

# Effect of Temperature on Fracture of Spruce in Compression, Investigated by Use of Acoustic Emission Monitoring

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*Acoustic emission (AE) monitoring during compression of wood has been used to investigate the fracture history with specific emphasis on its dependence on temperature, moisture content, strain and loading direction. The wood was compressed in both the lateral and longitudinal directions in order to select preferred modes of deformation to achieve desired irreversible changes in the wood structure. The elastic modulus, the compressive strength and the cumulated number of AE events decreased with increasing temperature. It was concluded that the most efficient loading direction is longitudinal in order to introduce flaws in wood under compression and that a longitudinal compression of 24%, corresponding to a specific energy input of 3 kWh/t, is needed in order to achieve substantial changes in the wood structure. The compression should be carried out at temperatures well below 120°C in order to introduce many failure sites.*

*Les ondes acoustiques ont été examinées pendant la compression du bois afin de déterminer l'origine des fissures et plus particulièrement pour évaluer l'importance de celles-ci sur la température, le degré d'humidité, la contrainte et le sens du chargement. Le bois a été comprimé dans le sens latéral et le sens longitudinal, afin de choisir le mode de déformation permettant d'obtenir les modifications internes irréversibles désirées dans la structure du bois. Le module élastique, la résistance à la compression et la fréquence des émissions acoustiques ont diminué lorsque la température s'accroissait. Nous avons conclu que le sens de chargement longitudinal entraîne davantage de fissures dans le bois faisant l'objet de compression, et qu'une compression longitudinale de 24 % correspondant à une énergie spécifique de 3 kWh/t est nécessaire pour modifier la structure du bois de façon substantielle. La compression doit être faite à des températures très inférieures à 120 °C pour que le bois présente des défaillances en plusieurs endroits.*

## INTRODUCTION

In the mechanical pulping process the chips are deformed even before refining. The chips are compressed both in chipping and in feeding the chips into the refiner. It is known that, during mechanical loading, wood exhibits a number of different deformation and failure phenomena, e.g. plastic/viscoelastic deformation of the fibres, breaking of fibres, delamination of the cell walls, fibre fracture and the introduction of microcracks in the fibre walls [1-3].

Frazier and Williams [4] precompressed wood cubes longitudinally to 35% of their original thickness and thereby reduced the required refining energy by 9% for thermomechanical pulp and by as much

as 40% for chemithermomechanical pulp.

From photographs they discovered that nearly every fibre was separated from the adjacent fibres by the buckling and splitting that occurred and that the separation between fibres occurred at the interface of the outer and middle layer of the secondary fibre wall, leaving the middle lamella largely intact and attached loosely to one fibre or the other.

Numerical simulations [5] showed that a wood chipper produces chips with large parts of plastic deformation. A higher knife angle or a higher friction between wood and knife increased the parts of plastic deformation.

Hartler [6] found that longitudinal compression of fibres during chipping produces misalignments of the microfibrils in the middle layer of the secondary wall,  $S_2$ . Because of the birefringent nature of the microfibril, these deformations show up bright against a dark background when the

fibre is examined in polarized light with crossed polaroids and the fibre axis oriented in the extinction position.

A major contribution to our present understanding of failure in wood due to longitudinal compression was made by Dinwoodie [7,8]. He observed that small dislocations of microfibrils in the cell wall, slip planes, progressed to grow to microscopic creases and successively to larger creases, up to failure with increasing load. It was concluded that this process contributes considerably to the plastic deformation of wood compressed parallel to the grain. The appearance of dislocations were more likely to be found at parts of the tracheid which are in contact with rays. The angle at which the slip plane traverses the cell wall was found to be a function of the angle of the microfibrils in the middle layer of the secondary wall together with the ratio of the modulus of elasticity in the longitudinal and radial planes.

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Kollmann [9] speculated that, due to the viscoplastic properties of lignin, stresses of very short and very long duration initiate cracks in the middle lamella. An estimation of the time where the influence of plasticity was minimum was in the order of 10 s.

