Tensile Fracture and Failure Behavior of Technical Flax Fibers

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ABSTRACT: The apparent tensile strength of technical flax fibers was determined in single-fiber tests at various clamping lengths (20, 40, and 80 mm) and the outcome was compared with literature data. It was demonstrated that the strength of flax at each clamping length obeyed the two-parameter Weibull model. The failure mode and sequence were studied *in situ* (i.e., during loading) by SEM and acoustic emission (AE). The failure sequence (axial splitting of the technical fiber along its elementary constituents, radial cracking of the elementary fibers, multiple fracture of the elementary fibers) concluded reflected the hierarchical build-up of the flax bast fibers. To the above failure events AE amplitude ranges were assigned. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 90: 3638–3645, 2003

Key words: biofibers; failure; fracture; acoustic emission; Weibull distribution

INTRODUCTION

Natural fibers (agrofibers) are considered as potential candidates to replace man-made reinforcing fibers in both thermoplastic and thermoset composites. Renewed interest in the use of natural fibers is triggered by various aspects (environmental consciousness, global sustainability, biodegradability, legislative actions, etc.). One should also keep in mind that the density-related (specific) stiffness and strength characteristics of natural fibers are competitive with traditional reinforcing fibers, especially with those of glass.[1] The major drawback of natural (annual) fibers is their quality, which depends on many external factors (climate, soil, plant cultivation, fiber extraction, etc.). The potential use of flax fibers in polymer composites is well demonstrated (Mieck,[2] Stamboulis et al.,[3] Bledzki and Gassan,[4] and references therein). The widespread use of flax is attributed to its easy production (especially in Europe and in the United States) and to the outstanding mechanical performance of the related bast fibers. However, the quality and thus the ultimate mechanical properties of flax fibers also depend on intrinsic parameters. Flax fiber is characterized by a very complex composite structure. Technical bast fibers (length ~ 1 m, apparent diameter: 100–200 μm) separated from the flax plant consist of elementary fibers (length ~ 50 mm, apparent diameter: 10–30 μm). The polyhedron-shape elementary fibers overlap one another at a rather large length interval. They are held together by a "natural glue" on polysaccharide base (pectin and hemicellulose). The elementary fibers are composed of a very thin primary cell wall, a secondary cell wall (dominating the cross section), and a lumen (small, open channel in the center of the elementary fiber). The secondary cell wall contains crystalline cellulose microfibrils and amorphous hemicellulose. The hierarchical structure of the elementary flax fiber is even more sophisticated because the microfibrils are bundled in mesofibrils that are spirally oriented along the fiber axis.[5] Figure 1 depicts the microstructure of the flax fiber.

Based on Figure 1 it is intuitive that the ultimate tensile properties of flax fibers are largely experimentally indeterminate—given the wide scatter range—and thus the related properties can be determined only by statistical methods. The Weibull description, which has proved to be suitable for several man-made fibers (Pan et al.[6] and references therein), has also been adopted for flax fibers.[5,7] Nevertheless, a reliable description of the tensile strength as a function of clamping length is still missing for technical flax fibers, although knowledge of pertinent data is of paramount importance to advance prospective work with these composites. Note that the interfacial shear strength is often determined in single-fiber composites by the

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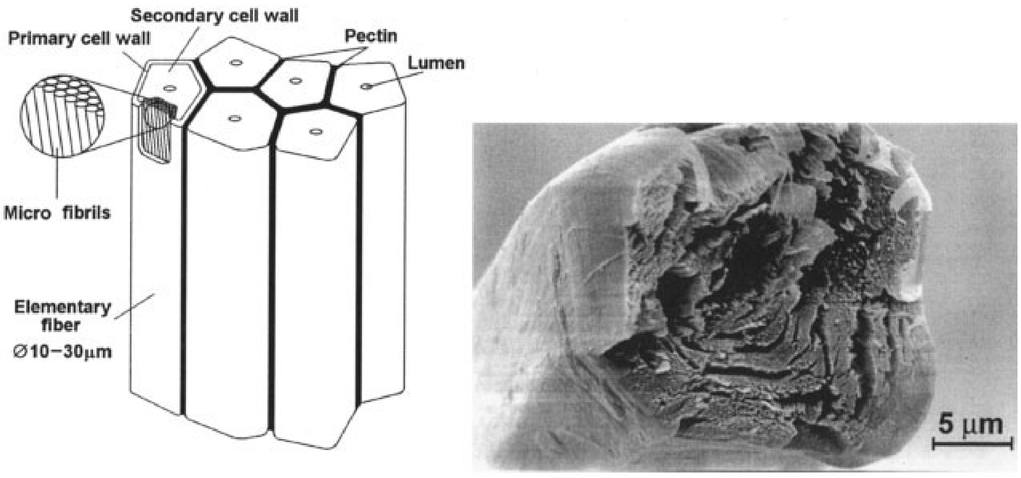


