TENSILE PROPERTIES OF SPRUCE UNDER DIFFERENT CONDITIONS[1]

Zoltan Koran

Professor, Université du Québec à Trois-Rivières, C.P. 500,
Trois-Rivières, Québec, Canada G9A 5H7

ABSTRACT

The mechanical properties of spruce were studied at various temperatures ranging from −190 C to +250 C. Diagrams are presented to show how maximum tensile stress, strain, modulus of elasticity, and work-to-maximum load changes as a function of temperature. The fracture energy reaches a maximum value around −40 C and is reduced to a low level at 150 C and over. Tensile stress increases in a linear manner with a decrease in temperature reaching a theoretical maximum at zero Kelvin degree.

Keywords: Defibration, separation energy, subnormal temperature, strength, thermal degradation, thermal softening, lignin, hemicellulose.

INTRODUCTION

The tensile properties of wood have been the subject of numerous studies, mostly from the point of view of its performance as a structural material. A number of these investigations examine wood strength as a function of such variables as moisture content and temperature (Comben 1964; Greenhill 1936; Kollmann 1940; Stanley and Rees 1938; Youngs 1957).

During the past few decades a new generation of research work has evolved on the strength properties of wood and on its failure mechanisms from the point of view of mechanical defibration. The ultimate objectives have been to find the specific conditions under which wood could be defibered mechanically into suitable paper-making fibers at high yield with a minimum amount of energy consumption.

It is with these considerations in mind that Atack et al. (1961) undertook a fundamental investigation on the cleavage and tensile failure of spruce. The present study follows the same line of thinking; it is intended to determine the fracture energy of spruce under controlled experimental conditions in a temperature interval ranging from −190 C to +250 C and involving pure tensile forces acting in the radial and tangential directions.

Some investigators went a step further and measured the energy consumption in the mechanical pulping processes (Lamb 1960 and 1962). Other researchers subjected wood to a slight chemical treatment prior to tensile fracture in order to reduce energy consumption in mechanical defibration (Stone 1955; Lagergen et al. 1957). Presently, this approach is the subject of a number of studies, mostly in industrial research laboratories.

Another group of investigators accomplished the same goal by an initial heat treatment of the wood, thus taking advantage of the purely physical phenomenon

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of the plasticization of the lignin binder in wood (Goring 1963). This approach has long been employed by the Masonite (Boehm 1944) and Asplund (1953) processes, and at present the commercial thermomechanical pulping processes are based on the same principle.

The characterization of the various types of mechanical pulps and the prediction of their paper-making properties led to another series of investigations. At the beginning it was necessary to develop suitable methods of pulp evaluation and define pulp properties by a series of well-defined parameters, such as fiber length, specific surface, coarseness, etc. (Forgacs 1963 and 1967; Atack 1972; Koran et al. 1978).

Soon it was realized that the surface structure of the fiber is another important parameter in pulp characterization, especially from the point of view of fiber bonding in paper. A series of microscopic studies gave detailed accounts of the significance of this parameter as a function of separation temperature (Koran, 1967, 1968, 1970, 1978; Atack 1972).

The present article is intended to complement the latter series of studies on the energetics of tensile failure. The specific objective is to determine the fracture energy of wood as a function of testing temperature. It is hoped that the energy versus temperature curve obtained under highly controlled experimental conditions can be useful in the prediction of energy consumption in thermomechanical pulping.

MATERIALS AND METHODS

A 71-year-old green black spruce (*Picea mariana* [Mill.] B.S.P.) tree, was used in this investigation. This tree was 15 inches in diameter and was free from any visible defects.

Eight sample rods (A_1, A_2, B_1, B_2, C_1, C_2, D_1, D_2), 30 inches long and 3×3 inches in cross section, were cut from the log. The rods were well matched both longitudinally and tangentially, each including the same annual rings (Fig. 1).

Ten standard test specimens were prepared from each rod for the tension perpendicular-to-grain tests in accordance to the ASTM standard (1964). In every sample rod the test specimens were numbered from the top to the bottom from 1 to 10. (Fig. 1). All samples had a uniform growth rate of 9 rings per inch and an average specific gravity of 0.34.

