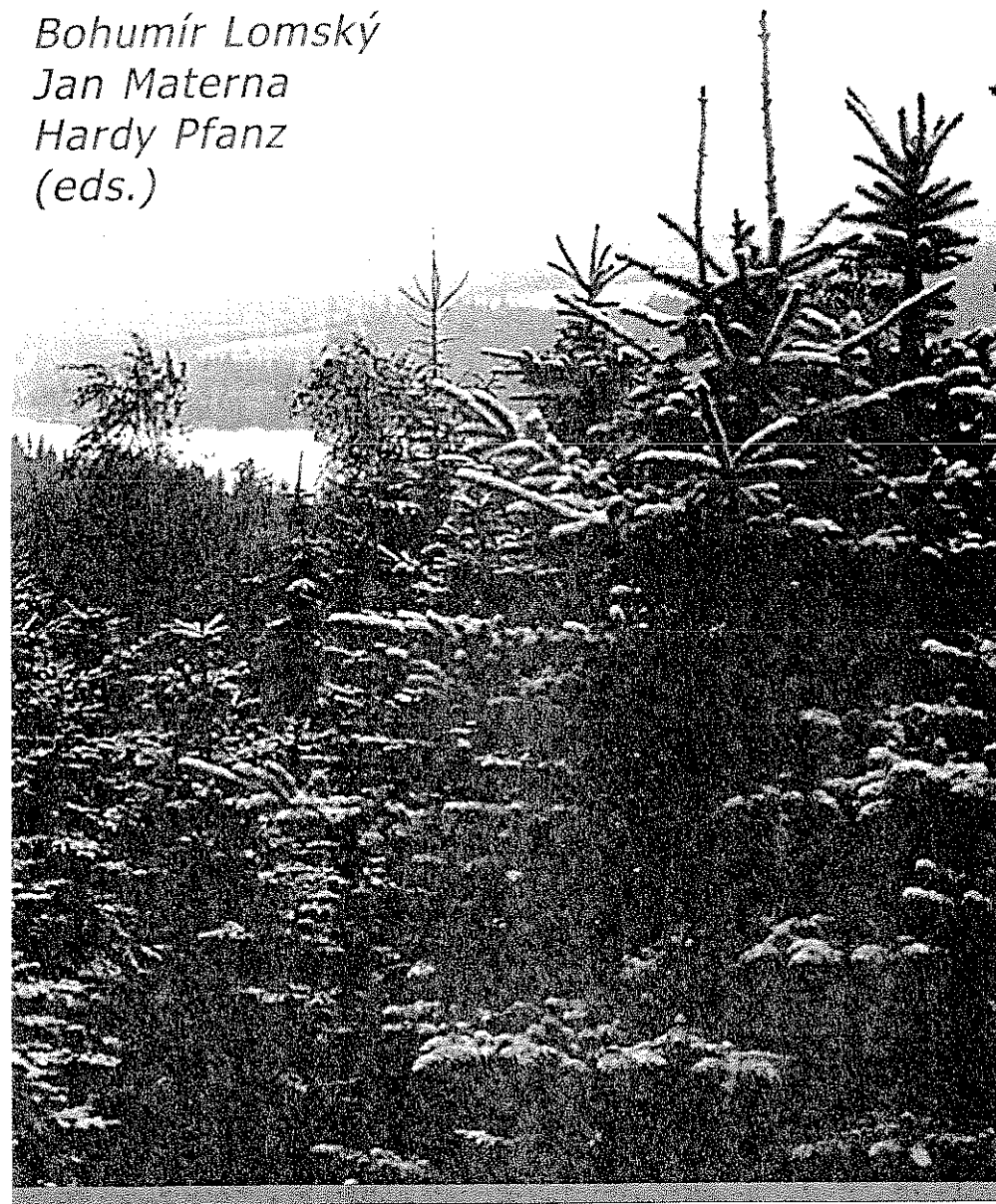


# SO<sub>2</sub>-Pollution and Forests Decline in the Ore Mountains

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## CHAPTER 12

### ANATOMICAL, CHEMICAL AND MECHANICAL TRENDS IN NORWAY SPRUCE (*PICEA ABIES* [L.] KARST.) TREE-RINGS AS INDICATORS OF ENVIRONMENTAL STRESSES, IN PARTICULAR SO<sub>2</sub> - POLLUTION

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#### 12.1. Introduction

Potential influences of atmospheric pollution on forests have been recognized for more than a century and first scientific reports of damages in pollution-related forests originate in Tharandt in the East Ore Mountains. In 1866, Stöckhardt started investigating sulphur dioxide effects on plants in Tharandt and postulated negative effects on the growth of spruce trees. Later, Gerlach (1908) has shown negative effects of SO<sub>2</sub> on radial growth of forest trees. Halbwachs and Kissler (1967) were the first looking at wood anatomical changes of a spruce and a birch grown under high concentration of airborne fluoride. Liese et al. (1975) investigated the anatomical structure of a SO<sub>2</sub> polluted spruce tree. Since the "Neuartige Waldsterben" appeared publicly in the early 1980's, a number of workers have considered wood anatomy (e.g. Grosser et al. 1985, Wimmer and Halbwachs 1992, Wimmer 2002) but also wood chemistry (e.g. Rademacher et al. 1986, Fengel 1987a) as well as mechanical properties (e.g. Frühwald 1986, Wimmer 1993) in their studies.

This chapter reports on three types of data collected from tree-rings of Norway spruce grown in the East Ore Mountains. The data are grouped into anatomical, chemical and mechanical parameters. All measurements were related to the time when the wood was formed which allowed direct comparisons of these parameters. Therefore, the primary focus of this investigation was to measure anatomical, chemical and mechanical parameters on identical specimens at a high temporal resolution in order to indicate the particularly high SO<sub>2</sub> - pollution stresses.