a.) b.)

Figure 1 (a) Scheme of the microstructure of the technical flax fiber and (b) SEM micrograph from the fracture surface of an elementary fiber.

fragmentation technique (used also for flax-reinforced systems; see, e.g., Zafeiropoulops et al.[8]). The related data reduction necessitates knowledge of the fiber strength at very low clamping length. This can hardly be determined experimentally but may be estimated by a suitable mathematical (e.g., Weibull) description.

The technique of acoustic emission (AE) has proved to be a suitable tool for studying the failure behavior of composite materials.[9] Applying AE to flax fiber–reinforced systems it is necessary to study the fracture and failure behavior of the flax fibers separately; otherwise, no reliable discrimination among the AE parameters is possible. Assigning well-selected AE characteristics to the failure events occurring in flax fibers may be very helpful to check differences in the failure mode because of their different treatments (alkali, acetylation, etc.; see Bledzki and Gassan[4]).

The aims of this work were to give a reliable description of the tensile strength of flax fiber as a function of clamping length, and to clarify the related failure mode by AE technique. The latter task also covered the assignment of suitable AE parameters to given failure mechanisms.

EXPERIMENTAL

Tensile tests

Technical fine-quality flax fiber was delivered by Hungaro-len Ltd. (Komárom, Hungary). Single-fiber fracture tests with simultaneous AE monitoring were performed at clamping lengths of 20, 40, and 80 mm, respectively. The lower threshold (i.e., 20 mm) was attributed to the size of AE sensors (diameter 10 mm; Micro-30D, Dunegan, USA). Flax fibers were cut and fixed by an epoxy glue onto a paper frame at the required distance. Before their tensile fracture on a Zwick 1474 machine (Germany) the paper frame was cut, allowing the undisturbed loading of the fiber. Figure 2 depicts the testing configuration used.

Tensile testing occurred at ambient temperature with a deformation rate of 1 mm/min. At each clamping length 50 fibers were tested. The apparent diameter of the flax fibers was determined before the tests in a light microscope (Olympus, Japan).

Acoustic emission (AE)

Figure 2 also indicates how the AE microphone was attached to the flax fiber. Burst-type AE signal characteristics (amplitude, rise time, event width, ring down counts, etc.) were collected during loading of single flax fibers by a Defectophone NEZ 220 device (AEKI, Budapest, Hungary). To eliminate noise from

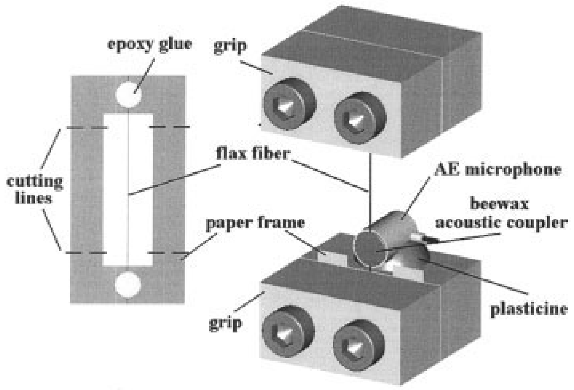


Figure 2 Experimental setup of the single-fiber fracture test.