Half of the test specimens were kept in the green condition, while the remaining half were impregnated with glycerine. Prior to the tensile tests, the specimens were preheated or precooled to the following temperatures: -190 C in liquid nitrogen, -60 C in the mixture of dry ice and acetone, 0 C in melting ice. The temperatures between 0 C and 250 C were achieved by heating the specimens in water and glycerine. In each case, a 2-h preconditioning period was employed. All specimens to be tested below 0 C were maintained in the green condition.

The tensile tests were performed on an Instron tensile tester, which was equipped with a built-in conditioning chamber (Fig. 2) enclosing the jaws, and the sample. The conditioning chamber included a heating element, a stirrer, and a thermometer, and was filled with water or glycerine to assure that the tensile tests were carried out at the desired temperature. Glycerine was selected because it possesses a high boiling point (290 C) and has similar physical properties to

1

11TH ANNUAL RING

B   C

A   33RD ANNUAL
         RING

D

X

2

A_1-1	B_1-1	C_1-1	D_1-1
A_1-2	B_1-2	C_1-2	D_1-2
A_1-10	B_1-10	C_1-10	D_1-10
A_2-1	B_2-1	C_2-1	D_2-1
A_2-2	B_2-2	C_2-2	D_2-2
A_2-10	B_2-10	C_2-10	D_2-10

