Developing a micro-laboratory for dynamic in-situ characterisation of single wood fibres: toward high-performance sustainable composites

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Executive Summary

This ÅForsk project was run between 2020 and 2023. We have developed a micromechanical tensile testing method, which can test short single natural fibers such as wood fibres.
We have achieved the following in the project:

- Used a hypersensitive load cell for single wood fibre testing;
- Established protocol for testing single wood fibres;
- Obtained the tensile properties of wood fibres conditioned at different humidity levels;

One of the objectives could not be fully achieved – in-situ single natural fibre testing in a focused ion beam and scanning electron microscope (FIB/SEM). The original idea was that we perform tensile test and simultaneous imaging using SEM, and after fibre breakage we use the FIB cutting to reveal the cross-section for area measurement. However, due to the bulky size of the tensile testing machine and the size limit in the FIB/SEM chamber, this could not be realized. However, we developed an alternative method to reveal the cross-section area of the single fibres.

Sustainability aspects of the outcome of the project
The project contributes to sustainability in two significant ways:
Firstly, replacing, at least partly, the glass and carbon fibres in the composites with natural fibers means less carbon footprint.

Secondly, this project paves the way to computation driving material design of natural fibre composites, thus save time and resources compared to the extensive experimental tests by the traditional approach. Natural fibers need always be chemically modified, in order to be used in a composite. How the chemical modification influences the mechanical properties of the fibers is critical to the final performance of the composites. Traditionally, researchers first conduct modification on a large quantity of fibers and then prepare macroscopic samples and finally perform mechanical testing. The entire procedure demands a large amount of material and time. With our new methods, we can make very fast testing directly on the modified fibres, which largely shortens the testing procedure and time. More importantly, the results can feed into computational models to predict the properties of

final composite and to optimize material design, which further increase the time and resource efficiency.

Contribution to education and the Swedish workforce in fibre reinforced composite materials:

Master's student Emelie Seignér finished her Master's project on this topic, and employed as research assistant in the project. She is now employed as a research engineer at the Composite Lab of Chalmers University of Technology.

New collaboration between Chalmers and Swedish industry:

Thanks to the promising results obtained from this project, we also established collaboration with two companies: Tetra Pak and Juteborg Sweden AB (a SME). In a long term, we would like to see that the method developed in the research can be used by industry on exciting research questions in the future.

Technical report

Wood fibres provide exciting opportunities for future sustainable, lightweight, low-cost, and high-performance products. However, the properties of wood fibre-based products are not on par, partly due to the fact that wood fibres tend to absorb water, and consequently their mechanical properties deteriorate. How different humidity levels affect the properties of wood fibres is not yet fully understood, particularly on the single fibre level. Today, the experimental methodology development remains challenging due to the severe difficulties with the small size and complex structure of wood fibres. In this project, we aim to develop a first-of-its-kind testing platform, which can provide insights into the influence of humidity on wood fibres. The fundamental understanding gained in this project will greatly accelerate the development of high-performance wood fibre products. Below I summarise the results we have obtained in the project.

1) The hypersensitive load cell for micromechanical testing

A hypersensitive load cell was acquired (from Kammrath & Weiss GmbH) (Fig 1). It has a force measurement range between 0.00001 N to 1 N, and maximum cross-head displacement rate 20 μm/s. The measurement range fits well to perform micromechanical tensile testing on single wood fibres.

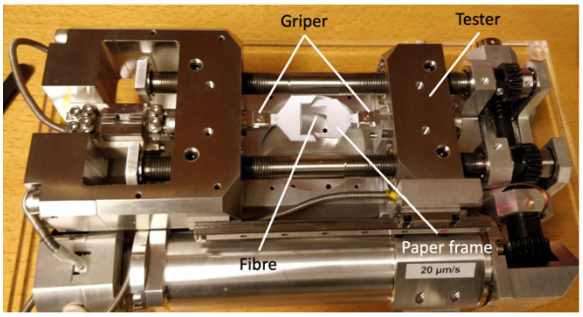


Fig 1. *Photo of the testing rig of the hypersensitive load cell with a single fibre sample mounted on a paper frame.*

2) Measurement of tensile properties of commercial carbon fibres

In order to test the equipment and validate the measurement protocol, commercial carbon fibres (T800 from Toray) were used. They have continuous long fibres, thus easy to handle; and they have known dimensions and mechanical properties, thus convenient to validate. Stress strain curves were obtained (Fig 2), and stiffness was calculated. It was found that the stiffness value fits well with the data provided by the manufacturer (278 ±33 GPa vs. 294 GPa). The results confirm that the equipment is well-calibrated and suitable to measure individual fibres.

