The Effect of Moisture Content on the Micro-damage Processes of Spruce Wood, Investigated by Acoustic Emission Method and Electron Microscopy

Antal KÁNNÁR[*]

Institute for Applied Mechanics and Structures, University of West Hungary, Sopron, Hungary

Abstract – Spruce was investigated by acoustic emission during tensile tests. The examined moisture contents were 0%, 12%, and 26%. The fracture surfaces were photographed using electron-microscopy. The experiments showed, that micro-damage processes start near the ultimate strength, regardless of moisture content. This indicates the brittle behaviour of wood.

The analysis of detected acoustic events at different moisture contents indicated that the number and properties of events supposedly resulting from breaking do not change with increasing moisture content. Decrease occurs in the total number of events at higher moisture contents as a consequence of the increasing acoustic attenuation of wood and the elimination of friction type events. Electron microscopic analysis of fracture surfaces supports the result of acoustic emission experiments. The fracture surfaces showed characteristic brittle tension and shear across the cell wall of different cells. Based on both investigations we can say that wood has brittle fracture characteristics in the 0-30% moisture content range.

acoustic emission / wood / brittle fracture / damage process / moisture content

Kivonat – **A nedvességtartalom hatása a lucfenyő mikro-tönkremeneteli folyamataira, vizsgálatok akusztikus emissziós módszerrel és elekron mikroszkóppal.** Lucfenyő húzóvizsgálat során mutatott akusztikus emisszióját vizsgáltuk 0%, 12%, 26% nedvességtartalom mellett. A tönkremeneteli felületekről EM képeket készítettünk.

A vizsgálatok azt mutatták, hogy a nedvességtartalomtól függetlenül a tönkremeneteli folyamatok csak a törőterhelés közelében indulnak meg döntően. Ez a faanyag rideg törési természetére utal. A különböző nedvességtartalmi osztályokban kapott akusztikus események vizsgálata azt mutatta, hogy feltehetően törésből származó jelek száma és tulajdonságai nem változnak a nedvességtartalom növekedésével. A nagy nedvességtartalom mellett megfigyelhető eseményösszeg csökkenést feltehetően a faanyag növekvő akusztikus csillapítása és a súrlódásos jellegű események eltűnése okozza. A törési felületek elektronmikroszkópos vizsgálatai alátámasztották az akusztikus emissziós vizsgálatok eredményeit. A törési felületek jellemzően sejtfalon átmenő rideg szakadási és nyírási törési felületeket mutatnak. Mindezek megerősítik, hogy a faanyag nedvességtartalomtól függetlenül rideg törési természetű a 0-30% nedvességtartalmi tartományban.

akusztikus emisszió / fa / rideg törés / tönkremeneteli folyamat / nedvesség tartalom

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[*] corresponding author: toni@fmk.nyme.hu; H-9400 SOPRON, Pf. 132

1   INTRODUCTION

The phenomenon of acoustic emission (AE) is related to elastic waves originating from the release of stored energy due to mechanical loading in solid materials. The source of these acoustic events in case of wood are fibre breakage, cracking, crack propagation and friction type events due to the movements of crack surfaces. The fracture of wood is accompanied by noise in the audible range. However, this phenomenon often starts just above the 90% of the ultimate strength. On the other hand, the damage process start earlier, at about the 40-50% of the ultimate strength. According to Hansel (1980), the frequency range of AE events is 50 kHz to 1.5 MHz. Most researchers typically use a much narrower range. In case of wood, piezoelectric transducer with 150-200 kHz resonant frequency were used (Rice 2001; Kowalski 2004). Based on our own frequency analysis of AE events we established that the analysis of 20-100 kHz frequency domain is also necessary, because wood shows significant AE activity in this range. This range was examined by other researcher too (Reiterer 2000). Because of this, our researches were also carried out with wideband transducers, sensitive between 20 and 250 kHz frequencies (Kánnár 2004).

The AE method was applied successfully in many fields of wood science and technology in the last 30 years. Porter (1972) used AE to predict failure in finger joints. The closer the load was to the ultimate strength, the more precise the predictions were. Ansell (1982) examined the effect of different proportion of earlywood to latewood in Parana pine and Douglas-fir. He established that the logarithmic sum of events as a function of strain shows a linear characteristic in case of Parana pine that has a narrow late wood, but is more irregular in case of Douglas-fir that has a more pronounced late wood.

Molinski (1994) examined the crack formation in wood due to cyclic moisture content changes. He observed partial damage in wood as early as in the first cycle of wood drying/wetting treatment. This decreases the resistance of wood to internal stresses created during wetting. Wood shows a kind of fatigue. Cracks created - like mechanical loads - cause increase in the AE activity, even at stress levels lower than the original.

Poliszko (1994) examined the relationship between water bonding energy in wood and its mechanical strength. He found it theoretically possible to determine the long term behavior and the structural fracture of wood based on short term tests. Reiterer (2000) investigated the AE of notched hardwood and softwood specimens during splitting test. He experienced that there are characteristic differences between the two groups. Conifers showed tenacious fracture and produced a high number of AE events, while hardwoods showed brittle fractures and created a lower number of events. Gozdecki (2005) used the method for detecting failures of adhesive-bonded joints. He found it possible to predict the development of the destruction of an adhesive joint on the basis of the increasing cumulative AE count combined with tangential stresses determined by the finite element method.