Acoustic emission (AE) is a phenomenon in which stress waves are emitted from a rapid, localized change of strain energy in a material, meaning, for example, that flaws which are created in the material can be detected by AE. By detecting the AE with a sensor and amplifying the signal, it is possible to define an AE event by its energy content, peak amplitude, duration, etc.

AE monitoring during compressive longitudinal loading of wood was first used by DeBaise et al. [10]. However they observed very low emission densities, 0.001–0.02 events/cm<sup>3</sup> at failure in compression compared to approximately 10 000 events/cm<sup>3</sup> in tension. This result confirmed their expectation that compressive load was closing intrinsic flaws in the material. A more thorough introduction to and literature review of AE on wood can be found in a paper by Ansell [11].

In the present work, AE monitoring is carried out during compression of wood to investigate the fracture history with specific emphasis on its dependence on temperature, moisture content, strain and loading direction. The wood is compressed in both the lateral and longitudinal directions in order to select preferred modes of deformation to achieve desired irreversible changes in the wood structure.

## EXPERIMENTAL

Samples were prepared from a freshly cut 30-year-old log of Norway spruce, *Picea abies*, with an annual radial growth rate of 2.2 mm in the sapwood zone. The diameter of the bark-free log was 0.16 m. Slices approximately 20 mm thick were cut in the longitudinal direction and sample specimens with the dimensions 20 × 20 mm in the tangential and radial directions were taken out from the sapwood of these slices at the same radial location. The average dry content, in the green condition, was 50%; and the average density, based on dry samples, was 310 kg/m<sup>3</sup>.

The compression tests were performed using a 200 kN Instron testing machine in the first experimental set part A. In parts B and C, a 50 kN MTS testing machine, equipped with an environmental chamber, was used.

In order to distribute the force over the end surfaces, pieces of a rubber cloth, with a measured E-modulus of 1090 MPa, were inserted at the top and bottom of the sample specimens. The E-modulus for the rubber cloth was evaluated at approximately the same stress as in the measurements on the wood.

The average strain,  $\epsilon_0$ , in the samples was calculated from the displacement of the crosshead and compensated for the defor-

mation of the rubber cloth.

The samples were positioned in the testing machine and an acoustic sensor was attached to the wood sample, as shown in Fig. 1.

The acoustic emission from the samples during compression was analyzed by the procedure and using the equipment described in the Appendix.

The first set of experiments, part A, was performed at room temperature with the wood samples in green condition, i.e. at a solids content of about 50%.

In the second set of experiments, part B, both temperature and solids content were varied. The impregnated samples were presteamed at atmospheric pressure for 15 min followed by 10 min impregnation in water or glycerol at a pressure of 500 kPa.

Glycerol was selected because it possesses a high boiling point (290°C) and has physical properties similar to those of water [12]. Thus the use of glycerol enabled testing above the boiling point of water, without the need to develop a rather complicated pressurized conditioning chamber. Below 100°C, tests were carried out separately in water and glycerol in order to establish the effects of these two liquids on the mechanical properties of the wood.

The solids content for the glycerol-impregnated samples was calculated with a correction for the difference in liquid den-

sities and with the assumption that all water was replaced with glycerol (density 1270 kg/m<sup>3</sup>).

All samples and the AE sensor were immersed in glycerol during the compression. The water-impregnated samples were also immersed in glycerol to keep the acoustic coupling between sample and AE sensor constant.

In the third set of experiments, part C, the sample orientation was varied. Samples were compressed not only in the longitudinal direction but also in the radial and tangential directions. All samples and the AE sensor were immersed in glycerol during the compression.

Parameter variation and conditions held constant, in experiments parts A, B and C, are summarized in Table I.

## RESULTS

### Part A

Typical curves from one of the compression tests are shown in Fig. 2.

