probably more important. In these cases inter-fiber sliding motions in the axial direction, achieved by a flow behavior of the middle lamella, seem to be the dominant deformation processes.

A difference in the deformation of wood at different temperatures could also be observed by means of polarized light microscopy. Studies were made of end-region specimens compressed parallel to the grain at 70 C, 110 C, 130 C and at 150 C. With increasing temperature from 70 C up to 130 C, the amount of microscopic deformation (slip planes) increased, and the microscopic deformation also penetrated deeper into the specimen. In the earlier room-temperature experiments (Winter and Mjöberg 1984), an increase in softening achieved by increasing the moisture content or by decreasing the frequency also increases the amount of microscopic fiber deformation.

At 150 C, where less fiber damage occurred, the presence of deformation processes other than the axial sliding mentioned above was also observed. In accordance with the appearance at lower temperatures, microscopic deformation as slip planes was still frequently distributed along the fibers, but a wavy appearance due to buckling of the fiber walls was also present. It is suggested that this process is obtained when the middle lamella region becomes so well plasticized that the fibers will be able to behave to a certain degree as isolated columns. In such a state, there will be less support for the fiber walls from the middle lamella and lateral fiber deformation through side-wise buckling will be promoted. Some support from adjacent fiber walls leads to a wavy appearance of the fiber matrix.

Structural breakdown in relation to energy consumption

Energy will be absorbed as a result of both the overall compression straining and the superimposed stress-strain oscillations.

Figure 4 presents the energy absorption due to the overall straining after compression at different temperatures. The energy absorption decreases with increasing temperature. The maximum stress during straining and the extent of straining (indicated by the total permanent strain) will influence the energy absorption. Since the permanent strain increases with increasing temperature, the decrease in energy absorption with temperature is mainly a consequence of the decreasing compression strength.

The energy absorption due to the superimposed oscillation was determined in the parallel case and was also found to decrease with increasing temperature (Fig. 5). Since the stiffness decreases in a similar fashion (Fig. 1), the increased softening with temperature is thus responsible for this reduction in energy absorption.

Considering the structural breakdown of wood with respect to energy consumption and fiber damage, the most favorable conditions are obviously obtained at the highest temperatures used in these studies. At 150 C and at 170 C (sulfite), both the energy consumption and the extent of fiber damage are reduced. In the

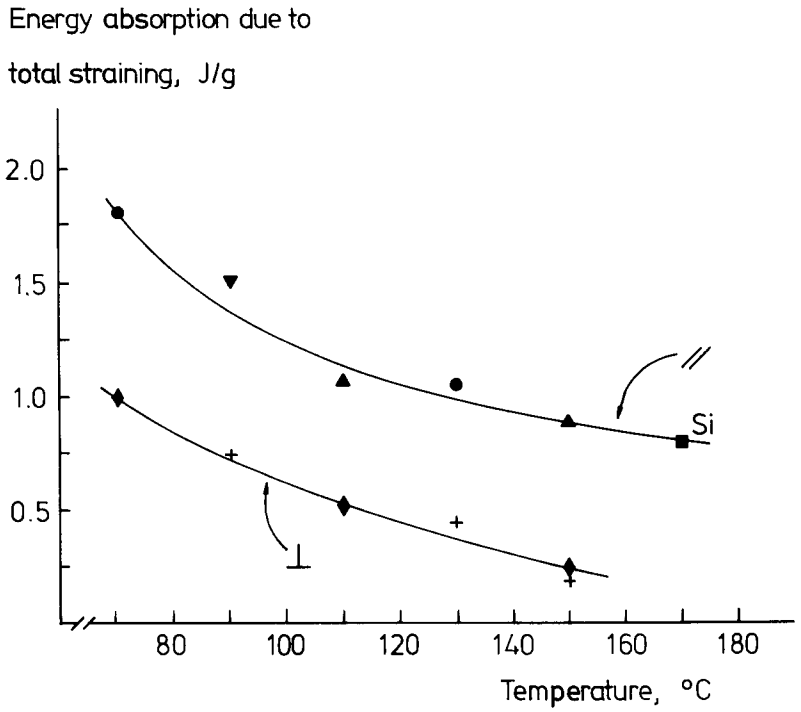

FIG. 4. Energy absorption due to total straining after compression 7.0% parallel (//) or 10.0% perpendicular (⊥) to the grain versus temperature. Symbols according to Fig. 2.

direction of the load a considerable wood-deformation effect (as permanent strain) is also obtained at these temperatures.