Based on the literature, the AE technology is applicable for testing wood and wood products. The presented investigation shows that the number and properties of detected AE events depend on the wood species, moisture content, applied stresses and the load history of the specimen. Research orientation is very diverse; each researcher used different AE apparatus and detectors, therefore results are hard to generalize. Most of the investigations are applied research projects. However, the underlying relationships between the measured AE signals, mechanical properties and fracture processes of wood are not very well known. Basic research is therefore needed to reveal these relationships.

2   MATERIAL AND METHODS

Spruce (*Picea abies*) species were tested in tension parallel-to-grain in RL plain, at three moisture contents (*Figure 1*). The weakening of the specimens' middle section caused the fracture to occur there or start from that location. The measurement setup allowed events that originated from locations other than the tension mid-region (the mid 30 mm) to be filtered out, based on time delay differences between the two transducers. This is important, because many events originated at the grips because of the combined stress state and events originated in the 8mm thick section too. Each series contained 25 specimens. The moisture content levels used were 0%, 12%, 26%. The applied acoustic emission (AE) apparatus was a Defectophone (KFKI Hungary) with two logarithmic amplifiers (*Annex*) and two wideband piezoelectric-transducers type SE1000-H (*Figure 2*). The examined frequency domain was 20-250 kHz. The applied threshold was 22 µV. The examination of 20-100 kHz range seemed to be essential due to the preliminary analysis, which showed ca. 30% of the total events are in this range (Kánnár 2004). The coupling material was silicon grease. The characteristic fracture surfaces were captured via electron microscopy (EM) (ETH Zürich).
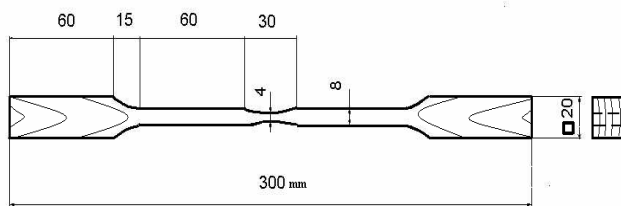
Figure1. Specimen detail   (units: in mm)

Figure 2. Fastening the transducers on the specimen with rubber bands

3   RESULTS

The experiments showed that the AE processes start at stress levels near the ultimate strength in most cases (*Figure 3 and 4*). Because the sources of AE are breaks, cracks and the friction of fracture surfaces, this implies brittle behaviour in wood. This behaviour did not change with   increasing moisture content. At higher moisture content levels fewer events were detected, but the starting point of the micro-damage process did not vary. Drawing further conclusions concerning the relationship between AE processes and moisture content was difficult, because the investigation of AE processes of several specimens - made of the same wood species - did not give an unequivocal picture about the AE behaviour of wood. Some specimens broke producing a few events while in other cases several hundred events could be detected (*Figure 3 and 4*). The reason for this anomaly is that failures may be initiated at various points within the full volume of the stressed specimen, which progress randomly towards one another (Bariska 1985). Additionally, the advancement of micro-damage processes is a function of the particular specimen's biological and anatomical structure.
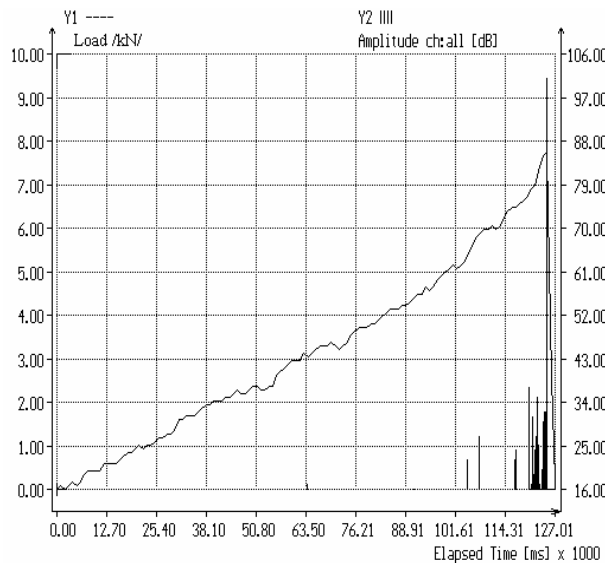
Figure 3. Acoustic activity of spruce specimen no. 6 at 12% MC
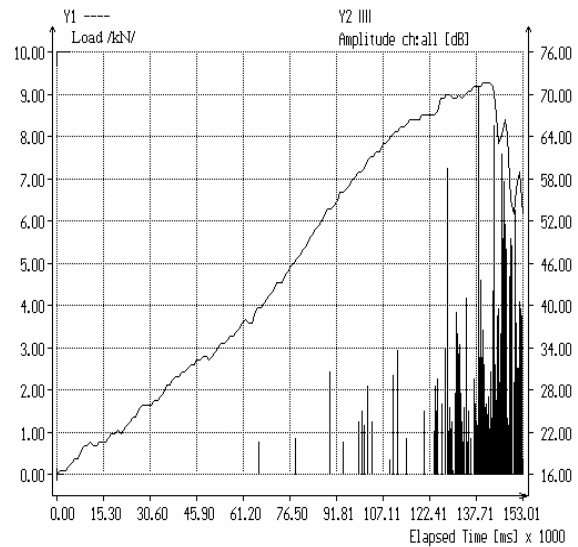

Figure 4. Acoustic activity of spruce specimen no. 10 at 12% MC

Based on the above considerations, the data resulting from specimens with equal moisture content were pooled and examined in clusters. These clusters consist of 3000-6000 acoustic events each, which are representative in terms of the AE properties of wood. Valuable conclusions can be drawn from the examination of the distribution of AE events concerning the fracture behavior of wood.