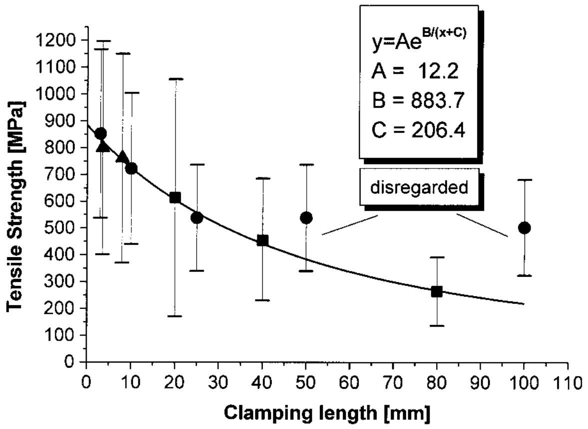


Figure 3 Tensile strength as a function of clamping length for flax fibers. Designation: ■, present data; ▲, data from Stamboulis et al.[3]; ●, data from Bos et al.[5]

the surrounding environment, the threshold value was set for 15 dB.

Scanning electron microscopy (SEM)

SEM was used to elucidate the failure sequence in flax fibers. Individual fibers were glued onto paper frames, similar to that of the tensile testing, although at a clamping length of about 5 mm. Before the SEM inspection (JEOL JSM-5400, USA) during loading, the fiber assembly was sputtered by gold alloy (Balzers SCD 050 equipment, Liechtenstein). The fiber was stretched by a custom-built device that was placed in the SEM. After locating the initial failure site the entire fracture process was monitored by taking serial SEM micrographs.

RESULTS AND DISCUSSION

Tensile strength

The tensile strength (σ) of the flax fibers was determined by dividing the breakage force (defined from the load–displacement curves of the single-fiber tensile tests) with the apparent circular cross section. The σ values as a function of the clamping length are displayed in Figure 3, which also contains the literature data from Stamboulis et al.[3] and Bos et al.[5] During fitting, however, the σ-values reported by Bos et al.[5] at high clamping length were disregarded because the related data not only differed considerably from ours, but also were in disharmony with the structure of the flax fibers. Recall that the length of the elementary fibers is limited, which suggests a steady decrease with increasing fiber length. Figure 3 clearly shows that the scatter range increases with decreasing clamping length.

As noted earlier the mechanical properties of fibers can satisfactorily be described by a two-parameter Weibull equation that expresses the cumulative probability $[W(\sigma)]$ of the tensile strength:

$$W(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{a}\right)^{b} \right] \qquad (1)$$

where a and b are the so-called scale and shape parameters, respectively.

The a and b parameters were determined by the maximum likelihood estimation at each clamping (see Table I). To check whether the Weibull distribution is correct the results were confirmed by the Kolmogorov–Smirnov goodness of fit.[6,10] The principle of this approach is as follows: the n measured data (σ_i, $i = 1, \ldots, n$, $n = 50$) are ordered in an ascending sequence. If the maximum difference ($d_{\max}$) between the theoretical distribution (Weibull distribution in the present case) and the statistical distribution [calculated using $(i - 0.5)/n$] does not exceed a critical value (d_{crit}; calculated for significant level $\alpha = 0.05$ according to $d_{\text{crit}} = 1.36/n^{1/2}$), then the theoretical model can be accepted as a good representation of the data distribution.

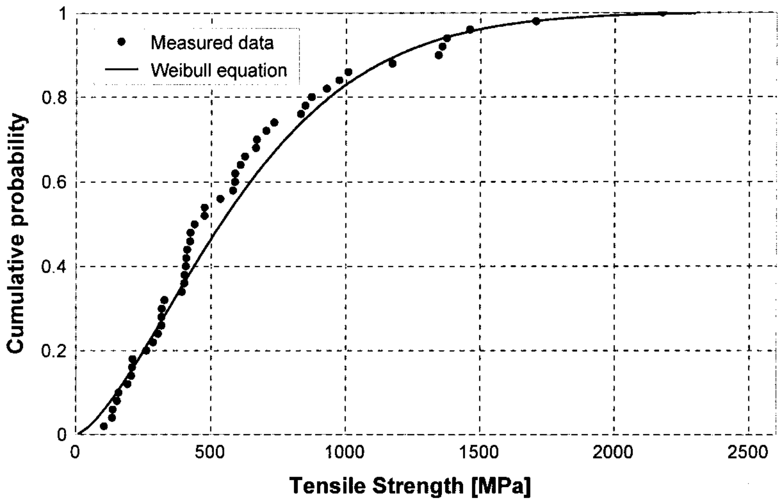
At $n = 50$ measured data $d_{\text{crit}} = 0.19$. Given that at all clamping lengths $d_{\max} < d_{\text{crit}}$, the Weibull distribution correctly represents the tensile strength distribution of the flax fibers. This can also be seen in Figure 4, which shows the cumulative probability of σ at 20- and 80-mm clamping lengths, respectively.