$2\frac{1}{2}'$

$2\frac{1}{2}'$

3

$2\frac{1}{2}"$

$2"$

11TH ANNUAL RING

$\frac{1}{2}"$

$1"$

$\frac{1}{2}"$

R

T   X

$\frac{1}{2}$

$\frac{1}{4}"$

$A_1 - 1$

T

R   X

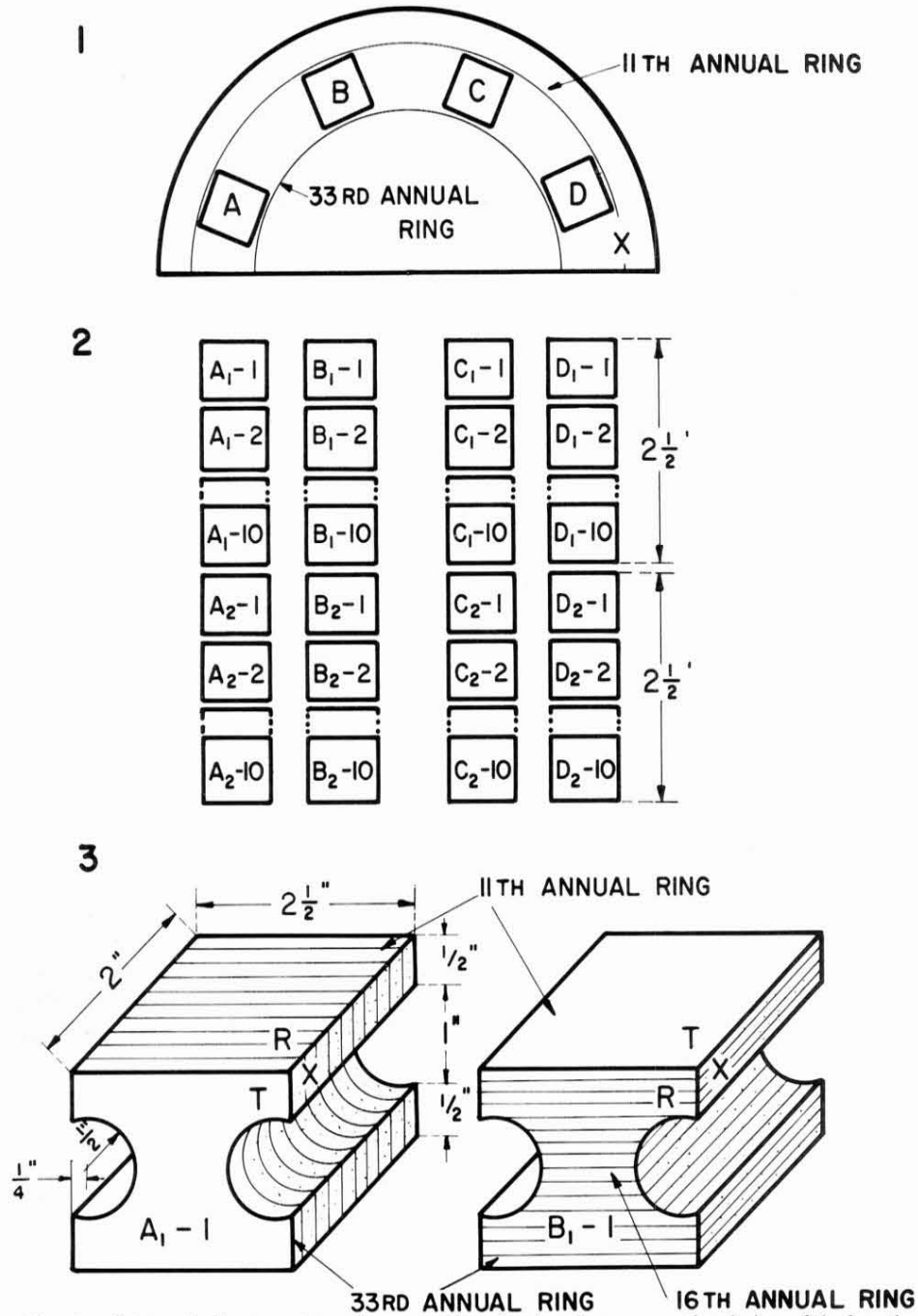$B_1 - 1$

33RD ANNUAL RING   16TH ANNUAL RING

FIG. 1. Schematic diagram of sample preparation showing the cross-sectional view of the log, the sample rods, and the test specimens.
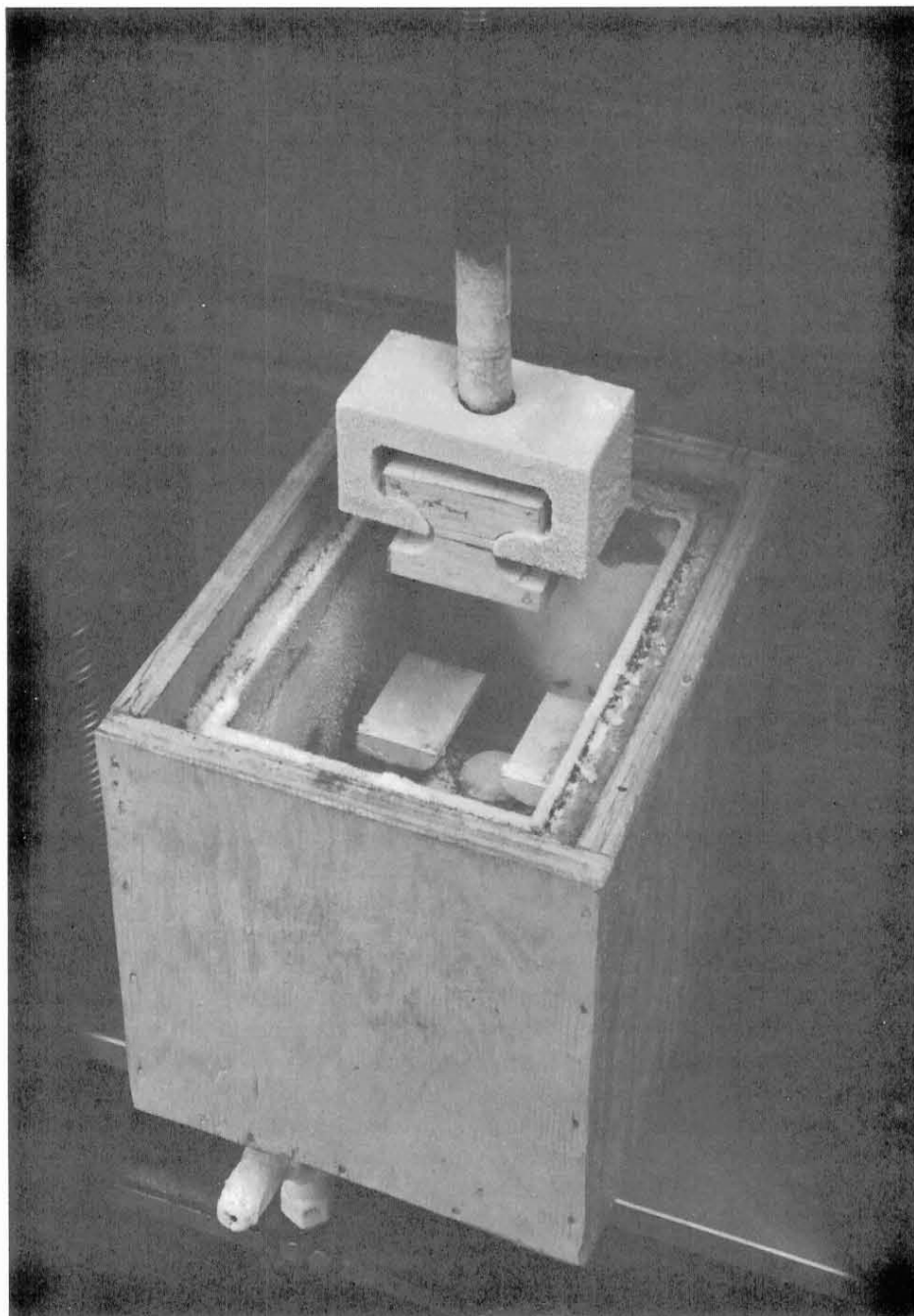
FIG. 2.   A photograph of the conditioning chamber enclosing the jaws of the Instron tensile tester.