## 12.2. Material and Methods

### 12.2.1. Study sites and trees

The study area consisted of two established sites in the forest district Seyde, Eastern Ore Mountains, Saxony, 50 km south of Dresden, Germany, close to the Czech border. The sites were even-aged, approximately 70-years old stands of Norway spruce (*Picea abies* [L.] Karst.), both located on quartzporphyric bedrock. The natural forest community is a mixed beech-fir-spruce forest at altitudes of 700 - 820 m a.s.l. Spruce trees covered 80% of the area and the predominant brown forest soils are partially podsolized. Basic site data are given in tab 12.1. The two approximately 80 m x 80 m plots were similar in age, soil and vegetation structure but differed in their degree of disturbance including heavy sulphur dioxide pollution from industrial facilities located in the south and in the east. Site I, located at 730 m a.s.l., was less exposed to SO<sub>2</sub> pollution (damage zone II-III) whereas site II, located at 810 m a.s.l., was highly exposed (damage zone I). The linear distance between site I and site II was 3,000 m. The stresses were reflected in crown and needle measures (crown transparency, needle retention, tip yellowing) as well as high levels of sulphur in the foliation. Sulphur contents of one-year old spruce needles at site II were higher than at site I and ranged between 1,700 ppm and 3,000 ppm. Further, mean annual SO<sub>2</sub>-concentrations in the air were above 100 µg.m<sup>-3</sup> with hourly maxima reaching 1,000 µg.m<sup>-3</sup> (Nebe et al. 1995).

Tab. 12.1: Basic descriptions of the investigated sites. Site I (less stressed) and site II (heavily stressed), means and coefficients of variation CV

Parameter	Site I		Site II	
Altitude (m)	730		810	
Exposition	north-west, 0-5°		0°	
Stocking density	0.7		0.4	
Age at breast height	65		63	
Damage zone*) (according to the forest inventory)	II - III		I	
Number of trees	10		10	
	Mean	CV(%)	Mean	CV(%)
Tree height (m)	22.0	3	19.5	3
Crown length (m)	11.0	16	7.9	23
Breast height diameter (cm)	31	11	28	13
Crown transparency class (1 - 5) (after Neumann and Pollanschütz 1988)	2.3	12	3.9	10
Needle retention (%) (after Knabe 1983)	372	10	157	54

\*)Damage zones: I = dying (100 µm/m<sup>3</sup> SO<sub>2</sub>); II = heavily damaged (85 - 90 µm/m<sup>3</sup> SO<sub>2</sub>); III = little damaged (55 - 65 µm/m<sup>3</sup> SO<sub>2</sub>)

The Ore Mountains are located in a transition from atlantic to continental climate types. East Ore Mountains experience especially high fluctuations in temperature with cold winters and little precipitation. Annual total precipitation for these sites is 965 mm, 38% of which is snow. Annual mean temperature is 5.5 °C, with -23 °C as the lowest temperature measured. Cloudiness and wind are more prevalent on site II than on site I. Main wind directions are north and north-west with a mean annual wind speed of 6.1 m.s<sup>-1</sup>.

In April 1993, ten dominant and codominant trees were felled on each site and several stem disks were removed at about 4-m above ground. This height was chosen – beside saving the first 4-m log of the stem - because wood formation operates physiologically more sensitive higher up in the tree than at lower parts (e.g. breast height) (Wilpert 1990). In addition, the stems were less affected by mechanical induced wounds. Disks were immediately transported to the laboratory for the preparation of the different types of samples. Cross-dating was carried out carefully according to Stokes and Smiley (1968) which secured exact dated samples for all types of measurements. This step was especially important for the quality of this study because a number of missing rings and microrings (Swetnam et al. 1985) were detected in the trees of site II.

## **12.2.2. Methods**

### **12.2.2.1. Anatomical procedures and specimens**

To evaluate the foliation of the trees back in time we applied the needle-trace-method introduced by Jalkanen and Kurkela (1990) and modified for spruce by Sander and Eckstein (1994). Instead of reconstructing the full foliation history by analysing all lateral shoots we investigated only single shoots, which were cut from identical stem heights of all trees. The number of needle traces counted at the innermost tree-ring was set 100% and the loss of needle traces over the years was measured and averaged for each site. This procedure provided a quick and straight-forward indication of defoliation processes some 50 years ago.

The anatomical parameters were measured on transverse and longitudinal sections - 20-µm in thickness – cut with a sledge microtome from 1 cm x 1 cm cubes. Sections were dehydrated, stained with methylene blue and mounted in Malinol on slides (Gerlach 1984). The transverse sections continuously covered the tree-rings from pith to bark. Likewise, tangential sections were prepared from each individual tree-ring. For the strength of our data, two radii (north and south) were prepared from each disk. Most anatomical parameters were obtained using a light microscope with a CCD-camera connected to a Macintosh computer that was loaded with the NIH-Image analysis system (Wayne Rasband, National Institutes of Health, USA). On the cross sections we have measured the parameters cell wall percentage in earlywood and latewood (De Kort 1991) and the number of resin ducts per mm<sup>2</sup> (Wimmer, Grabner 1997). On longitudinal sections the parameter mean height of uniseriate rays (van Oever et al. 1981) and the microfibrillar angles in earlywood and latewood (Senft and Bendtsen 1985) were measured. On parallel disks taken from each felled tree dendrodensitometry was performed according to Schweingruber (1989) and the standard parameter set was extracted including latewood percentage and ring widths.

#### **12.2.2.2. Chemical procedures and specimens**

Branches from all 20 trees, approximately 5 mm in diameter, were taken from lower and middle regions of the crowns for measuring the peroxidase activity. A standard procedure was used according to Worthingtons Enzyme Manual (1972) partly modified by Korori (1989). Enzymatic activity of peroxidase was measured for wood, bark and needles separately.

Specimens for lignin analysis were dried, milled, and extracted with acetone (24 h) as well as ethanol: toluol (1:1, 6 h). Lignin was determined by means of DRIFT (Diffuse Reflectance Infrared Fourier Transform) - spectroscopy using a Perkin Elmer System 2000® and the calculation was done by relating the band at 1,510 cm<sup>-1</sup> (aromatic skeletal vibration) to 896 cm<sup>-1</sup> (C1-H vibration in cellulose and hemicellulose) after baseline correction (Ulreich 1995). MWL (milled wood lignin) was used for calibration.

Cold water extracts of milled wood were used to determine the content of soluble sugars (fructose, glucose and sucrose) through HPLC (High-Performance-Liquid Chromatography). Prior to measurement the extracts were filtered and the phenolic compounds were precipitated with PVPP (polyvinylpolypyrrolidon) (Hinterstoisser et al. 1996). Lignin and sugars were determined in each ring of the 20 trees investigated.