During single-fiber fracture two types of load–displacement curves were observed. One curve displays a monotonous load increase [Fig. 5(a)], whereas the other curve displays a sudden load drop superimposed on a monotonous increasing force [Fig. 5(b)]. In fracture mechanics such a load drop is termed "pop-in." The "pop-in" phenomenon is usually caused by crack bifurcation during which an earlier secondary (satellite) crack becomes the major one.[11] Considering the complex microstructure of flax several changes in the plane and direction of the running crack can be expected. Note that Figure 5 also contains the amplitudes of the collected AE signals.

TABLE I
Tensile Strength (σ), Weibull Parameters, and Goodness of Fit Parameter ($d_{\max}$) Determined for Each Clamping Length[a]

Clamping length (mm)	σ (MPa)	Weibull parameters a	Weibull parameters b	$d_{\max}$
20	613 ± 442	684.7	1.5	0.089
40	454 ± 231	515.4	2.1	0.049
80	264 ± 127	299	2.2	0.040

[a] For the units a and b consider eq. (1).



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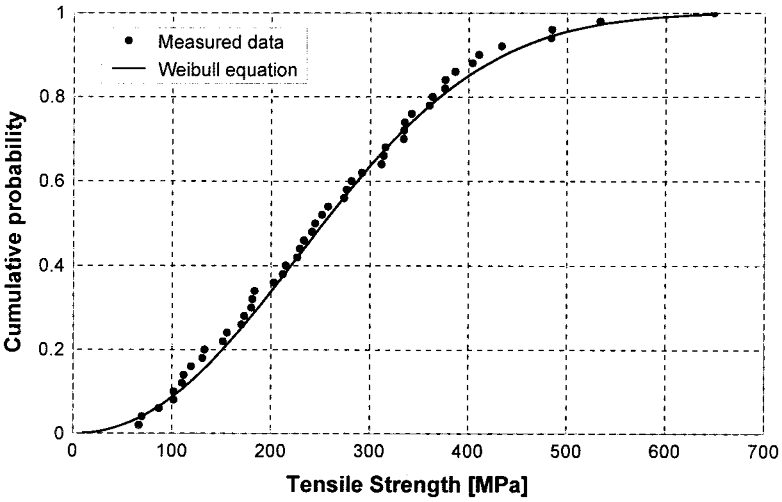


b.)

Figure 4 Weibull fitting on the tensile strength data of flax fibers measured at a clamping length of (a) 20 mm and (b) 80 mm.

Tensile failure

SEM inspection revealed that the failure of flax starts by axial (longitudinal) fibrillation [Fig. 6(a)]. Fibrillation is the result of a combined effect of the Poisson ratio of the fiber and its microstructure. Recall that the microfibrils within the elementary fibers are spirally aligned along the fiber axis under an angle of about 10°. This causes the breakup of the polysaccharide glue (amorphous pectin, hemicellulose), the strength of which is considerably lower than that of the crystalline entities (elementary fibers, microfibrils). Axial fibrillation occurs along the boundaries of the elementary fibers. The onset of load drops triggers radial (transverse) microcracking in the elementary fibers [Fig. 6(b)]. The same also happens near the maximum load during single-fiber tests. This transverse microcracking along with the fibrillation process redistribute the stresses locally, creating a weak site that may be quite remote from the initial failure site. Here a tearing-type process occurs [Fig. 6(c)], which involves a zigzag crack advance throughout the split, fibrillated elementary fibrils. Failure is completed by multiple fracture of the elementary fibers and their microfibrils, which may take place along a rather long fiber length [Fig. 6(d), (e)].