water. Thus the use of glycerine enabled tensile testing above the boiling point of water, without the development of a rather complicated pressurized conditioning chamber to enclose the jaws of the tensile tester.

Below 100 C temperature, tensile tests were carried out separately in water

and glycerine in order to check out the effects of these two liquids on the mechanical properties of wood. Before each test the liquid in the conditioning chamber was heated or cooled to the desired temperature. Then the preconditioned test sample was placed in the jaws, which were completely immersed in liquid, and the load was applied at a crosshead speed of 0.25 cm per min. The tensile stress, strain, modulus of elasticity, and work-to-maximum load values were determined individually for each test specimen.

RESULTS AND DISCUSSION

Maximum tensile stress

The maximum tensile stress values (σ) are plotted in Fig. 3 for both radial and tangential fracture as a function of temperature. The average values of all points are plotted in Fig. 6. These figures show that below about 150 C the tensile strength of wood increases in a linear fashion with a decrease in temperature. An extrapolation of the straight line to 0 K yields an average tensile value of 56.4 kg/cm², which is 2.56 times higher than the corresponding value (22 kg/cm²) at 0 C (273 K).

This inverse linear relationship can be expressed with the following equation:

$$\sigma_M = a - bT \quad \text{(Fig. 6)}$$

where σ_M = maximum tensile stress in kg/cm²
      T = temperature in °K.
      a = the average intercept on the stress axis = 56.4 kg/cm².
      b = the average slope of the straight line = 1,244 kg/cm² °K.

However, the above straight line relationship holds only up to about 150 C, beyond which the change becomes more gradual approaching the temperature axis asymptotically.

It should be noted that at subnormal temperatures (−60 C and −190 C) several of the test specimens split partially or completely during the cooling period prior to tensile testing. In fact, at −190 C testing temperature, only sample D_1 (Fig. 3) was free from internal checking; the others possessed checks and splits of various forms and sizes. The presence of these defects would account for the low tensile stress values obtained (Fig. 3) at this temperature. It is believed that the newly formed checks and splits developed because of a rapid cooling technique, resulting in unequal thermal contraction and a nonuniform increase in the volume of the test specimen upon ice formation. In the case of future studies, a more gradual cooling method is recommended in order to confirm the linear increase in maximum tensile stress as it approaches 0 K.

Strain

Figure 6 reveals that in perpendicular-to-grain fracture the strain increases with an increase in temperature in a linear manner in the temperature range of −183 C to about 300 C. According to the kinetic theory, there is no random molecular motion at 0 K temperature. Therefore, an extrapolation of the straight line to absolute zero (0 K) is expected to pass through the origin. Under these conditions, the straight line portion of Fig. 6 can be represented by the following linear equation:

$$\delta = \frac{\Delta l}{l} = \frac{C}{l} T,$$

where Δl = actual extension in cm

C = slope of the straight line in cm/°K

T = temperature in °K

δ = strain at maximum load in cm/cm

l = length of the sample in cm

The strain-temperature curve remains linear up to about 25 C (348 K), at which point it begins to level off, reaching a maximum value at about 125 C. It is interesting to note that the maximum rate of decrease in fracture energy corresponds to the same temperature range (from 25 to 125 C; Fig. 6). This is the temperature interval in which the hemicellulose and lignin fractions of wood undergo a progressive and cumulative softening process, reaching their optimum plastic state in the neighborhood of 125 C.