The compressive stress and the cumulative AE events are both plotted vs time, which is easily translated to displacement as the crosshead speed is constant at 0.1 mm/s. The maximum stress of 13.4 MPa is achieved at about 4% compression and remains relatively constant up to about 20% strain where the sample collapses. It is at this point that substantial AE is achieved. The unloading

TABLE I  
PARAMETER VARIATION AND CONDITIONS HELD CONSTANT

	Part A	Part B	Part C
Solids content	50%	32–75%	32–81%
Deformation direction	longitudinal	longitudinal	longitudinal, radial or tangential
Temperature	22°C	23–180°C	23°C
Crosshead speed	75 µm/s at strains >30% 100 µm/s at strains <30%	100 µm/s	100 µm/s
Impregnation	no	water or glycerol	water
Strain	0–36%	20%	30%

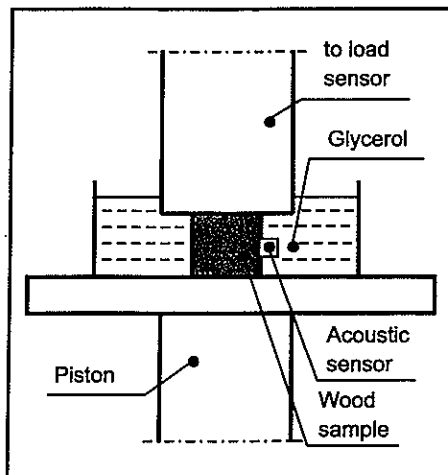


Fig. 1. Schematic drawing of the experimental set-up showing the testing machine and the acoustic sensor.

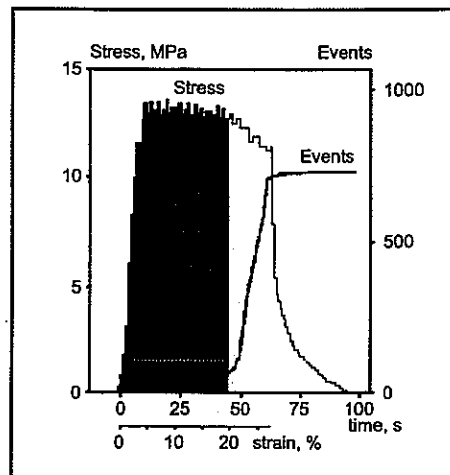


Fig. 2. Average compressive stress and cumulative emissions vs time.

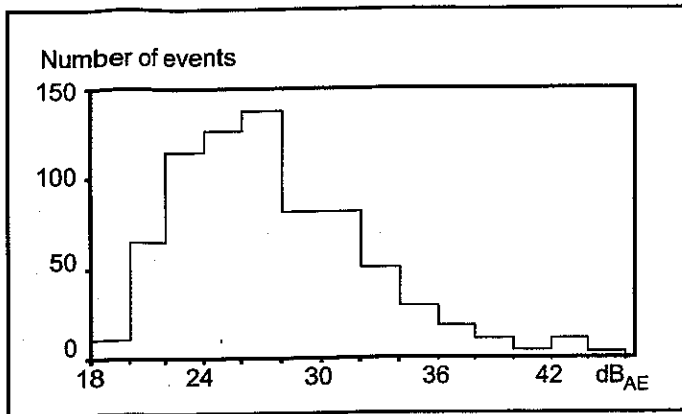


Fig. 3. Distribution of number of events by peak amplitude.

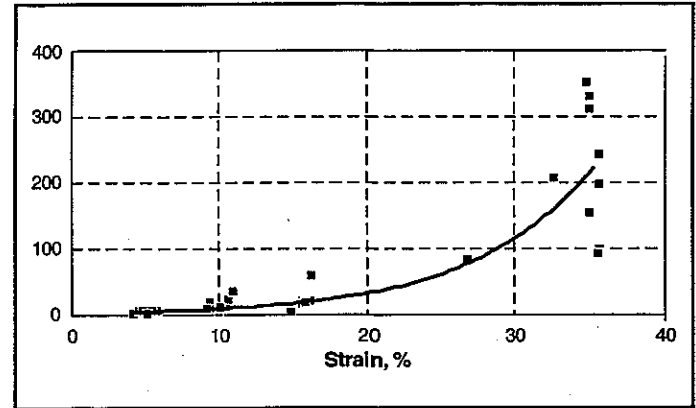


Fig. 4. Events vs total average strain at an average solids content of 49%.