First the frequency curve of event amplitudes are presented for the three investigated moisture content (MC) classes (*Figure 5*).
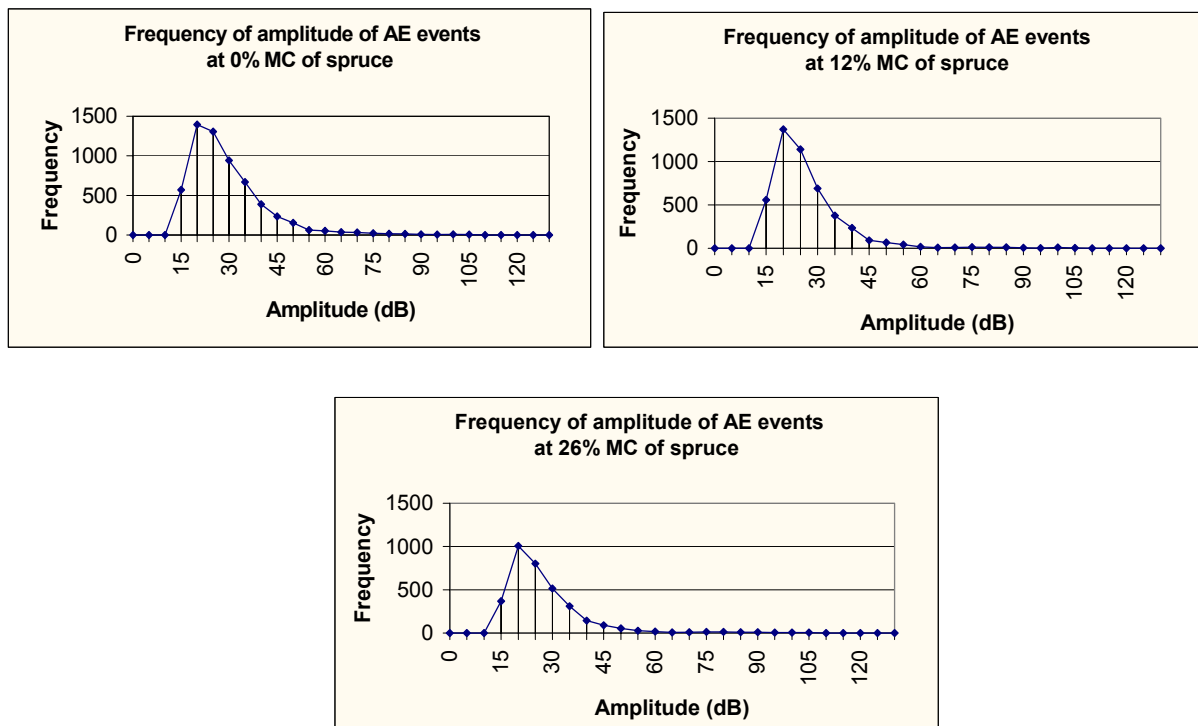

Figure 5. Frequency of amplitude of AE events at different MC of spruce

Comparing the three amplitude frequency functions, it is established that they are almost the same. Maximum frequencies are in the 20 dB range and the amplitude domain is 15-60 dB in all cases. (Amplitude values may be converted from dB to Voltage using the $U_{amp}(V) = 0.4x10^{(AMP\ (dB)-100)/20}$ relationship. Accordingly, 20 dB and 60dB correspond to 4μV and 4 mV, respectively). Let us compare the frequency functions with the total number of events detected in each moisture content class, which have an equivalent with the sample size. At 0%, 12% and 26% moisture content levels 5707, 4697 and 3180 events were registered, respectively. Comparing the frequency functions we can say that there is a considerable decrease mainly in the higher amplitude domain in case of 0% and 12% MC. The decrease did not influence the maximum frequency. The additional increase of MC affected the maximum frequency, and all other frequencies decreased proportionally. Based on the ditribution function the following conclusions can be drawn:

Despite the fact that most mechanical properties, e.g. strength, change considerably with increasing MC (Kollmann 1982), the most-frequently-experienced micro-damage event amplitude does not change. Accordingly, changes in the nature of the damage processes cause the frequency to decrease in the higher amplitude range. This due partly to the increased acoustic attenuation, which increase with 5-7% at the 0-30% MC range in a distance 0-30mm (Kánnár 2004).The other reason is the changes of deformation properties with increasing MC.
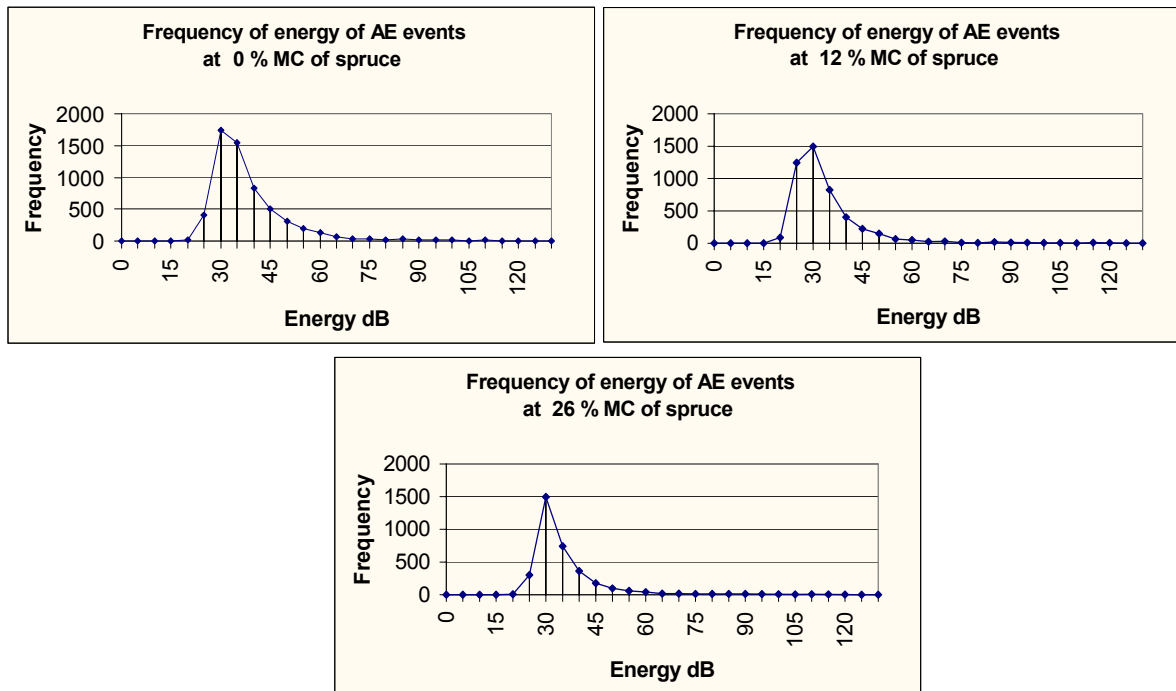

Figure 6. Frequency of energy of AE events at different MC of spruce

Further conclusions can be drawn based on the analysis of energy-frequency distribution functions (*Figure 6*). The energy-frequency curves show that the maximum frequency is given in the 30 dB energy domain in all cases. (The conversion of dB to picoJoule $E\ (pJ) = 2.75x10^{-6}x\ 10^{(E\ (dB)/10}$ accordingly, 30 dB and 60dB correspond to $2.75x10^{-3}$ pJ and 2.75 pJ, respectively.) The character of the curves is similar, but the second most frequent energy class varies, being at 35 dB ($8.69x10^{-3}$ pJ), 25 dB ($8.69x\ 10^{-4}$ pJ) and 35 dB, at 0%, 12% and 26%, respectively. Also, at 26% MC, the frequency in the second most frequent class is considerably smaller than at 0% MC. At 12% MC, the second highest frequency occurs at an energy level ten times lower than at 0%. The observable decrease in the sum of

events with increasing MC causes the decrease of high-energy events. In the meantime, the event number is three time bigger in the 20 dB class at 12% MC as it is at 0% MC.

Further increase in MC causes event numbers to decrease at 20dB energy range, which falls back to the value detected at 0% MC. The energy domain is 20-60 dB in all cases. The following assumptions may explain the phenomena experienced:

- In case of 0% MC, the existing drying cracks generate a high number of friction type events, due to the friction of crack surfaces. Coupled with the smaller acoustic attenuation of dry wood, this gives rise to a large number of high energy events.
- At 12% MC, bound water between cellulose microfibrils and between cellulose and lignin allows bigger deformations without micro-damages. Wet wood contains fewer cracks, therefore there are fewer friction type events too. The measured event energy is lower due to the higher MC.
- In case of 26% MC the fibre walls are nearly saturated with water. The water molecules relax the hydrogen-bonds between the micro-fibrils and allow slipping without generation friction-type acoustic emission, or the energy level of friction-type events is below the threshold level. Accordingly the disappearance of small energy friction-type events causes less event frequency in the 20 dB energy range.

The number of events in the maximum frequency class was nearly the same in all three cases. This indicates that the number and character of fracture-type events do not change with changing MC. Any change in the frequency curve probably originates from changes in friction-type events and acoustic attenuation.