The inorganic constituents were determined employing Neutron Activation Analysis (NAA), which has - beside low detection limits - the advantage of no chemical pre-treatment required for the samples. Elements were either measured by  $\gamma$ -spectroscopy directly (Ca, Mn, Al), after sufficient cooling time (Ba, Cs, Rb, Fe, Zn, Cr, Co, Br, Na and K), depending on the half-life period of the activated elements. Element concentrations were related to Mn, Al and Co standards, which were irradiated together with the samples. Tree-rings were pooled in 5-year and 10-year segments for all trees according to Smith and Shortle (1996).

#### **12.2.2.3. Mechanical tests and specimens**

Because sampling side at the tree might be of relevance, preliminary mechanical test runs were performed with samples taken from all four tree-sides. As a result, no significant influence of the cardinal direction was found. Therefore, instead of keeping always the same cardinal direction we were especially careful in avoiding reaction wood. Specimens for the mechanical tests were prepared from six trees of each site. Tensile strength in longitudinal direction was measured with 1 mm, 50  $\mu$ m and 100  $\mu$ m thick specimens, which were 10 mm wide and 80 mm long (Kennedy, Ifju 1962). Micro-tensile tests were combined with acoustic-emission detection to study mechanisms of deformation. Due to the high scatter of the measurements in latewood evaluations we restricted to earlywood samples. Earlywood density-profiles revealed that no differences between the sites existed, which led us to the conclusion that the effect of wood density in earlywood is of minor relevance for the mechanical tests.

We adopted a testing method to characterize the fracture properties of wood by performing wedge-splitting experiments according to Tschegg (1986). This technique allowed the performance of load-displacement-curves (F vs.  $\delta$ ) until final fracturing. From the measured curves the "specific fracture energy" G<sub>f</sub> was derived by integrating over the enclosed curve and dividing the obtained value by the generated fracture area A (fig. 12.1). This value did not only characterize the maximum force and thus the usually determined "fracture toughness" consumed to initiate fracturing, but characterized

particularly the total energy required to generate the crack through the specimen. So far, only a few studies dealing with fracture energy measurements of wood appeared in the literature, among Porter (1964), Mindess et al. (1975), Boström (1992), Aicher (1994) and Schatz (1995).

Choice of specimen size and shape were based on previous studies on the influence of the "size effect" (Stanzl-Tschegg et al. 1995). The specimen size was 42 x 40 x 7 mm with a 20 mm deep starter notch, ensuring the crack plane being formed in longitudinal direction within earlywood or latewood of a tree-ring. This implied that the resulting fracture surfaces were oriented perpendicular to those of the tensile tests. The experimental details are fully described in Stanzl-Tschegg et al. (1995). The splitting experiments were also equipped with acoustic emission detection. Specimens were conditioned at 20°C and 65% relative humidity, resulting in equilibrium moisture content of 12%. In order to obtain statistically relevant data, six trees from each site were measured with a minimum of five up to twenty specimens for each position per tree.

After completion data acquisition, smoothed curves (Stineman function) were fitted to all data. The output of this function has a geometric weight applied to the current point and  $\pm 10\%$  of the data range. With this method, mainly the high frequency variation was removed to better visualise low and medium termed variations (5 - 10 years).

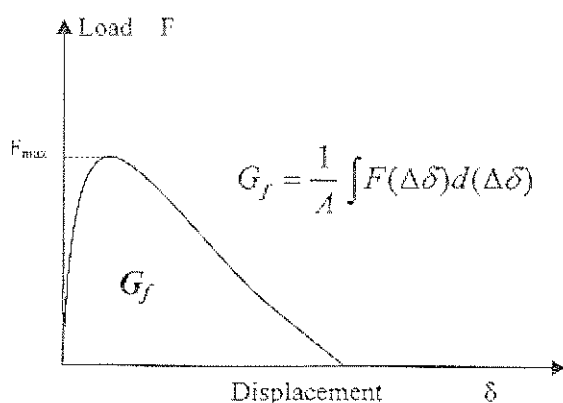


Fig. 12.1: Load-displacement curve for the determination of the specific fracture energy ( $G_f$ ) (acc. to Tschegg (1986)). This curve is recorded during crack propagation until complete separation of the specimen

### 12.3. Results and discussion

At the time of sampling the heavily polluted and more stressed site (II) showed needle retention less than half of site I. But how was the foliation on site II before the widespread forest decline was recognized in that area during 1950's (Schilke, pers. comm.)? Fig. 12.2 shows the needle retention over needle age for single lateral shoots on each site. One can see that the decline of foliation on site II is less pronounced than on site I. As a conclusion, back in the 1940's the foliation and longevity of the needles were equal or slightly better on site II compared to site I. Therefore, the reduced needle retention at the time of sampling may be related to increased stresses at site II with SO<sub>2</sub> as the dominating factor (Nebe 1995).

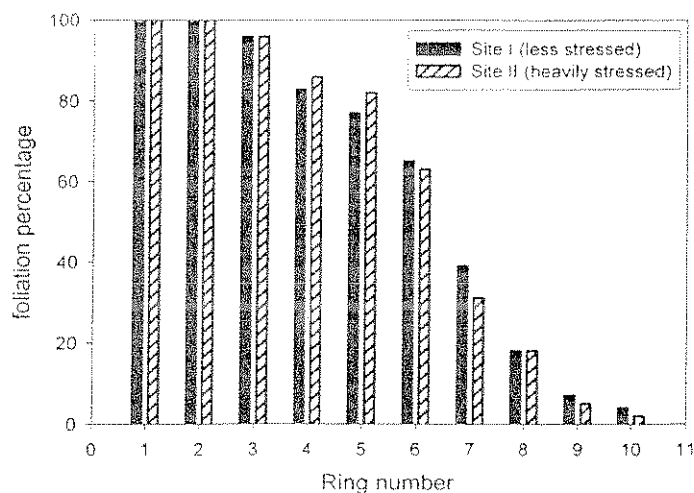


Fig. 12.2: Defoliation processes on the two sites of lateral shoots initiated in the years 1936 to 1944 by means of the needle trace method (Sander and Eckstein 1994). One shoot per tree was measured and the number of needle traces in the first ring is set 100%

### **12.3.1. Trends in wood anatomy**

#### **12.3.1.1. Growth rate, latewood percentage and wood density**

In fig. 12.3 two curves are shown for each parameter representing the less and heavily stressed forest sites. First, the curves for ring width (fig. 12.3A), latewood percentage (fig. 12.3B) and maximum density (fig. 12.3C) are discussed. The ring width curves decline with increasing age on both sites as expected in closed-canopy forests. Beginning in the late 1970's, the two site curves diverge in a way that the heavily stressed site declines more rapidly than the less stressed one. For both sites a growth release can be seen for the most recent years. The latewood percentage curves show trends concurrent with the ring widths. Latewood percentage increases with age on both sites and curves start to differ in the mid 1970's. The less stressed site curve runs inverted to the ring width curve reaching a maximum at around 1986. Further, the heavily stressed site curve is flatter than the less stressed site curve meaning that latewood formation is less pronounced. Additional evidence is provided from the maximum density curve reflecting mainly the cell wall proportions of the terminal tracheid rows in a tree-ring. The heavily stressed site curve reveals for the period between 1976 to 1987 significant lower maximum density compared with the less stressed site for the same period.