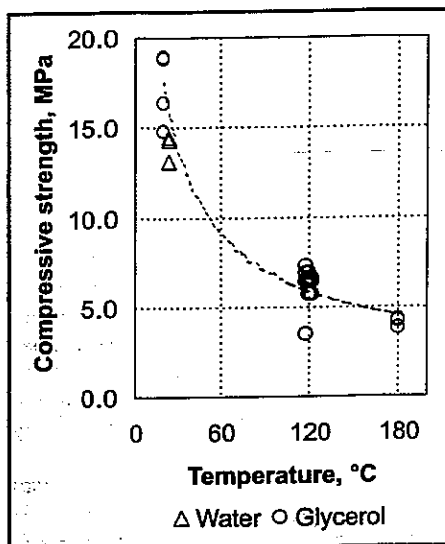


Fig. 5. Compressive strength vs temperature.

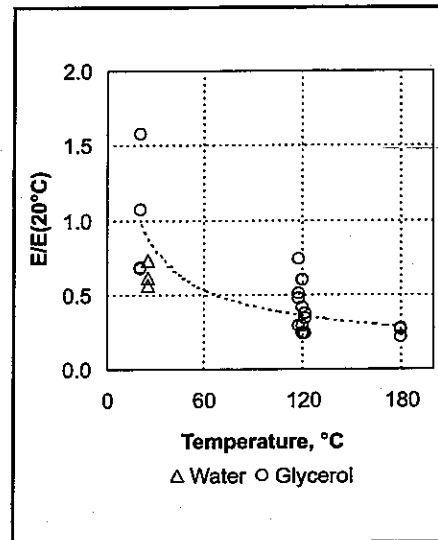


Fig. 6. Relative E-modulus vs temperature.

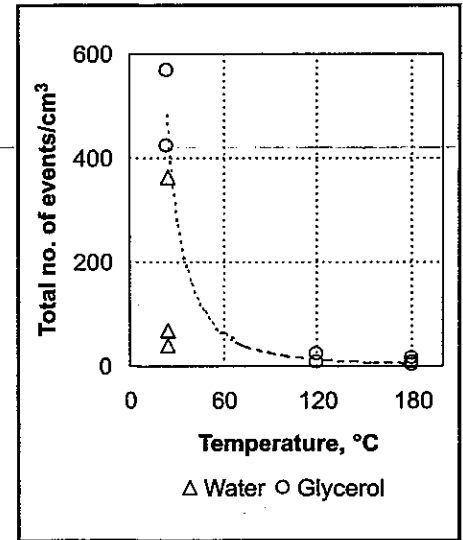


Fig. 7. Total number of events vs temperature at a total strain level of 20%.

starts at an average strain of 27% where the cumulative AE reaches 720 events. As can be seen, it is almost silent during unloading. The reason is that generation of AE occurs during plastic deformation and must intrinsically arise from dynamic microcracking. The shadowed area is proportional to the compressive work done before onset of substantial AE. A similar behaviour is also found in deformation of steel (e.g. see [13]).

Values based on nine tests at room temperature and at 50% solids content show that a longitudinal compression of 24% and an energy input of 3 kWh/t is needed in order to achieve substantial AE.

A typical distribution of number of events by peak amplitude is shown in Fig. 3. A maximum is found at 26–28 dB<sub>AE</sub> and decreases exponentially at higher amplitudes.

There are almost no counts above 46 dB<sub>AE</sub>. This exponential decline can be described by a constant *b*, which is characteristic of a certain type of failure. The constant *b* is defined as the negative slope of the distribution of number of events by peak amplitude in a log-log plot [14].