Next, event rise time functions were examined. The rise time is defined as the time elapsed until events reach their peak amplitude. The rise time characterizes how fast micro-damage process arises. With the use of rise time the approximate ratio between fracture- and friction-type events may be determined. The following figures show the rise time-frequency distribution function for the three MC ranges (*Figure 7*).
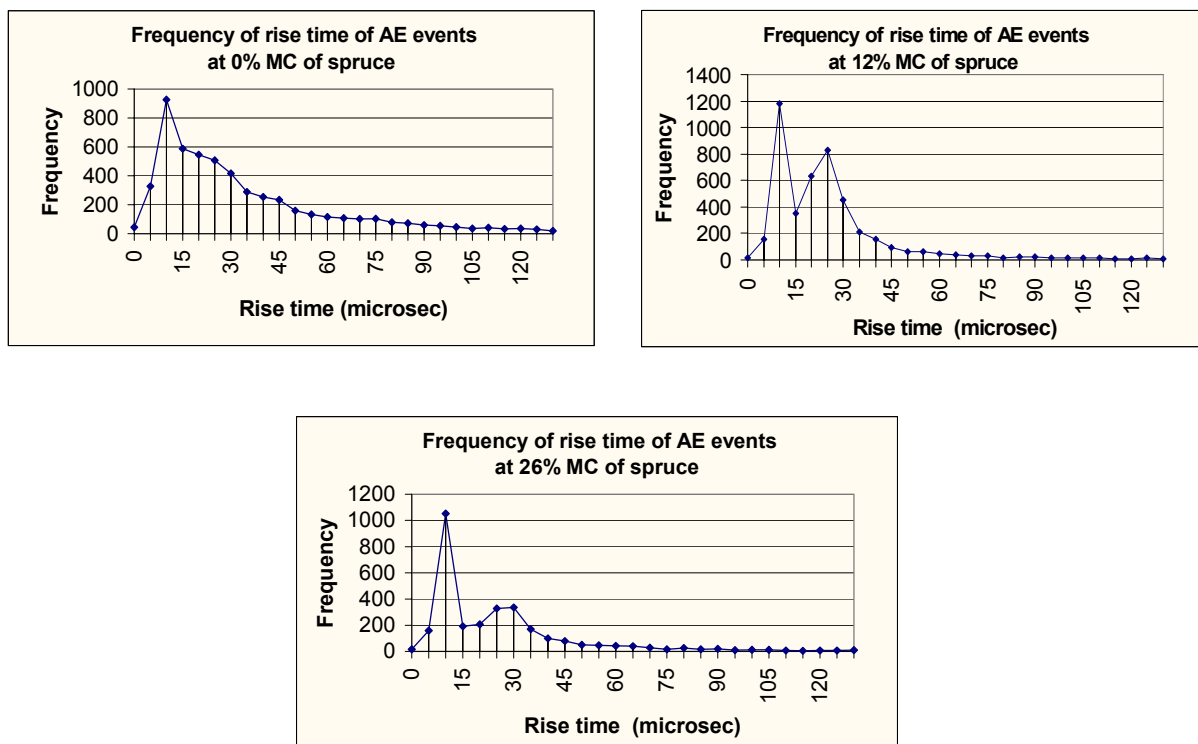

Figure 7. Frequency of rise time of AE events at different MC of spruce

The analysis of the three functions showed that they are similar, but observable differences can be found in the 15-100 μs rise time range. In all three cases, events with 10 μs rise time, presumably originating from fracture-type events, are dominant, and this does not change notably with MC. The characteristic rise time range is 0-100 μs at 0% MC. This range shrinks to 0-60 μs at 12% MC and shows no further change at 26% MC. However, there are differences between the latter two MC classes in terms of frequencies in the 20-50 μs range. At 26% MC, half as many events were detected in 20-50 μs range than at 12% MC. Comparing this phenomenon to the sample size of AE events at the three MC levels we can say that the decreasing event number detected at increasing MC's did not affect the number of events in the maximum frequency class. The decrease occurred in the higher rise time domain. If we accept the assumption, that shorter and longer rise times are related to fracture and friction, respectively, we can make the following statements:

- At 0% MC friction-type events, generated at drying crack surfaces, cause higher frequencies in the 15-60 μs rise time range.
- In case of 12% MC the frequency of the 10 μs class slightly increases. This supposedly indicates an increase in the number of fracture-type events. The characteristic rise time range shrinks from 0-100 μs to 0-60 μs. The total number of events in this smaller range is approximately the same as at 0% MC. Increasing MC causes long rise time events to disappear.
- Increasing MC to 26% MC does not change the typical 15-60 μs rise time range. The number of events in the 15-60 μs range, however, decreases to one half, however it remains unchanged at its maximum of 10 s.

In summary, we can say that increasing the MC causes decrease in the number of friction type events. The number of fracture type events does not change considerably with changing MC. Thus, analysis of the rise time function supports the results of energy function analysis.

To verify the conclusions of AE experiments, SEM images were taken of the fracture surfaces at each MC level. The characteristic fracture modes were tension and shear at all MC content levels, so these two types were analysed in all cases (*Figure 8*).
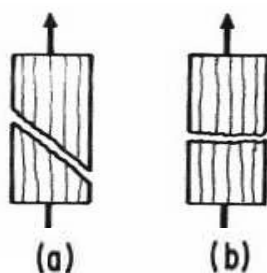
*Figure 8. Fracture modes of spruce specimens (a.) shear, (b.) tension*

Figure 9 shows the fracture surface of spruce at 0% MC. The left-hand picture shows a view of the fracture surface. It demonstrates that most tracheids showed brittle transwall failure, but at certain points, bundles of tracheids were pulled out. The right-hand picture shows one of the extracted tracheids packets, along with brittle shear fracture on their lateral surfaces.

Shear failure is actuated along ray parenchyma cells, that constitute critical cross-sections in terms of shear.
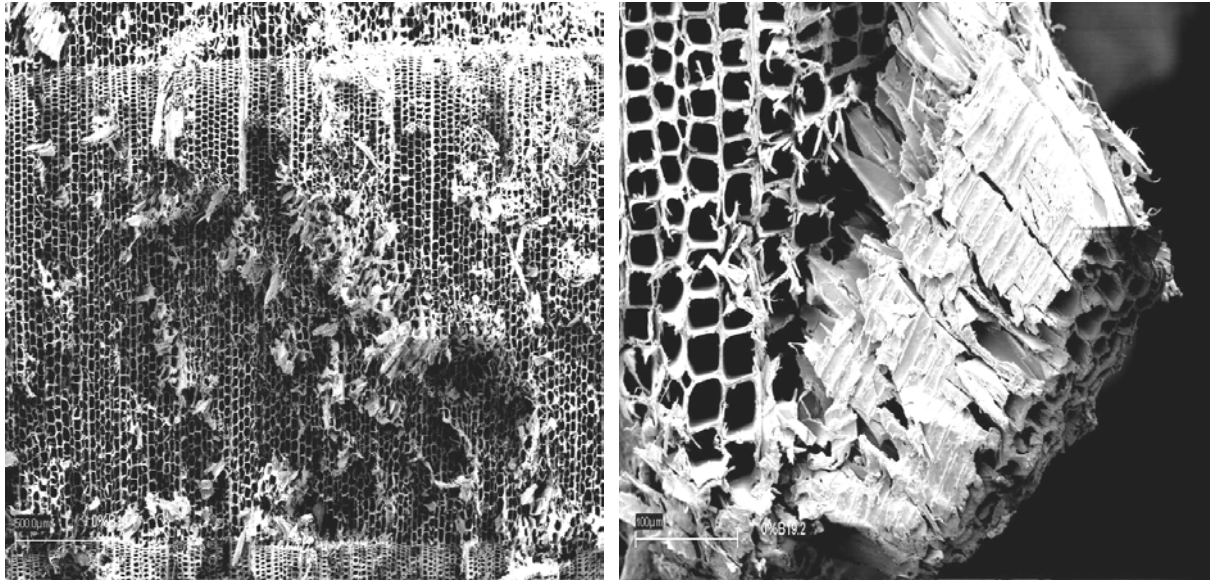
Figure 9. Tension fracture surface of spruce at 0% MC after tensile test