A number of authors found radial growth decline along with latewood percentage changes in polluted areas. In a parallel study, Dünisch et al. (1996) investigated spruce trees from the same area and found ring widths < 1 mm with heavily polluted trees, starting in 1973. Similar results were presented by Bitter et al. (1995) showing also a growth decline since the mid 70's. Evertsen et al. (1986) investigated SO<sub>2</sub> polluted spruce trees and found decreased growth rate along with reduced latewood percentage. De Kort (1993) in a comparison of vital and non-vital Douglas firs has found the same results. Reduced wood density caused by SO<sub>2</sub> pollution was reported also for tree-rings of pine trees by Yokobori (1986) and for spruce by Evertsen et al. (1986). Bemmann et al. (1995) reported reduced growth rate along with reduced maximum density of spruce in the Ore Mountains. Other workers have seen no decrease in latewood percentage along with reductions in growth rate (e.g. Grill et al. 1979, Greve et al. 1985, Kim et al. 1987).

#### **12. 3.1.2. Microfibrillar angle, resin ducts and rays**

The trends for microfibrillar angles in earlywood (fig. 12.3D1) and latewood (fig. 12.3D2) differ significantly among sites for the previously mentioned time period. The microfibrillar angles in the secondary wall layers of the heavily stressed trees are smaller compared to the less stressed trees, which means the helix of cellulose microfibrils is steeper oriented with respect to the longitudinal cell axis in the wall layer. Age is the factor that correlates most with a continuous decrease of the microfibrillar angles in radial direction. But growth rate correlates also to a certain degree with microfibrillar angles as demonstrated in a thinning experiment by Erickson and Arima (1974). These workers have measured increased microfibrillar angles along with accelerated radial growth after thinning and fertilization treatment of a Douglas fir plot.

Wardrop and Dadswell (1953) found that the fibril spiral tends to be flatter that have elongated rapidly. They concluded while fibril angle is determined by cell length, it is also influenced by the rate of extension of the cambial initials. In the spruce trees investigated, growth has slowed down expressed as a decrease of periclinal cell divisions (Dünisch et al. 1996) on the heavily stressed site. This has allowed more time for the reduced number of tracheids to elongate, which resulted in smaller angles of the microfibrils.



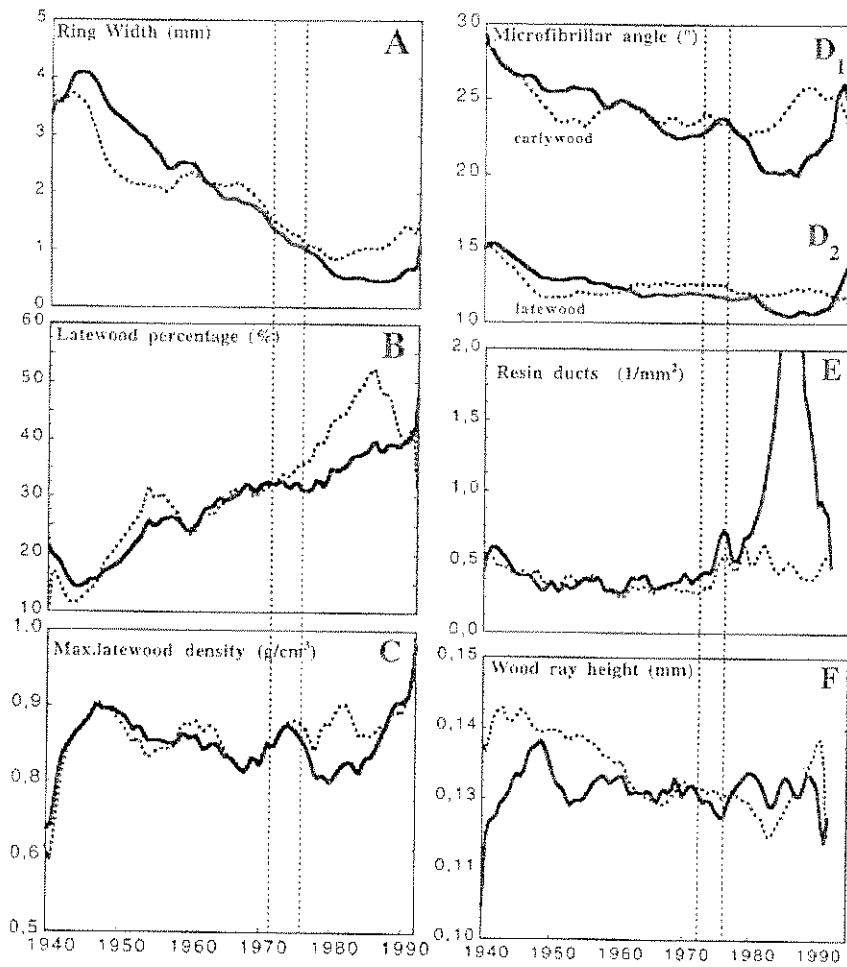


Fig. 12.3: Trends (smoothed curves) of the anatomical parameters. Dotted line: site I (less stressed), solid line: site II (heavily stressed), 10 trees per site, each annual ring measured. Vertical dotted lines: stress interventions (clearcuts) in 1971 and 1976. A: Radial growth rate. B: Latewood percentage. C: Maximum latewood density. D1: Microfibrillar angle earlywood. D2: Microfibrillar angle latewood. E: Axial resin duct density. F: wood ray height

The trends found for microfibrillar angles are similar to those for resin duct density (fig. 12.3E). In fact, the significant increase of the resin duct density in the years starting in the mid 1970's is probably the most significant one (fig. 12.4). This is another indication for a severe impact that has negatively affected the heavily stressed site. Fengel (1987b), Aszmutat and Knigge (1987), and Nimmann and Knigge (1989) reported an increase of axial resin ducts due to air pollution stress. Resin duct formation is normally linked to growth hormones demonstrated by a number of laboratory experiments. Fahn and Zamski (1970) have shown indole-3-acetic acid and 1-Naphtyl acetic acid to be highly involved in generating more resin ducts. Ethylene seems to play a key role for linking exogenous factors with resin duct formation. There are many examples of increased ethylene production following various disturbances or stress. Examples of abiotic stress include chemicals, temperature extremes, drought, hypoxia, mechanical wounding, bending, and the impedance of soil to growth (Abeles et al. 1992) and phytotoxic air pollution.