The nine samples subjected to 35% compression were averaged with *b* calculated to be 1.36 and the peak amplitudes

showing maxima at 26.3 dB<sub>AE</sub>. The unit dB<sub>AE</sub> is defined in the Appendix.

The measured cumulative emission densities vs total average strain for a number of compression tests are given in Fig. 4. The mean value for the nine samples, subjected to about 35% compression, is 220 events/cm<sup>3</sup>.

The measured data are summarized as mean values and standard deviations in Table II.

### Part B

The measured compressive strength vs temperature is shown in Fig. 5. The mean value of the compressive strength is 17 MPa for the glycerol-impregnated samples at room temperature. The strength decreases to 6.2 MPa at 120°C, and a further increase in temperature to 180°C results in a compressive strength of 4.0 MPa. At room temperature, the corre-

sponding value for the water-impregnated samples is about 3.3 MPa lower.

The measured relative E-modulus vs temperature is shown in Fig. 6. The mean value of the E-modulus is set to 1.0 for the glycerol-impregnated samples at 23°C. The relative E-modulus decreases to 0.41 at 120°C, and a further increase in temperature to 180°C results in a relative E-modulus of 0.25. At 23°C the corresponding value for the water-impregnated samples is 0.63.

Figure 7 shows the total cumulated acoustic events vs temperature. For the glycerol-impregnated samples, the total number of events at the 20% strain level decreases from 500 to 20 events/cm<sup>3</sup>, when

TABLE II MEASURED DATA FROM EXPERIMENTS, PART A			
	Mean value	Standard deviation	
Solids content, %	49	0.1	
Density, kg/m <sup>3</sup>	1530	100	
Maximum stress, MPa	14.7	0.6	
Events/cm <sup>3</sup> , 35% strain	220	30	
Strain to substantial AE, %	24.4	3.8	
Work to substantial AE, kWh/t	3.1	0.5	
Peak, dB <sub>AE</sub>	26.3	0.19	

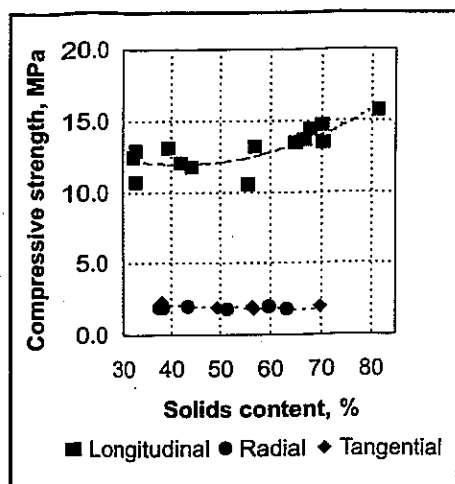


Fig. 8. Compressive strength vs solids content.

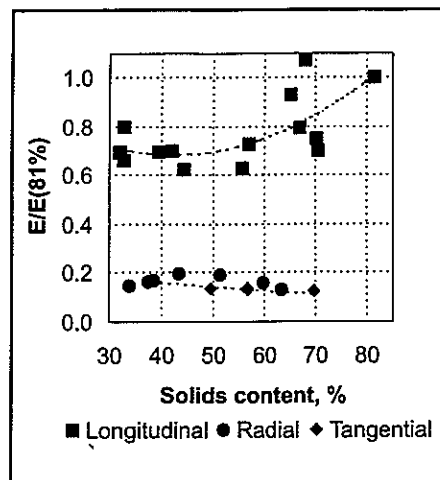


Fig. 9. Relative E-modulus vs solids content.