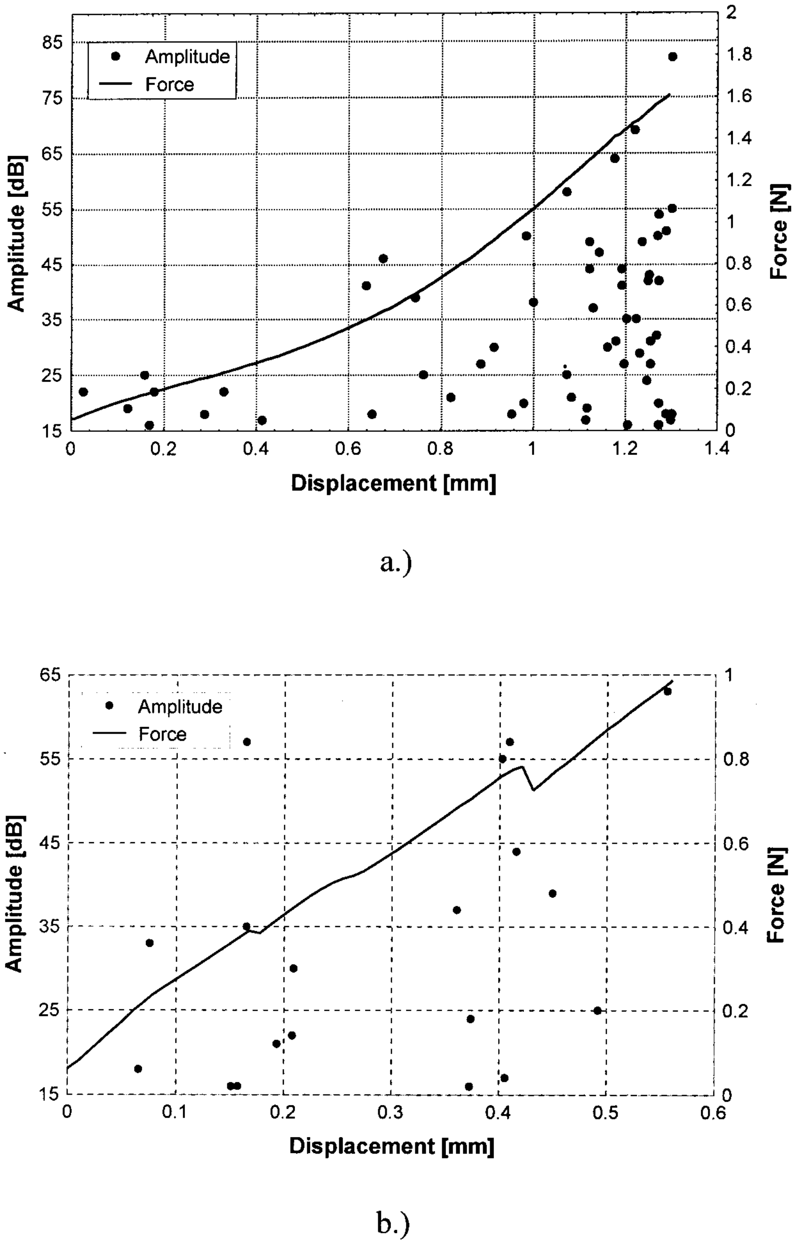


Figure 5 Characteristic force versus displacement traces for technical flax fibers along with the collected AE amplitude values.

The AE amplitude values in Figure 5 already indicated that small amplitudes appear in the initial fracture, whereas high AE amplitude signals are emitted close to the final fracture of the fibers. Attention should be paid to the fact that AE amplitude discrimination proved to be a suitable tool to assign them to the various individual failure events.[12] To obtain reliable statistics the test results (3 × 50 measurements) were grouped in three clusters based on the related σ values (i.e., <270, 270–450, and >450 MPa). AE amplitudes from the last second before fracture were extracted from the stored AE files and AE amplitude histograms were constructed for each cluster (cf. Fig. 7).