If the fracture mode is shear (*Figure 10*) then tracheids shear longitudinally. The fracture surfaces are brittle, but the S2 cell wall layer is also often extracted. Bordered pits do not shear; the shear line avoids the pits.
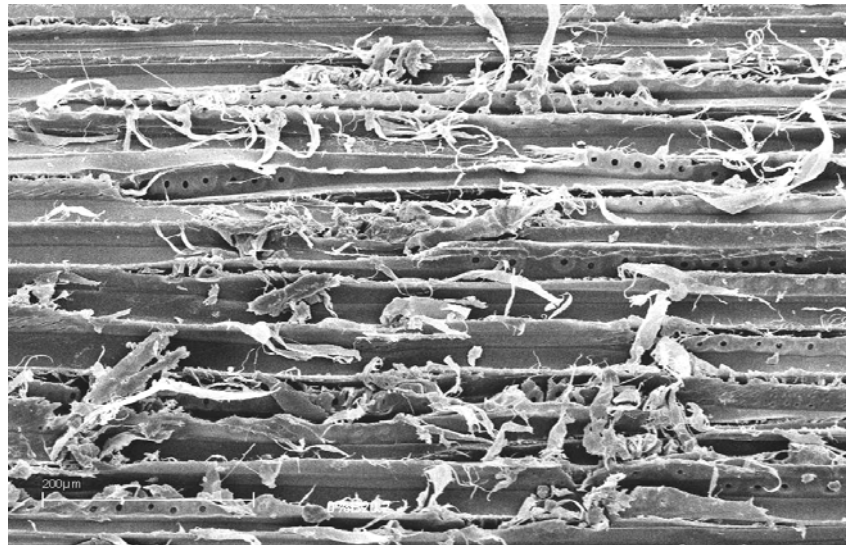
Figure 10. Shear fracture surface of spruce at 0% MC after tensile test

At 12% MC, the fracture surfaces were very similar to those at 0% (*Figure 11*). Most tracheids showed brittle transwall failure. At some locations, bundles of tracheids were pulled out and brittle shear fracture can bee seen on their sides. The MC increase did not change the character of tensile type fracture surfaces.
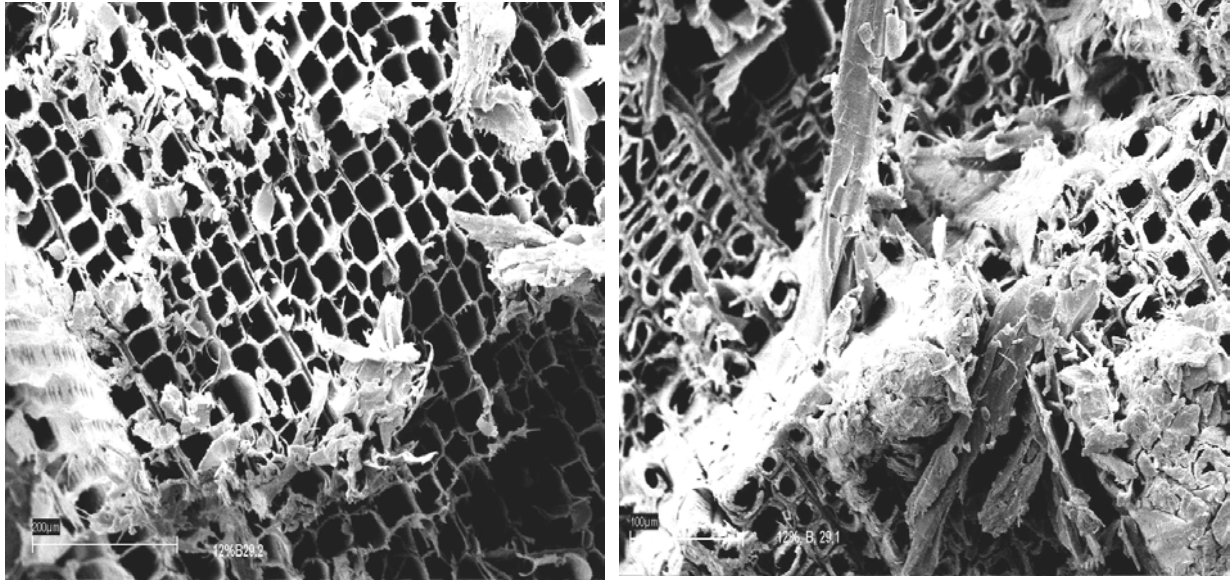
Figure 11. Tensile fracture surface of spruce at 12% MC after tensile test