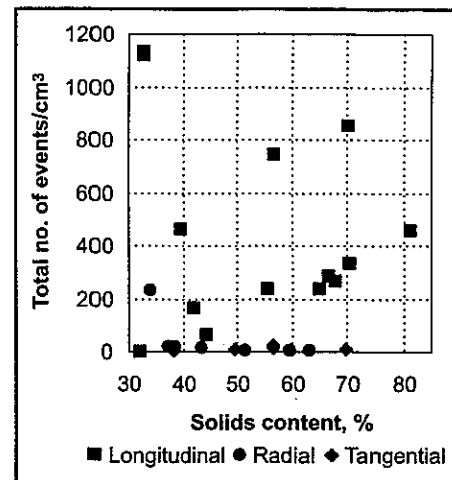


Fig. 10. Total number of events vs solids content at a total strain level of 33%.

the temperature was increased from 23 to 120°C. A further increase in temperature to 180°C results in about 8 events/cm<sup>3</sup>. For water-impregnated samples, the average total number of events is 200 events/cm<sup>3</sup>.

### Part C

The measured compressive strength is shown in Fig. 8. The mean value for the longitudinal compressive strength is 12 MPa up to 60% solids content where it starts to increase. In both the radial and tangential directions it is 2.0 MPa.

The relative E-modulus shows a similar behaviour (Fig. 9). The mean value of the E-modulus is set to 1.0 for the longitudinal compression at 81%. The mean value for the longitudinal relative E-modulus is 0.7 up to 60% solids content where it starts to increase. It is about 0.2 in the transverse directions.

As can be seen in Fig. 10, there are more AE counts when the samples are subjected to longitudinal compression, 600 events/cm<sup>3</sup> as a mean value, compared to radial and tangential, 30 events/cm<sup>3</sup>.

### DISCUSSION

SEM photographs of the compressed samples (see Fig. 11) show many cracks in the RL and the TL planes. The cracks are introduced mainly in the upper and lower parts of the specimen, i.e. end crushing [7,15].

Figure 4 shows that a major part of the AE events occurs at a strain level above 35%. This strain can be compared to 65% precompression, which was used by Frazier and Williams [4], and resulted in 9% energy saving in the refining of TMP.

The reason DeBaise et al. [10] observed very low emission densities, 0.001–0.02 events/cm<sup>3</sup> compared with about 200 events/cm<sup>3</sup> at failure in compression, was probably due to compressions well below 20% together with a higher threshold value for AE counts and/or a less sensitive AE sensor.

In Figs. 8 and 9, compressive strength and relative E-modulus are plotted vs solids content. As can be seen there is no effect on strength and E-modulus below about 60% solids content in any of the plotted data series. Dinwoodie [16] reported no effect on strength and E-modulus below 77% solids content which was said to be the fibre saturation point (FSP). Other researchers (e.g. [17]) have reported FSP to be at 80%. Therefore it is possible to calculate mean values regardless of solids content. However, it should be mentioned that FSP values based on modern solute exclusion technique are lower, about 68% [18].

Tests were carried out for both glycerol- and water-impregnated samples at room temperature. The water-impregnated samples show lower compressive strength and E-modulus compared to the glycerol-impregnated samples at room temperature. The differences were 20 and 40%, respectively (Figs. 5, 6). This suggests that water swells the wood to a higher degree than glycerol. The same differences was also found by Koran [12]. However, it should be noted that he loaded the samples perpendicular to the grain rather than parallel.

The softening effect of temperature is also shown in Figs. 5 and 6. At 180°C the compressive strength has decreased to 23% of its value at 23°C and the E-modulus to 25%. The corresponding values from compression experiments perpendicular to the grain, estimated from Koran's figures, are 20 and 18%, respectively.



Fig. 11. SEM photograph showing a top view of a sample compressed to an average strain of 35%. Photo by S. Palovaara, SCA.

The total number of events at 20% compression (Fig. 7) shows a stronger dependence on temperature than strength and E-modulus. An increase in temperature to 120°C decreased the total number of events to 3% of its value at 23°C. Also the difference between glycerol and water is more pronounced. Water-impregnated samples show 60% lower emission densities compared to glycerol at 23°C. Obviously, our experiments show that the compression should be carried out at temperatures well below 120°C in order to introduce many failure sites in the wood.