According to Höglund et al. (1976), the conditions yielding the highest energy absorption during oscillation in the viscoelastic range would also be the most favorable conditions for obtaining structural fiber changes by means of internal bond breaking at a given stress. At maximum energy absorption, the maximum structural change would thus be obtained. For spruce, a maximum in energy absorption was obtained by Höglund et al. (1976) at about 80 C in torsional oscillation experiments at about 1 Hz. At the frequency used in the present work, the maximum should occur at a temperature of about 90 C since the maximum is shifted about 10 C per decade of frequency (Becker et al. 1977).

However, using the viscosity reduction to indicate the extent of structural fiber change, the most pronounced effect in the parallel to grain case is apparently at about 110–130 C (Fig. 3). This would also be in accordance with the results in oscillating compression experiments at 10 Hz perpendicular to the grain by Salmén (1982), although only two temperature levels (80 C and 100 C) were compared. At the higher temperature, a more efficient breakdown (greater change in dynamic elastic modulus) was obtained. Thus, there does not seem to be any positive relationship between energy absorption and structural damage. Instead, it seems probable that the energy absorption is related mainly to viscoelastic deformation where the energy only dissipates (heat losses). The fiber damage would thus mainly be an effect caused by the total overall compression straining and the quantity of

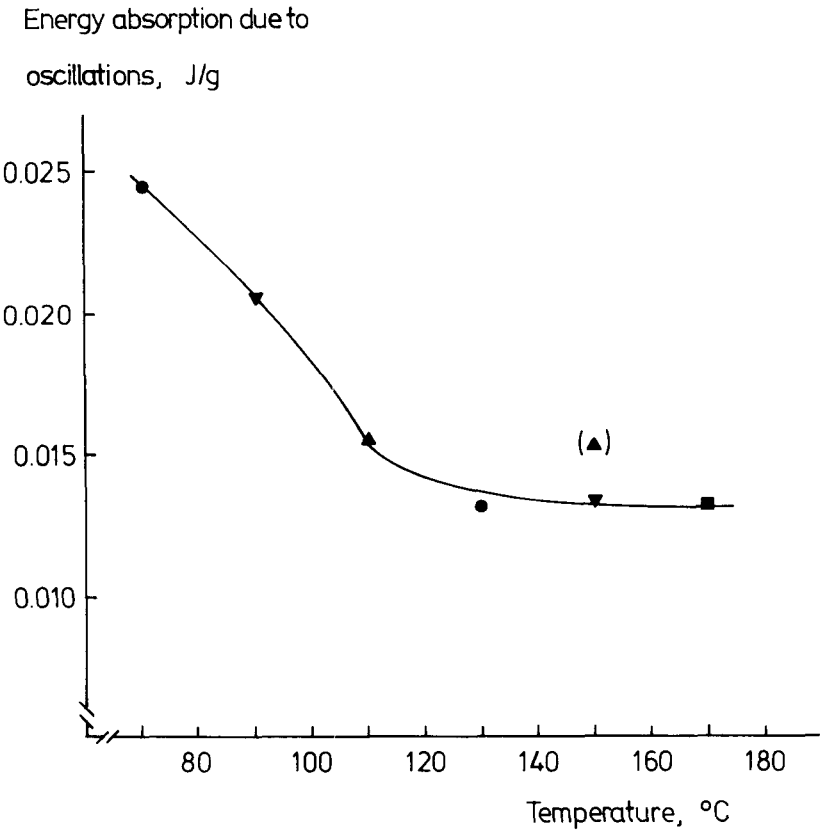

FIG. 5. Energy absorption in an average cycle due to oscillation after compression 7.0% parallel to the grain versus temperature. Symbols according to Fig. 1.

the damage (as drop in viscosity) will be determined by the actual softening conditions (e.g., by the temperature).