The shear fracture at 12% MC originated through the brittle transwall shear failure of tracheids (*Figure 12*). Ray parenchyma cells fail in shear too (right picture arrow). The pull out of S2 cell wall layers is also visible, but the number of pull-outs did not change considerably with increasing MC.
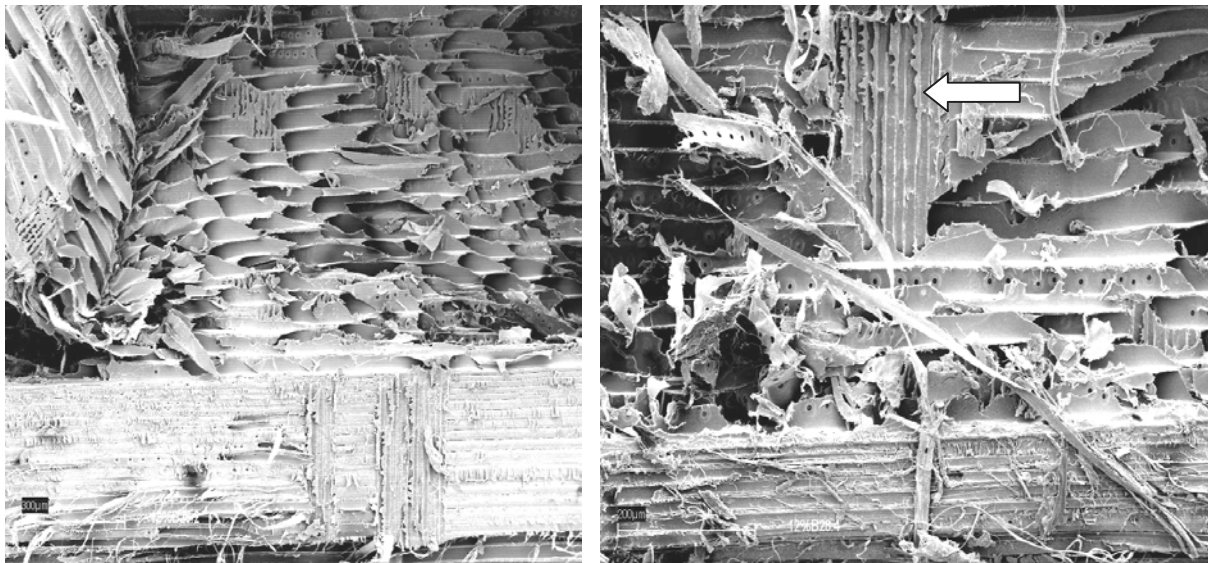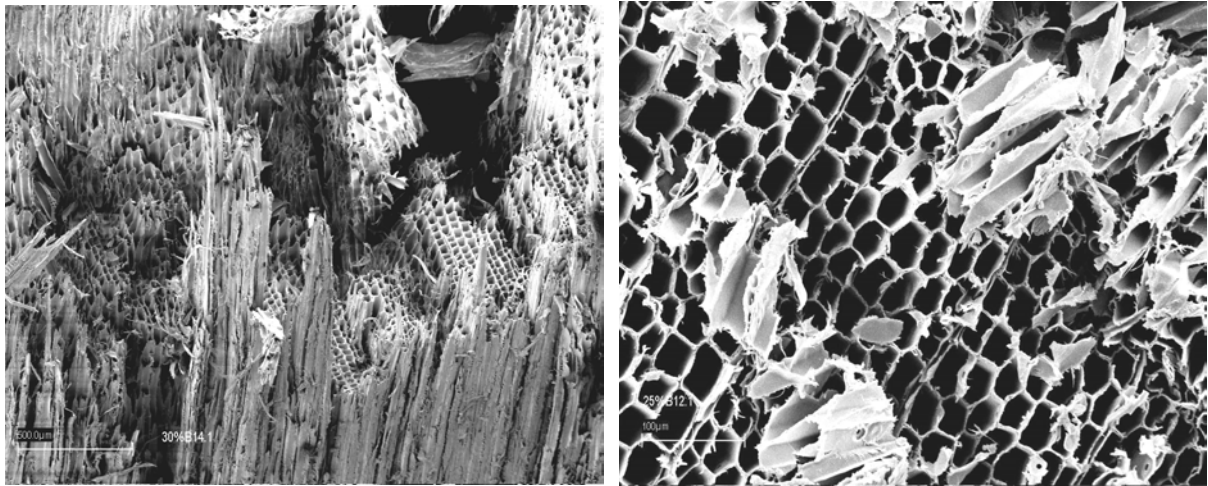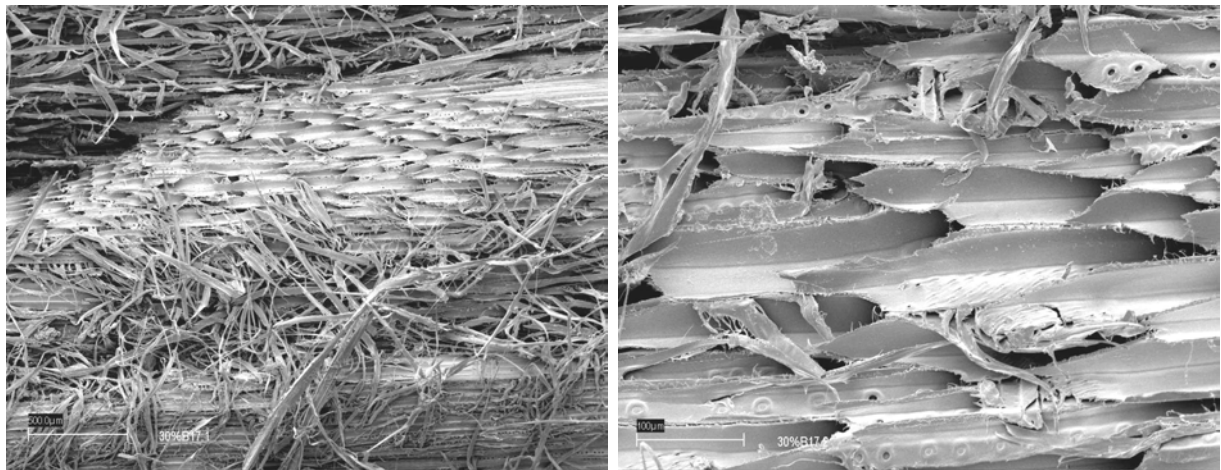