Above 125 C, the continuing softening effect is canceled out by a progressively increasing degree of thermal degradation due to hydrolysis by acetic acid and formic acids (Koran et al. 1978). This phenomenon would account for the increasingly high rate of decrease in strain above 125 C.

Figure 6 further reveals that the strain values of wood conditioned and tested in water are consistently higher than those of glycerine. This suggests that water swells wood to a higher degree than does glycerine.

It was also noted that strain in tangential failure is on the average 10% higher than that in radial fracture. This can be accounted for by the lower degree of structural organization in the tangential plane than in the radial plane. It stands to reason that the radially inserted rays and the radial rows of tracheids cause a slight degree of weakening in that plane, thus reducing the tensile stress and the strain values proportionally.

Modulus of elasticity

The modulus of elasticity (MOE in kg/cm²) is defined as the ratio between stress (σ) and strain (δ) within the elastic limit of the material. For wood, in tension perpendicular-to-the-grain, the plastic region is practically negligible; therefore the stress at the yield point is almost the same as the ultimate strength. Thus the modulus of elasticity can be calculated from the stress and strain at maximum load.

$$MOE = \frac{\sigma}{\delta} = \frac{(a - bT)l}{CT}.$$

This equation gives a hyperbolic relationship between the modulus of elasticity and temperature. Figure 3 reveals a family of concave curves that can be shown to be hyperbolic by suitable comparison.

The small slope at high temperatures (100 C and above) indicates the highly plastic behavior of wood in that region. In contrast, the rapid change in MOE below 0 C suggests that wood becomes extremely rigid at low temperatures.

The predominant increase in brittleness is partially due to the reinforcing effect of the ice lattice acting throughout the cell-wall structure, the stiffening of the
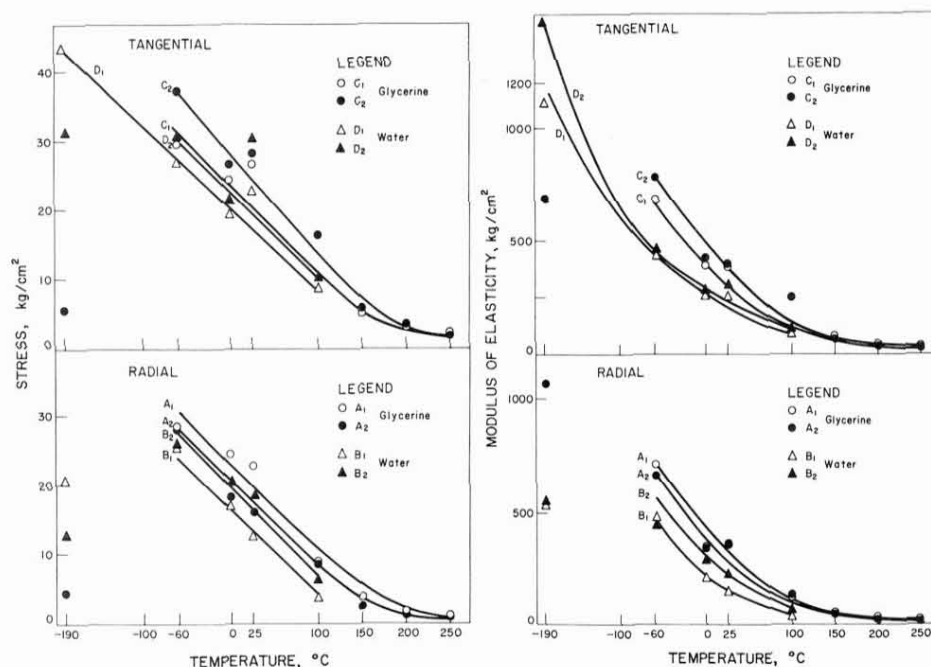
FIG. 3. Perpendicular-to-grain maximum tensile stress and the modulus of elasticity as a function of temperature.

cell-wall substance itself, and to an increase in the cohesive forces as a result of thermal contraction. The increased brittleness, caused by the lowering of temperature, was indicated by the decrease in deformation and by the increasingly abrupt failure, both observed from the load-deformation diagrams.