Radial trends of uniseriate ray heights are not as pronounced and therefore seem to react less sensitive to an environmental change (fig. 12.3F). The ray height curve of the heavily stressed site increased slightly during the second half of the 1970's, which is in accordance with the findings of Halbwauchs and Kisser (1967).

The patterns of the discussed trends lead to the conclusion that a pulse type local disturbance on the heavily stressed site has altered the stem growth as well as the wood structure. Most of parameters reveal a recovery over the recent 10 years indicating that the anatomical parameters have shifted back to normal.

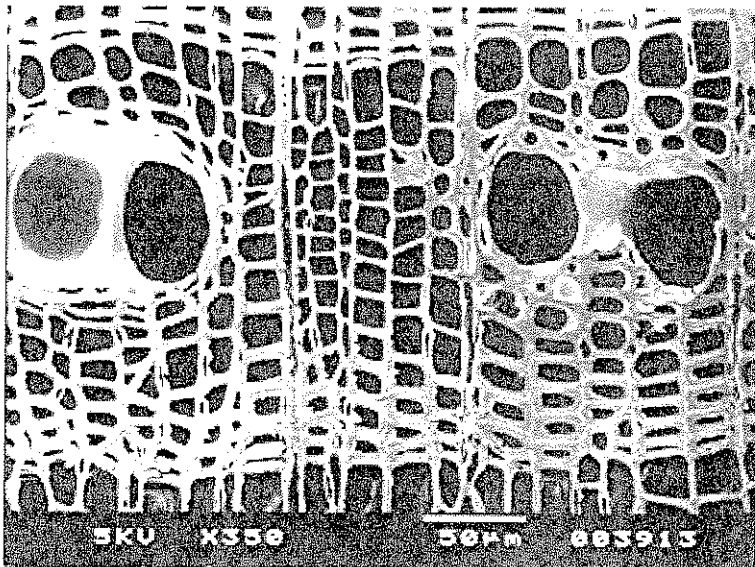


Fig. 12.4: Exemplary REM-Image of a 0.2 mm wide tree ring – frequently observed with the trees on the heavily stressed site. Latewood is poorly developed, resin ducts are more likely.

### 12.3.2. Peroxidase and trends in wood chemistry

#### 12.3.2.1. Peroxidase activity

Peroxidase plays an integral role in cell wall biosynthesis such as in the polymerisation of the monolignols into lignin. It is a key molecule involved in rapid adaptation of the whole plant to changes in the environment (Gaspar et al. 1991). Tab. 12.2 presents the peroxidase activity determined in branches (wood and bark) and the needles of the sample trees of the two sites.

Peroxidase activity of the wood, bark and needles was higher in the trees of site II than of site I, for both crown positions. On both sites, peroxidase was less active at the lower part of the crown. The differences were more pronounced for the wood and the bark than for the needles.

Generally, peroxidase activity indicates stress conditions in the plant. Peroxidase is also known to be involved in the plant defence system and increases of activity were observed with plant exposure to air pollutants (Lehmann et al. 1995). The modifications caused by stress can be assayed on the basis of changes in the substrate peroxidation activity (Castillo et al. 1987, Campa 1991).

Tab. 12.2: Peroxidase activity in branches of Norway spruce from site I (less stressed) and the site II (heavily stressed)

Site	Peroxidase Activity*					
	Wood		Bark		Needles	
	middle crown	lower crown	middle crown	lower crown	middle crown	lower crown
I	9.6	7.0	20.2	15.7	2.3	2.1
II	16.4	13.6	27.2	19.0	2.5	2.8

\* average peroxidase activity in  $\Delta E \text{ min}^{-1} \text{ g}^{-1}$  (dry wood)

Tab. 12.3: Lignin content of Norway spruce wood from site I (less stressed) and site II (heavily stressed), means and coefficients of variation CV are given

Site	Lignin (% dry wood)					
	1938 - 1942		1969 - 1971		1989 - 1992	
	Mean	CV(%)	Mean	CV(%)	Mean	CV(%)
I	29.0	4.9	29.1	3.5	29.2	4.8
II	27.9	4.7	28.0	3.0	28.1	3.3

#### **12.3.2.2. Lignin content and structure**

Although the average lignin content of site I was found to be slightly above the heavily effected site II (tab. 12.3), no significant difference was attested (t-test,  $p > 0.05$ ). In general, the average lignin content of the trees on both sites can be classified as "normal" (Timell 1986). Fengel (1986) found a slight increase in alkali soluble lignin in heavily damaged spruce trees. In contrast, we measured the total lignin by applying DRIFT technique, which did not require chemical pre-treatment of the milled wood. This may lead to the conclusion that possible changes in structure of lignin have increased solubility. Due to the increased activity of peroxidase, an increase of the lignification process would have been feasible, but no such effect was evident in this study. Dünisch et al. (1996) who have also measured wood from highly polluted spruce trees did find an increase of the acid-soluble lignin fraction. In the discussion these authors argue that an increase in phenolic compounds rather than altered lignification might have caused these results. Koch et al. (1996) looked specifically at heavily SO<sub>2</sub>-polluted and highly wind-exposed spruce trees and found slip-plane zones in the wood, which became subsequently infected by wood degrading fungi resulting in simultaneous decay of carbohydrates and lignin in the cell wall.

#### **12.3.2.3. Soluble sugars**

First, fig. 12.5A shows a fairly constant distribution of sucrose across the stem but the site II curve has increased sucrose content over the recent years. Second, a general increase of free glucose (fig. 12.5B) and fructose (fig. 12.5C) starting in the 1960's was observed. While this general increase is related to heartwood formation, the differences in their slopes reveal that the sapwood rings of site II have significantly higher concentrations of the sugars glucose and fructose relative to the less stressed site I. The sugar concentration in the trees of site I start to increase at the sapwood-heartwood boundary with 0.05% and end up with 0.6% sugar concentration in the final 1992 ring, whereas at site II the trees have reached their maximum sugar concentration years earlier (around 1985).