Figure 12. Shear fracture surface of spruce at 12% MC after tensile test

Near the saturation point of spruce the character of the tensile fracture surfaces does not differ from the other two MC classes (*Figure 13*). The left-hand picture shows well the typical step-wise break of wood cells. On the horizontal planes tracheids broke with a brittle transwall failure (right-hand picture); on the vertical planes cells failed in shear. The number of tracheid bundle pull-outs does not change at 26% MC compared to 12% MC. Pull-outs are results of combined tensile and shear fracture.
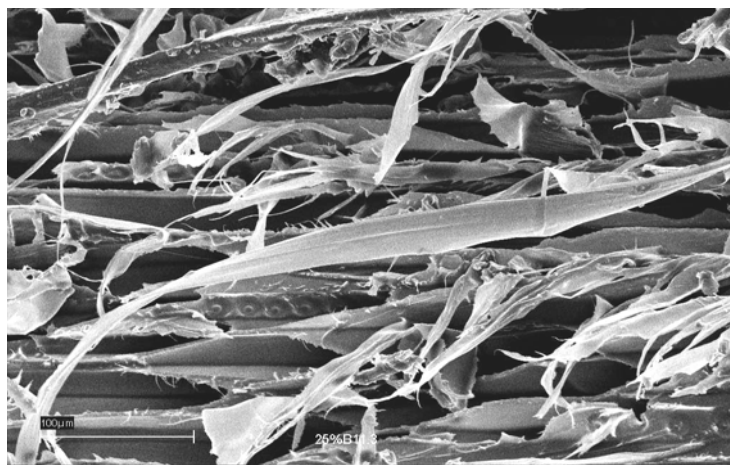
Figure 13. Tensile fracture surface of spruce at 26% MC after tensile test

Figure 14. Shear fracture surface of spruce at 26% MC after tensile test

The shear type fracture at 26% differs from the former cases (*Figure 14*).

Figure 15. S2 cell wall layer pull-outs in spruce at 26% MC after a tensile test

The brittle shear character partly remains, as shown in the middle of the left-hand picture and on the right-hand picture. However, the number of S2 layers pull-outs increases considerably (*Figure 15*). The predominance of such intrawall failures indicates ductile, plastic failure at 26% MC.

Summarizing the experiences of fracture surface analysis at different MC's, we can state that the characteristic fracture mode of wood is brittle tension and shear.

The brittle character does not change considerably with increasing MC. The effect of MC near the saturation point is considerable, where the predominance of intrawall failure - namely the extraction of the S2 cell wall layer - increases. In this case the failure mode is mixed, partly brittle, and partly plastic. This changes can promote the decrease of sum of events at the saturation point because during the pull out of cell wall layers originate supposedly smaller energy events than in case of brittle fracture and a part of this events remain under the thresh- hold level of measurements.

CONCLUSIONS

Based on the AE experiments, the fracture behaviour of wood is similar to brittle materials. The micro-damage process starts near the ultimate strength. The decreasing number of events with increasing MC is a consequence of the decreasing number of friction-type events and of the increasing acoustic attenuation of wood. The maximum frequency of measured events properties does not change, indicating that the number and character of fracture-type events do not change with changing MC. The increasing MC does not significantly influence the range and properties of AE events. This means that the micro-damage process does not vary notably.

The analyses of fracture surfaces supports the results of AE experiments. The main damage mode of wood is brittle tension and shear. This brittle character does not change considerably with increasing MC. This explains why the AE experiments gave nearly the same measured and calculated parameters. These results support the assumptions of other researchers i.e. the mechanical properties of wood change with increasing MC only gradually, but not fundamentally (James, 1961). This presumption is valid at the level of micro-damage processes too.

In conclusion we can state that the properties of micro-damage processes do not change significantly with increasing MC, and that main failure mode of wood is thus brittle tension and shear.

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Annex. Measuring equipment

AE measuring apparatus:
- manufacturer: KFKI Hungary
- type: DEFECTOPHONE 4 channel AE equipment

Logarithmic Amplifier:
- manufacturer: KFKI Hungary
- type: Nez-220-BP32
- frequency range: 20-600 kHz
- dynamic range: 80 dB

AE detectors:
- manufacturer: DECI CO.
- type: SE-1000-H
- frequency range: 20-250 kHz