It is very difficult to determine accurately the overall effect of subnormal temperatures on the modulus of elasticity of wood because of the interactions of a number of variables simultaneously acting in a highly complex system. For example, the stiffening effect of ice in wood does not reach its full measure until all of the water is frozen within the cell wall. Since the water in the cell wall is held in capillaries of variable sizes and shapes, its freezing will not occur uniformly at 0 C or even below. Furthermore, some solute content, such as the sugars and starches, may also cause a depression in the freezing point of water. Water held in larger capillaries will probably freeze somewhat below 0 C, but it is known that the bound water will not freeze until the temperature drops well below 0 F (−18 C). This is due to the relatively high pressure sustained by bound water.

An additional factor that may complicate the above phenomenon is that during the tensile tests part of the mechanical energy supplied to wood is released in form of heat energy. The latter would tend to melt the ice and eliminate some of the stiffening effect of ice, unless the temperature were sufficiently low to overcome melting. However, by the time wood reaches −40 C, most likely all of the water contained in the cell wall, including the bound water, is transformed into ice and the melting effect caused by the heat release during the tensile test is probably overcome by the sufficiently low temperature.
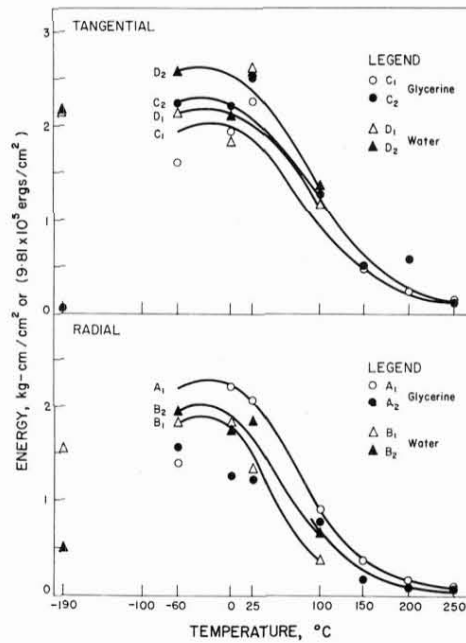
FIG. 4. Fracture energy as a function of temperature.

Fracture energy

The work-to-maximum load in perpendicular-to-grain tensile failure is the measure of the energy consumed in the fracture of the wood specimen. Therefore, the work-to-maximum load is named as "fracture energy," or simply "energy" measured in kg-cm. These values were determined from the load-deformation curves for the 2-square-inch fracture surface and were converted to the kg-cm/cm² basis by dividing by 11.9. These values were then plotted in Figs. 4 and 6 as a function of the testing temperature.

Figure 4 reveals that the energy absorbed in tangential fracture (E_T) is consistently higher than that of radial failure (E_R). At 25 C temperature, for example, $E_T = 1.8E_R$. This difference can partially be attributed to the weakening effect of rays and to the high degree of uniformity in the radial arrangement of tracheids. While in the radial direction the tracheids are arranged in distinctly uniform radial rows, tangentially such a systematic order does not exist.

Figure 4 also shows that the fracture energy of water-swollen samples does not differ consistently from that of glycerine-swollen specimens. This is in spite of the fact that the elongations of the water-swollen samples were consistently higher than those of the glycerine-swollen specimens (Fig. 6). At 25 C, for example $\delta_w = 1.38\delta_G$.