The work applied to the wood before substantial AE, at 24% compression, is 3.1 kWh/t according to Table II. These figures are in agreement with those of Frazier and Williams [4]. They concluded that optimal conditions for energy savings were 65% precompression at 20°C with an energy input of 5 to 10 kWh/t.

The factor *b* which describes the exponential decline of events by peak amplitude is almost the same, 1.36 in part A compared with 1.38 in part C. From SEM

photographs (see Fig. 11), it can be concluded that the type of failure that predominates is the separation of rows of fibres from adjacent rows of fibres. A similar failure mechanism was also found by Frazier and Williams [4]. For translaminar shear, before failure in a wood laminate,  $b$  was calculated to be 1.2 by Pollock [14].

As can be seen in Fig. 9, there are more AE counts when the samples are subjected to longitudinal compression, 600 events/cm<sup>3</sup> as a mean value, compared to 30 events/cm<sup>3</sup> in radial and tangential compression.

Thus, the most efficient loading direction is longitudinal in order to introduce flaws in wood under compression.

Wood is a viscoelastic material and the results are valid only at quasi-static conditions. In the present investigation the experiments were carried out at velocities of about 100  $\mu$ m/s, which is about five to six decades lower compared to those in industrial refineries. For water-saturated wood, it has been demonstrated that the elastic modulus follows the general time-temperature superposition principle which makes it possible to calculate the properties of the wood over a large velocity-temperature interval [19]. It is thus possible to predict the effects of higher velocities by shifting the temperature. This shift was found to be about -12°C and -8°C per decade for longitudinal and radial compression, respectively [20].

## CONCLUSIONS

In order to achieve substantial changes in the wood structure, it is crucial that:

- The wood be compressed in the longitudinal direction;
- The compression be carried out at temperatures well below 120°C; and
- The longitudinal compression should be carried out to 24%, corresponding to a specific energy input of 3 kWh/t.

What still remains is to describe the fracture surfaces, i.e. where in the cell wall the produced surfaces are located.

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## APPENDIX

Acoustic emission technology AET 5000B series equipment was used to transduce, amplify and process the detected signals. A sensor type MAC300L was used as acoustic transducer. The sensor has a resonant frequency of 300 kHz and has a sensitivity of better than -70 dB<sub>AE</sub> referred to 10 V/Pa. The unit dB<sub>AE</sub> is defined below.

An 160B preamplifier amplified the detected signals by 1000 $\times$  (60 dB<sub>AE</sub>) and contained a bandpass filter which passed frequencies between 250 and 500 kHz, which eliminates low-frequency noise and high-frequency overtones. The amplifier was set with a gain of 20 $\times$  (26 dB<sub>AE</sub>), giving a total gain of 20 000 $\times$  (86 dB<sub>AE</sub>).

The data then consisted of a variable-frequency, variable-amplitude voltage which was formed into discrete bursts of energy, each containing cycles of gradually decaying amplitude. Each rising cycle of 15  $\mu$ V (24 dB<sub>AE</sub>) or greater amplitude was counted by a digital counter which accepted amplified signals above a threshold of 0.30 V. The upper limit in measured amplitude was 77 dB<sub>AE</sub>. Output was passed to a computer. This output closely relates to the actual number of events taking place.

An amplified signal from a resonance frequency transducer (the most commonly used transducer) is shown in Fig. 12. When hit by a stress wave, the sensor will ring like a bell. Since the stress wave, on its way from the fracture site to the transducer, will be reflected, converted to other wave types, dispersed, suffer damping, etc. and, since the transducer has a transfer function dominated by its resonance frequency, it is in general impossible to say anything in detail

about the process which caused the recorded AE signal.

In Fig. 12 is defined what in AE terms is referred to as an event. The origin of such an event might be fibre bond failure, fibre rupture, external disturbances, etc. Some parameters that are used to describe an AE event are also defined in the figure.