Carbohydrates are subject to seasonal cycling within trees. Their accumulation is at its minimum in spring and early summer when growth is most rapid. In the late summer and autumn, after vegetative growth has slowed or ceased, carbohydrates accumulate in roots, stems, and twigs and in the next spring they are used when growth is resumed (Kozłowski et al. 1991). High amounts of soluble sugars are concentrated during the dormant season. In spring a surplus of mainly glucose and fructose is available for resumption of tree growth. Puls and Rademacher (1986) have shown that the concentration of these sugars was 2 - 5% higher in the outermost tree-rings of spruce determined between November and February whereas in May glucose and fructose concentrations were as low as 0.17 - 0.35% (referred to dry wood). Pollution stress, however, resulted in an increased content of free glucose and fructose in the sapwood (Puls and Rademacher 1986). The altered situation of carbohydrate supply in the cambium of the diseased trees may have led not just to a growth suppression, but also to another ratio of early and latewood cells (Rademacher et al. 1986).

Fengel (1987a) investigated the percentage of mono-, di- and oligosaccharides of squeezed sap. He found increased acidity along with lower concentrations of fructose, glucose and sucrose in severely damaged spruce trees. This finding does not necessarily contradict our previous reported results. The ray parenchyma cells in spruce are

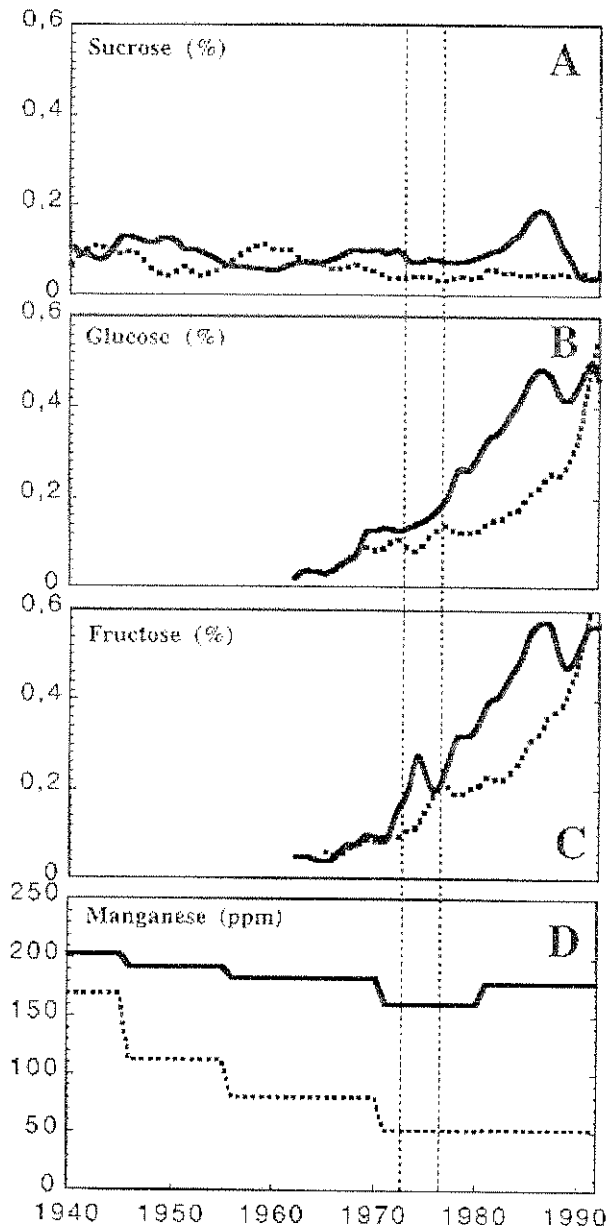


Fig. 12.5: Trends (smoothed curves) of the chemical parameters. Dotted line: site I (less stressed), solid line: site II (heavily stressed). 10 trees per site, each annual ring measured. Vertical dotted lines: stress interventions in 1971 and 1976 (in % per dry weight). A: sucrose, B: glucose, C: fructose, D: manganese.

interconnected with longitudinal tracheids through half-bordered crossfield pits. These pits are tiny and piceoid, typically for the spruce species. The ray parenchyma cells store photosynthates, often in form of starch. Such storage may relate to sudden growth flush and flowering of the tree. One portion of such sudden growth in springtime has been related to hydrolysis of starch in ray parenchyma (Sauter et al. 1973). The release of sugars directly into the water conducting system, which comprises mainly the earlywood tracheids, is of physiological importance. Due to the increased stress situation more soluble sugars and phenolic substances are produced and stored in the ray parenchyma cells which could probably not be released through the pits into the sap stream because of partial closure of these contact pits. Pit blockage through incrustations has been reported repeatedly for highly stressed trees (e.g. Bosshard et al. 1986) leading to increased soluble sugar concentrations in the wood but less sugars in the sap. On the other hand, an intensive study on the structure of pit membranes by Bemmman et al. (1995) did not show such incrustations, which could be related to SO<sub>2</sub> pollution stress.

#### **12.3.2.4. Cation trends**

The rhyolith (podsol) soils are highly acidic with relatively low pH values and low base saturation. The sulphur is mainly accumulated in the upper organic Of-horizon as well as in the A and Bi horizons, which contain higher proportions of organic matter. The present status as well as the history of the soil condition may be reflected to some extent in tree-rings (Baes, McLaughlin 1984, 1987; Bondietti et al. 1990). All trees show a continuous decrease in Ca with the radius - from approximately 900 ppm (pith) down to 600 ppm (bark). This is related to alterations in cation binding capacity in tree-rings of older spruce trees (Momoshima, Bondietti 1990). Ca accumulations rate according to Baes and McLaughlin (1984) was calculated. For the most recent decade (1981 - 1992) the Ca accumulation rate was lower on site II. Inorganic cations such as Ca and Mg are bound to uronic acid containing polymers (pectins and hemicelluloses) in various structures of the cell wall and middle lamella.