Figure 6 shows the overall change in fracture energy as a function of fracture temperature. It is evident that the fracture energy consumed in tensile failure reaches a maximum in the neighborhood of −40 C and decreases in a nonlinear fashion on either side of this peak. The fracture energy drops from 2.2 kg-cm/cm² at −40 C to a theoretical value of zero at −273 C over a temperature span of
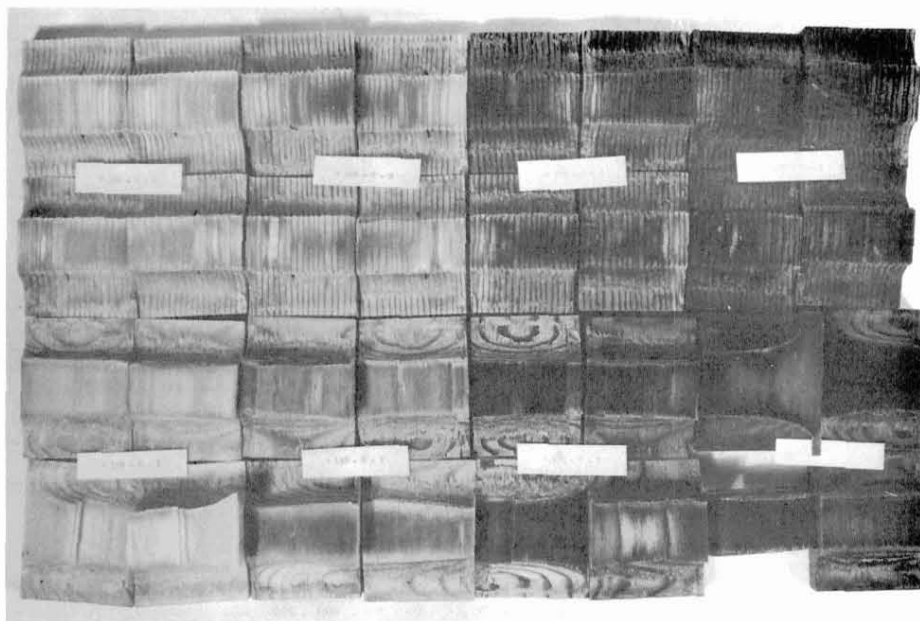
FIG. 5.   Perpendicular-to-grain tensile test specimens fractured at 100, 150, 200 and 250 C.

233 C. The same temperature span corresponds to a similar decrease in fracture energy (2.0 kg-cm/cm^2) on the high temperature side of the −40 C peak. This implies that the two descending parts of this curve on the two sides of the 40 C peak are nearly symmetric.

This diagram further shows that the rate of energy decrease per degree C varies as a function of temperature. Thus, the rate of energy drop increases gradually from −40 C to about 0 C, followed by a zone of high rate decrease in the temperature interval of 0 C to 150 C. Above 150 C the energy decrease levels off very rapidly. The maximum rate of decrease is situated at about 75 C. The high rate of decrease in fracture energy in the temperature interval of 25 C to 125 C can be attributed to the combined cumulative softening effect of the hemicellulose and lignin. According to Goring (1963), the hemicellulose and lignin begin to soften around 50 C and 90 C, respectively, and wet spruce sawdust that contains native lignin and hemicelluloses produces maximum bond strength at 108 C.

At 150 C the wood specimens became light brown in color, indicating a high degree of thermal degradation (Fig. 5). According to Stamm (1956), wood is subjected to a 1.25% weight loss, as a result of a 2-h heat treatment at 150 C. Both the color change and the weight loss indicate a high degree of thermal degradation. This would tend to cancel out the continuing plasticizing effect of heat on wood and thus account for the rapid leveling off of the energy curve beyond 150 C.