In thermomechanical pulping, internal bond breakage in the fibers is necessary to achieve acceptable paper properties since this increases the flexibility of the fibers and thus improves inter-fiber bonding. From some refiner experiments, a maximum in sheet strength properties was obtained at 129 C (Higgins et al. 1978). At lower temperatures, fiber shortening was favored, whereas at higher temperatures the fibers became difficult to fibrillate hence, the most favorable compromise between ease of fibrillation was obtained at about 130 C. This result may be compared with those obtained in the present study. Using the permanent strain in the range of 110–150 C to indicate the ease of fiber separation and using the pulp viscosity to indicate structural fiber changes, a temperature of 120–130 C apparently yields optimum pulp properties. At these temperatures, both fiber separation (indicated by permanent strain) and fiber breakdown (indicated by drop in viscosity) are promoted due to the softened state of the lignin. It should be emphasized that this is valid only for pulps where fiber flexibility is considered important for the sheet strength. Using the fibers in chemical pulping, the drop in viscosity should be kept at minimum.

However, as the softening of the wood is time-dependent, the comparison above presumes that the frequency in the present study and that in refining (Higgins et al. 1978) are of about the same order of magnitude. In refining, a frequency of 10^4–10^6 Hz is generally used if only the speed of rotation and the geometry of the plates are considered (Atack 1972; Becker et al. 1977; Higgins et al. 1978). If the probability of an impact on a fiber is taken into account the effective frequency is substantially reduced. The average number of impacts experienced by a single softwood fiber passing through a refiner has been estimated to be about 340 (Leider and Nissan 1977). Assuming a residence time of about 0.1 sec, this corresponds to an effective frequency of about 3,400 Hz. Recalling the small fiber damaging effect of impacts in the direction perpendicular to the grain, it is also likely that fiber damage during refining is mainly an effect of impacts in the axial fiber direction. If only such deformations are taken into account, the frequency of damaging impacts will be further, probably substantially, reduced. It is thus probable that the frequency leading to breakdown of the fibers in a refiner is considerably lower than has generally been considered. According to the frequency-temperature shift (Becker et al. 1977; Salmén 1982), the lower temperature limit in the range of optimum pulp properties (120–130 C) would correspond to a frequency of about 100 Hz. Hence, on the basis of the comparison presented, the frequency of the fiber damaging processes during refining might be as low as 100 Hz or less.

CONCLUSIONS

The deformation of wood as indicated from the permanent strain, in compression parallel or perpendicular to the grain, changes very little upon heating through the temperature interval of main softening, i.e., less than 120 C, but increases considerably upon further heating up to 170 C.

In compression parallel to the grain, the extent of fiber damage, as indicated from drop in viscosity of corresponding sulfite pulp, increases somewhat with increasing temperature between 70 C and 130 C. On a microscopic level, the effect was noticeable as more numerous microscopic fiber deformations penetrating deeper into the specimen at higher temperatures. Above 130 C, the extent of fiber damage decreases with increasing temperature due to decreased stresses in the fibers. At 150 C, local buckling of the fiber walls was observed yielding a wavy appearance of the fibers.

In compression perpendicular to the grain, the fiber damaging effect is considerably lower than in compression parallel to the grain and the temperature has only a minor effect on the extent of the damage.

The energy absorption decreases with increasing temperature. Temperatures above 150 C are favorable with respect to minimum energy consumption and structural deformation without fiber damage. Around that temperature considerable permanent deformation occurs with low energy consumption as well as low fiber damage. At 170 C and at higher temperatures, thermal degradation is extensive. The thermal degradation can be counteracted as in this study by pretreatment with sodium sulfite.

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