On both sites mean Al concentrations increased along the radius from 14 ppm to 22 ppm. No differences between the two sites could be found for the other elements except Mn. Mean Mn concentrations in the tree-rings of the heavily stressed site ranged between 160 to 200 ppm, with exceptional high values of 550 ppm measured in 3 trees on site II. Manganese - like Ca - decreases from pith to bark resulting in a more or less constant Mn/Ca ratio of 0.18 ± 0.01. In contrast, Mn concentrations at site I decline from 170 ppm for the innermost tree-ring, down to 50 ppm for the outermost ones whereas site II remains at a Mn level of 200 ppm (fig. 12.5D). In the 1980's site II has reached the fourfold level of site I. The chemistry of Mn in the soil is related to soil pH. Plants uptake Mn<sup>2+</sup> and the higher the concentration of H<sup>+</sup> in the soil the more Mn<sup>2+</sup> is available. A reduction of the pH by one degree increases the water soluble Mn<sup>2+</sup> by 100 times (Scheffer 1984). In addition, Mn has a very low mobilization factor compared to other metals whose solubility is pH dependent. It was shown that Mn concentration in tree-rings provide a tool for examining long-term changes in soil pH due to acid deposition or ecological disturbances (Guyette et al. 1992). The soils of our research sites are sufficiently acid buffered and Mn is available for the trees. Therefore, high concentrations of Mn were found in the tree-rings. The higher levels of Mn in the tree-rings on site II combined with the fact that the decline over age is less than on site I, could be a reflection of the historical soil pH dynamic. Due to the long-termed higher SO<sub>2</sub> deposition soil acidification on site II is more advanced than on site I.

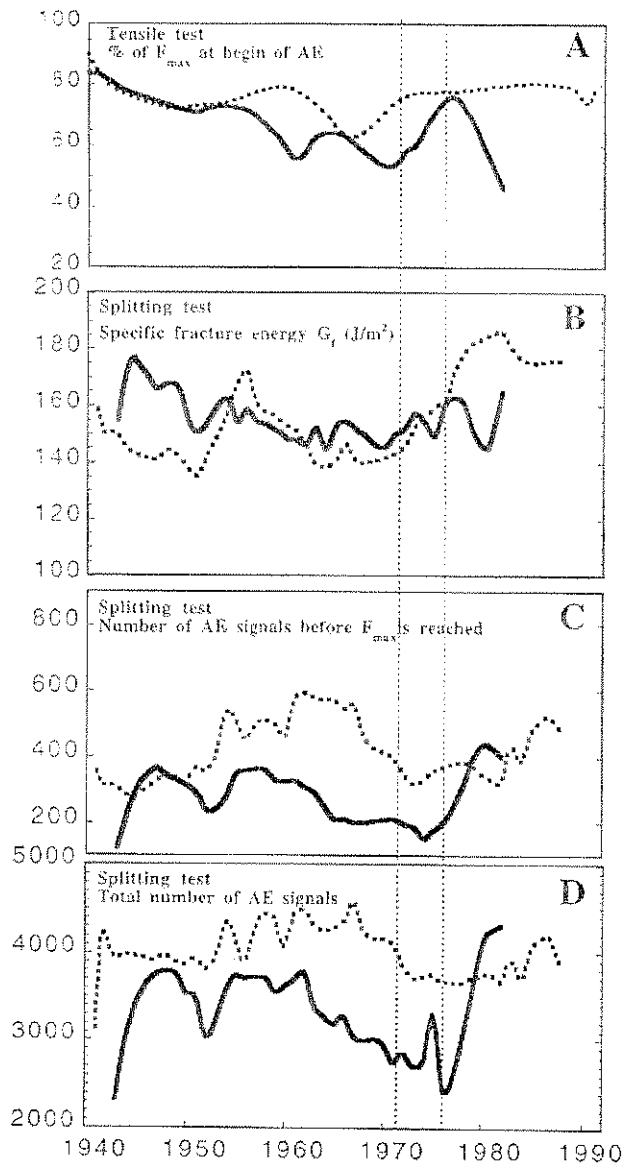


Fig. 12.6: Trends (smoothed curves) of the mechanical parameters. Dotted line: site I (less stressed), solid line: site II (heavily stressed). A is represented by 4 trees for each site and figures through E include 6 trees for each site. Vertical dotted lines: stress interventions in 1971 and 1976. A: Tensile test - percentage of force at which acoustic emissions (AE) signals occur (4 trees per site). B: Splitting test - specific fracture energy ( $G_f$ ) (6 trees per site). C: Splitting test - AE signals before  $F_{max}$  is obtained (6 trees per site). D: Splitting test - total number of AE signals until final fracture (6 trees per site).

### **12.3.3. Trends in mechanical properties**

#### **12.3.3.1. Tensile tests**

The percentage of force (% Fmax) at which acoustic emissions (AE) signals occur (fig. 12.6A) as well as the total number of AE signals until final fracturing takes place were recorded in tensile tests with specimens cut from earlywood. Between the years 1940 and 1960, the acoustic emission activity started for both sites at similar stresses. But later (around 1976) the specimens of the heavily stressed site II exhibit a sharp downswing in the percentage of force at which AE starts (fig. 12.6A), while the site I curve stays more or less constant during this period (Moritz 1996). Similar trends of decreasing AE activity were reported by Niemz et al. (1990) and Lühmann (1991).

The onset of AE activity at lower stresses and the higher numbers of AE events could indicate reduced structural resistance against the initiation of microcracks as well as an increased number of such crack initiation sites in that time period. But as a restriction, we were only able to complete AE measurements with tensile tests on 4 trees per site and not with all tree-rings available.

No differences were found in tensile strength (not shown) between the two sites, which coincides with the finding of other authors who have measured tensile or bending strength of spruce and pine wood specimens from heavily stressed areas (Glos, Schulz 1986, Niemz et al. 1990, Bemmann et al. 1993, 1995, Wimmer 1993). Schober and Wagenführ (1991) reported a decline of the modulus of elasticity in bending as well as compression strength and explained this change with the occurrence of short-fiber cracking as a typical feature of wood from pollution stressed trees. This hypothesis could not be verified in subsequent investigations by Bemmann et al. (1995). Similarly, no pronounced changes of strength properties of wood from SO<sub>2</sub> polluted forest sites in the Ore Mountains were found in a recent study by Bäucker et al. (1996).