At +200 C the wood became dark brown in color, giving the appearance of partially burned wood (Fig. 5) and the weight loss reaches a relatively high level (9%). In the temperature interval of +200 to +250 C there is only a small reduction in fracture energy, but a considerable increase in weight loss (40%) and a very severe change in wood color. All of these indicate a severe degree of thermal
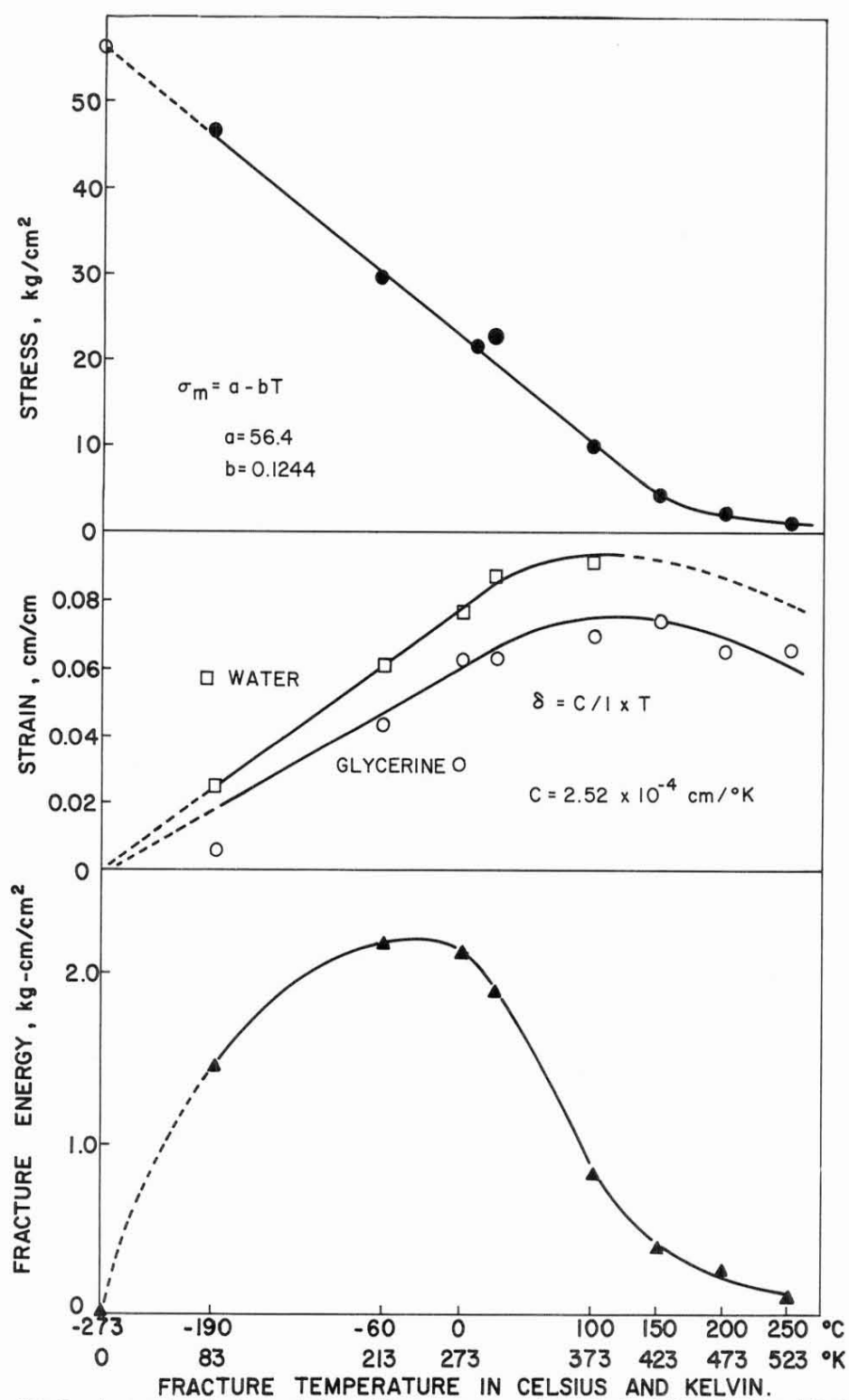
FIG. 6. Average tensile stress, strain and fracture energy values calculated from the data plotted in Figs. 3 and 4.

degradation. At 250 C, the summerwood already possesses the appearance of charcoal, although the springwood maintains a somewhat lighter color (Fig. 5). At this temperature the cellulose portion of wood may also become softened, as the glass transition point of isolated cellulose, determined by the powder collapse technique, was found to fall between 231 and 253 C temperature (Stamm 1956).

CONCLUSIONS

A gradual increase in temperature from −190 C to about 100 C results in a linear decrease in tensile stress and a corresponding linear increase in strain. These data reveal that spruce is 14 times weaker at 150 C (423 K) than at the hypothetical temperature of 0 K.

In comparison, the fracture energy changes in a highly characteristic manner as a function of testing temperature (Fig. 6). It increases rapidly from −190 C upwards, reaching a maximum at about −40 C followed by a rapid decrease up to about 125 C and a leveling off between 125 C and 250 C. As expected, the maximum rate of decrease in fracture energy corresponds to the thermal softening range of the hemicelluloses and lignin (from about 25 C to 125 C). The strain value of wood tested in water is considerably higher (32%) than that of glycerine. However, the effect of the heating medium on maximum tensile stress seems to be canceled out by specimen to specimen variations.

These results have significant implications in the field of industrial wood utilization under subnormal temperatures and in high-temperature applications. Another important application of this research falls within the field of thermomechanical pulping. It is expected that the relationship between fracture energy and temperature holds in thermomechanical defibration as well.

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