To exclude the mechanical and electronic noise that, to some extent, always is present during AE-monitoring, a threshold value for the output voltage can be defined as in Fig. 12. This means that every signal with an amplitude less than this threshold will be discarded. The threshold value and the peak amplitude are most commonly given in dB<sub>AE</sub> defined as:

$$R [dB_{AE}] = 20 \log (V/V_{ref})$$

where  $R$  is the amplitude in [dB<sub>AE</sub>],  $V$  is the output voltage from the sensor and  $V_{ref}$  is a reference voltage at the sensor taken as 1  $\mu$ V.

Also shown in Fig. 12 is a dead time which is introduced to define separate events, i.e. the time the signal should be below the threshold value to define the next wave train as a separate event.

Parameters used to describe an AE event are, with reference to Fig. 12, duration, rise time and peak amplitude. Among these, the peak amplitude is the most frequently used.

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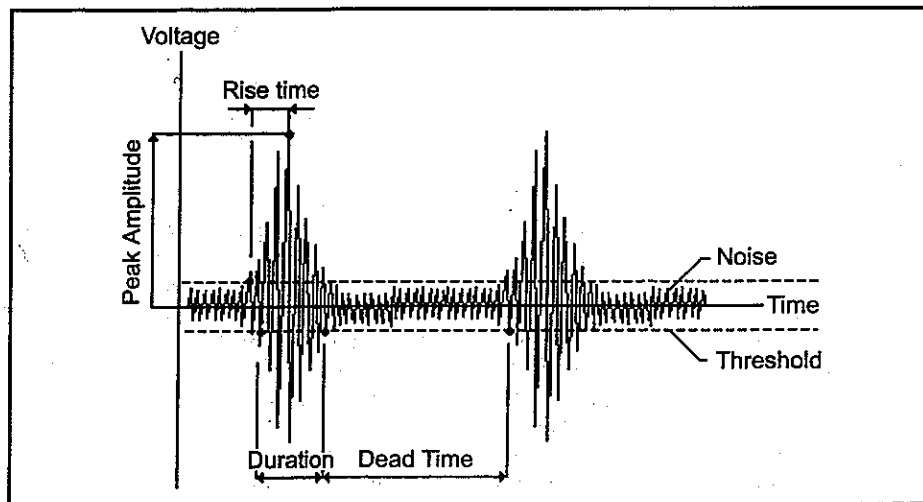


Fig. 12. AE parameters.

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#### TAPPI/ISA-PUPID Process Control, Electrical and Information Conf.

Mar. 25-29, 2001

San Antonio, TX, USA

#### Intl. Environmental Conf. & Exhibit

Apr. 22-25, 2001

Charlotte, NC, USA

#### TAPPI Advance Coating Fundamental Symp.

May 3-5, 2001

San Diego, CA, USA

#### 2001 Intl. Mechanical Pulping Conf., KCL, PI, PAPTAC, TAPPI, SPCL, PTF

June 4-8, 2001

Helsinki, Finland

#### 2001 Intl. Chemical Recovery Conf., PAPTAC, TAPPI

June 11-15, 2001

Chateau Whistler Resort, Chateau Whistler, BC, Canada

#### 2001 Intl. Symp. on Wood and Pulping Chemistry, ATIP, PAPTAC, EUCEPA, TAPPI, Chinese TAPPI, APPITA and Japan TAPPI

June 11-14, 2001

Acropolis Congress Centre, Nice, France

#### 2001 Intl. Symp. on Corrosion in the Pulp and Paper Industry, VTT Manufacturing Technology, Finnish Paper Engineer's Assoc., PAPTAC, SPCL, Swedish Corrosion Inst., NACE, TAPPI, KCL and European Federation of Corrosion

August 21-24

Helsinki, Finland

*For further information on the PAPTAC conferences listed above, please contact PAPTAC, 740 Notre Dame West, Suite 810, Montreal, QC, Canada H3C 3X6; Tel. 514-392-0265; Fax: 514-392-0369; e-mail: tech@paptac.ca; Internet: http://www.paptac.ca.*

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