#### **12.3.3.2. Splitting tests**

Determination of the specific fracture energy as well as AE during the newly developed splitting tests was done on a much better data basis than the tensile tests. Six trees per site were completed with almost each ring measured. Beyond 1982 many tree-rings were too narrow for the rather sophisticated preparation of the splitting samples. Fig. 12.6B presents the trends for specific fracture energy (Gf) values obtained with the splitting tests. Between the years 1940 and 1976 variations of Gf ranged from 140 to 180 J.m<sup>-2</sup> for both sites with only minor site differences. But after 1976, Gf of the heavily stressed site curve separates from site I and indicates that the wood formed during this time has failed at a lower energy level equivalent with a more brittle fracture. This is consistent to a certain degree with the AE results (fig. 12.6C and 12.6D) obtained with the splitting test. The AE signals collected before Fmax is reached – equivalent with the occurrence of the first crack (fig. 12.6C) – increased significantly on the heavily stressed site in the mid 70's. A similar trend was observed with the total number of AE (fig. 12.6D).

In summary, it was possible to measure time trends of mechanical properties of trees from two different stressed forest sites. The results show that the trees of the heavily stressed site (II) underwent a significant change in the 1970's, which has changed the structural integrity of the wood formed during that period.



### 12.4. Conclusions

The main focus of this investigation was to measure simultaneously anatomical, chemical and mechanical parameters on identical specimens and at a high temporal resolution in order to indicate stress situations, particularly high SO<sub>2</sub>-pollutions.

While there are no obvious differences for the years between 1940 and 1970 - even at high levels of SO<sub>2</sub> pollution (Nebe et al. 1995a) - the pulse type growth interventions in the 1970's have caused many tree-ring parameters to shift. But in the year of sampling, 1992, many of the parameters have recovered and shifted almost back to normal levels.

With the presented measurements we were able to reach annual resolution for many parameters. Trends in wood anatomy clearly showed that the two sites started to differ in the mid - 70's, with a recovery period starting in the mid 80's. The significant parameters were ring width, latewood percentage, maximum wood density, microfibrillar angles and finally axial resin duct density. Among the chemical parameters, the soluble sugar trends responded most significantly to the presumed stress impacts that happened in the mid 70's, while the other chemical parameters did not differ between the two sites. Cation trends were investigated at a 5-year or 10-year resolution, and particularly Mn trends indicated a long-term change in soil pH due to acid deposition, possibly linked to environmental disturbances. As far as the measured mechanical indicators are concerned, detection of specific fracture energy and acoustic emissions obtained during splitting experiments (Stanzl-Tschegg et al. 1995) were found to synchronize well with the changes that took place in the anatomy and chemistry.

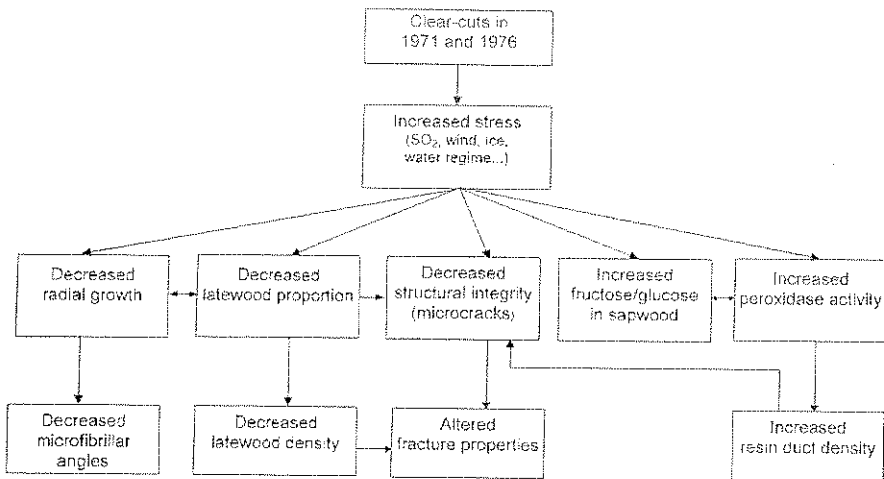


Fig. 12.7: Summary of the stress responses (including SO<sub>2</sub>) measured in the tree rings of the East Ore Mountains Norway spruce

Fig. 12.7 summarizes the most significant changes that were seen in the heavily polluted trees of site II, compared to the trees from site I. In the years 1971 and later in 1976 (Schilke, pers. comm. 1996) severe thinning and clear-cut activities took place south-east to the site II, which should have caused subsequent changes of the environmental conditions at this site. Many parameters must have changed: radiation, water regime, stronger winds, ice damage, nutrient cycling, and deposition rates of gaseous SO<sub>2</sub>. All these factors together caused severe stresses in the trees. Consequently, many parameters show sharp up or downswings in the years after the clear-cuts took place. But even with this comprehensive set of data, no causal relationships between the measured responses and SO<sub>2</sub> pollution can be stated. The measured responses are likely to be associated with SO<sub>2</sub>-pollution but it cannot be excluded that the measured results have no conclusive relationship with SO<sub>2</sub> at all. It has to be kept in mind that air pollution may affect growth processes without losses in needle retention and vice versa, with severe needle loss and crown damage caused by factors others than air pollution (Sterba 1996). Forest decline – even in severely SO<sub>2</sub>-polluted areas – is a multifactorial phenomenon and may rarely be explained by single parameters (Halbwachs 1988).

The resin duct density increases in the 1970's could be a consequence of high metabolic activity, since resin duct formation is linked to growth hormones such as ethylene (Wimmer and Grabner 1997). The increased number of normal and traumatic resin canals has made the wood more inhomogeneous (fig. 12.4), which could at least partially explain increases of acoustic emission events recorded during the mechanical experiments. The results of the mechanical tests indicate that structures of reduced resistance against the onset of microcracking were formed and the number of such crack initiation sites has increased since the 1970's. We think that the acoustic emissions recorded before F<sub>max</sub> during the splitting test - equivalent with the onset of the first crack - is especially sensitive for such structural inhomogeneities as the case with the increased number of resin ducts.

Finally, many of the measured parameters indicate a recovery during the most recent years, which is in accordance to the stress-response hypothesis by Härtel (1976). The mentioned stress factors acted like a pulse that caused the physiological system of the trees to shift. Since we only measured trees that survived the stress intervention back in the 1970's, these trees seemed to recover and reverse the negative trends bringing the system back to normal. Sulphur deposition rates have been declining since 1989, which ran parallel with declining sulphur contents in the foliation of several tree species (Nebe et al. 1995a). A slight recovery in tree growth since the mid 1980's was reported by Nebe et al. (1996), which is also in accordance with our findings.

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