The amplitude histograms in Figure 7 show three peak regions. AE amplitudes with <35 dB are always present. This peak can thus be attributed to the axial debonding, that is, splitting [cf. Fig. 6(a)]. AE amplitudes between 35 and 60 dB appear only after a given load level. Accordingly, this AE amplitude range can be assigned to the radial (transverse) cracking of the elementary fibers [cf. Fig. 6(b)]. The high-amplitude AE signals (>60 dB) based on their occurrence are linked to the fracture of elementary fibers and their fibrils. A scheme of the failure sequence with the corresponding AE amplitude ranges is given in Figure 8.

Despite great efforts to find further correlations between AE signal characteristics and individual failure

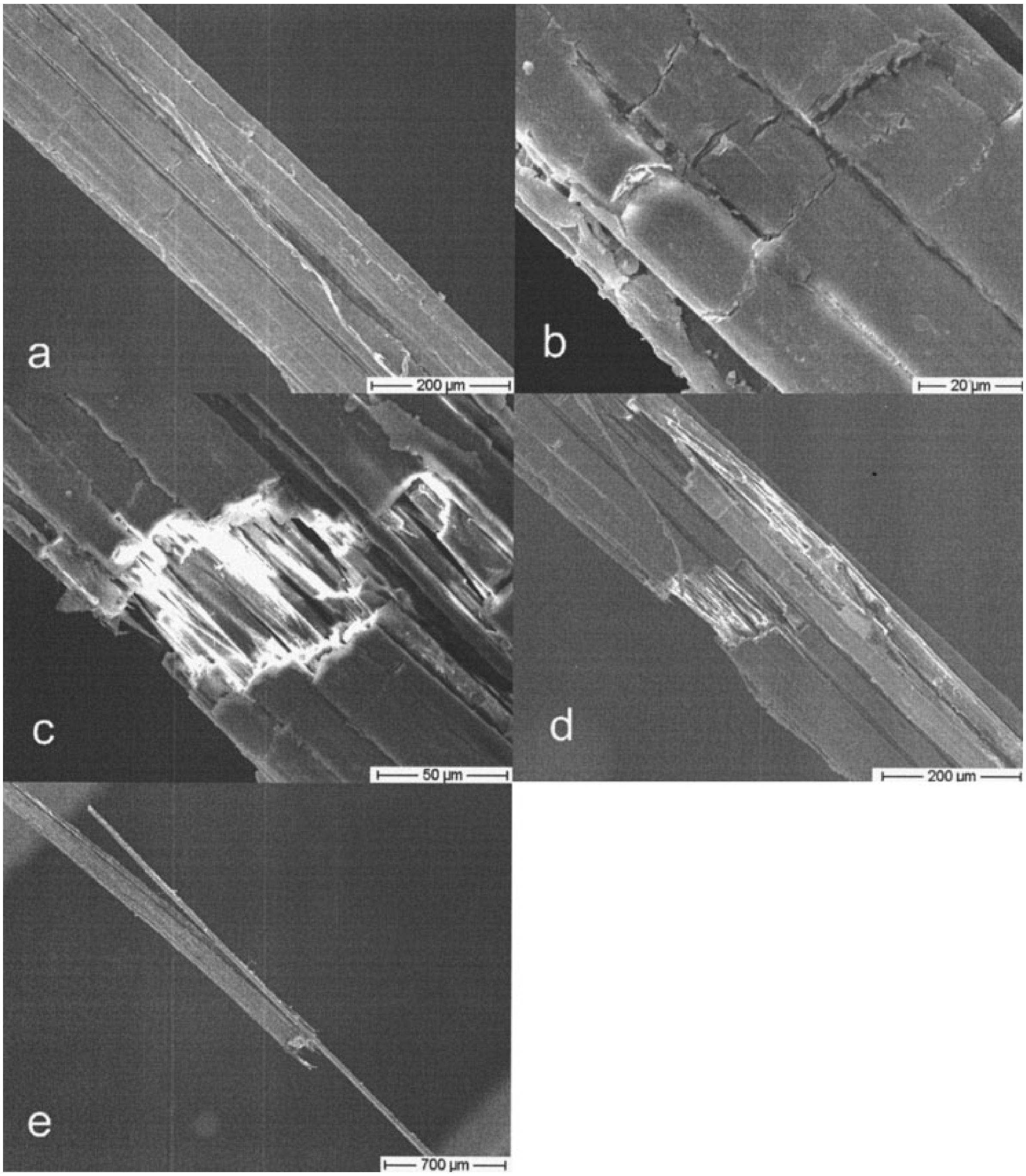


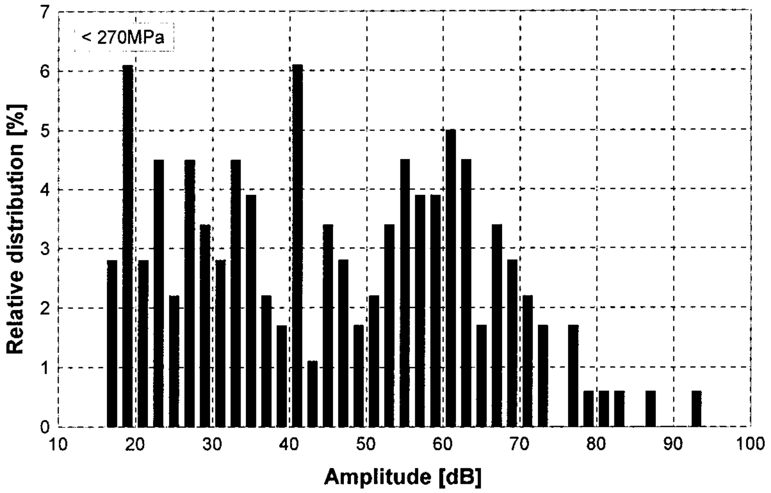
Figure 6 Failure sequence in a technical flax fiber: (a) axial (longitudinal) debonding and fibrillation along the elementary fibers; (b) radial (transverse) cracking in the elementary fibers (amplified effect attributed to stress concentration); (c) "tearing-type" fracture within and through the elementary fibers; (d) and (e) long-range fracture completed by fracture of the elementary fibers and their constituting microfibrils.

events in flax fibers, no reliable correlation could be found. Nevertheless, the above AE amplitude discrimination may be very helpful to study the fracture of flax fibers after various treatments.

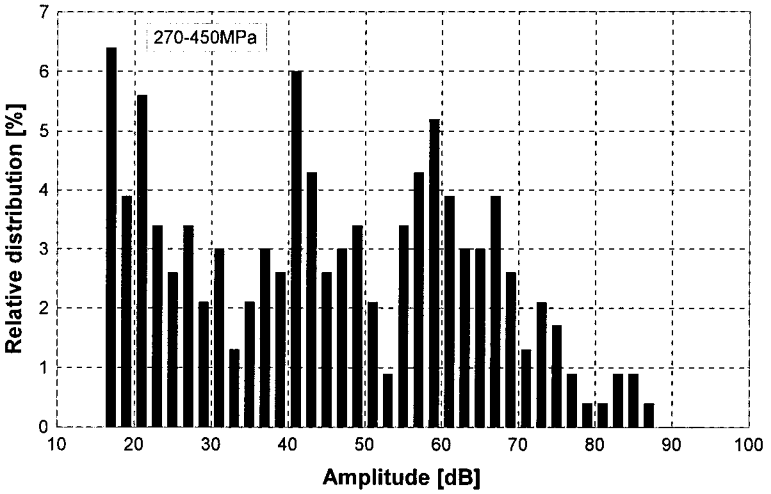
CONCLUSIONS

Based on this work dedicated to assess the tensile strength of flax fibers as a function of clamping length and to clarify their failure mode and sequence, the following conclusions may be drawn:

- The tensile strength of flax fibers decreases with increasing clamping length and at a given clamping length the distribution of the ultimate strength can be described by a two-parameter Weibull equation.
- The failure sequence detected by *in situ* SEM inspection is as follows: (1) axial (longitudinal) split-



a.)



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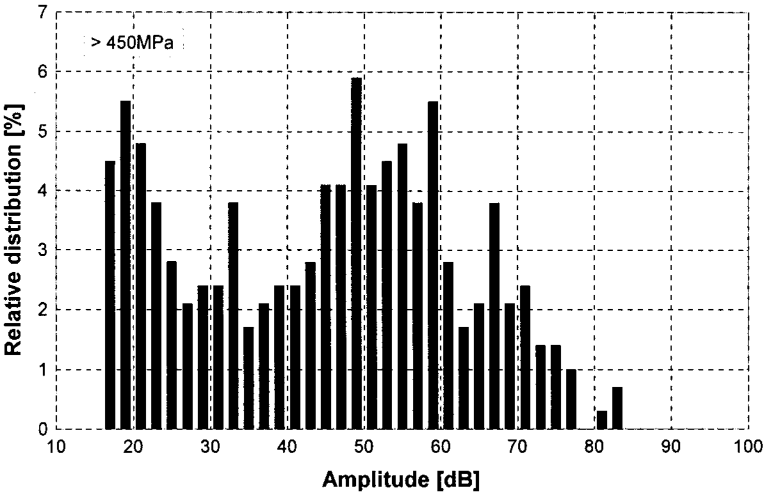

c.)

Figure 7 AE amplitude histograms for flax fibers having tensile strength values (in MPa): (a) <270, (b) 270–450, and (c) >450.

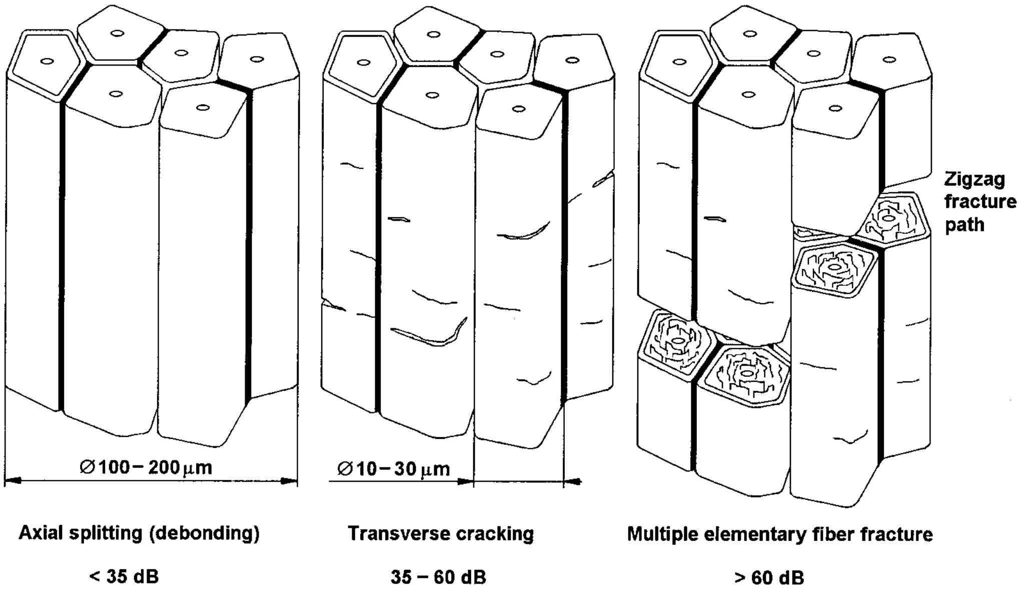

Figure 8 Failure sequence of a flax fiber and the related AE amplitude ranges deduced. Note: elementary fibers held together by pectin are indicated by bold line.

ting along the boundaries of the elementary fibers, (2) radial (transverse) cracking of the elementary fibers, and (3) fracture of elementary fibers and their microfibrils. The above events are superimposed on one another. Nevertheless, we succeeded in assigning an AE amplitude range for each of the above failure mechanisms [(1): <35 dB; (2): 35–60 dB; and (3): >60 dB].

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