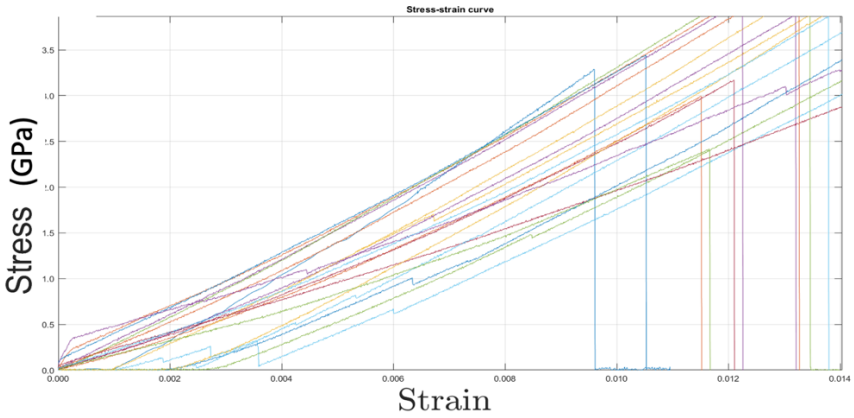


Fig. 2 *Stress strain curves of a few dozen commercial carbon fibres T800.*

Several potential challenges were revealed by the testing using the well-defined carbon fibres. First, although most of the fibres exhibit a stiffness value (slope of the curves) within a very narrow range (which is expected due to the relatively uniform properties of the commercial carbon fibres), there are a few outliers (Fig. 2). One of the reasons is the variety in the fibres' cross-sections. Despite the well-controlled manufacturing process, the fibres vary in the cross-section morphology and areas (Fig. 3). This distribution contributes, at least partially, to the difference in stress, when a nominal fibre area was used for the calculation. Second, the kinks in some of the curves are attributed to the fact that two or more fibres were mounted on the same testing sample. (The fibres are extremely fine, ~5 μm in diameter, which makes separation and manipulation of them difficult). Both challenges were encountered in testing of wood fibres.

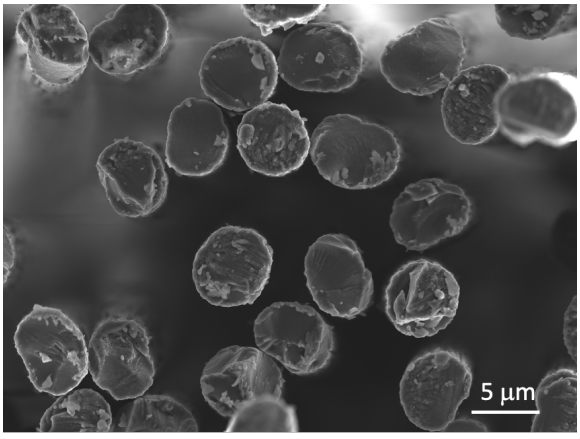


Fig. 3 *Scanning electron microscopy micrograph showing the cross-section of carbon fibres T800. Note the non-uniform cross section areas of individual fibres.*

3) Chemical treatment of wood fibres

We selected thermal mechanical pulp (TMP) fibres as the wood fibre source, due to the ease to access and the potential of providing better compatibility to polymers (due to the relatively high lignin content) and thus better performance of the composites.

The fibres were first sodium treated with 0.1 mol/L $NaHCO_3$ solution, then washed with water and acetone, followed by chemical treatment using alkyl ketene dimer (AKD) (a long chain alkyl with 36 carbon atoms was used, which allows good hydrophobicity) and with isopropanol at 90°C for 1 hour, where AKD react with the hydroxyl groups on the cellulose (Fig. 4), and finally washed with water and ethanol.

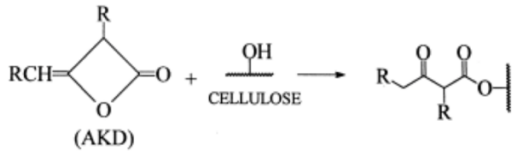
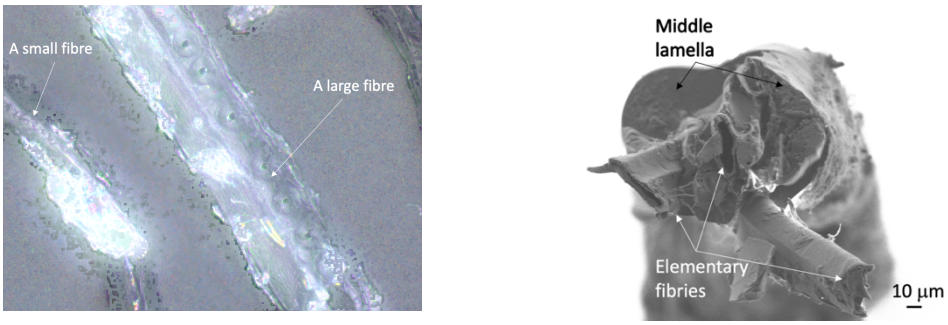


Fig. 4 *Alkyl ketene dimer reacts with hydroxyl groups on the cellulose*.

In order to reveal the size and morphology of the as-received TMP fibres, optical microscopy was used. The size of the TMP wood fibres varies largely. Fig. 5 shows an example of a large and a small wood fibre. Besides the natural variations due to the growth period (early wood vs. late wood) and other factors (weather and nutrition), some of the large fibres are fibre bundles that consist of several elementary fibres. The structure of fibre bundles is revealed clearly by scanning electron microscopy of the fractured cross-section (details in next section).



(a) (b)

Fig. 5 *Variation in wood fibres.* a) *Optical microscopy micrograph of two wood fibres with very different sizes. b) Scanning electron microscopy micrograph of the fractured surface of a TMP fibre. Note this apparent single fibre consists of a few elementary fibres.*

4) Preparing the single fibres

A wood fibre "lump" (weighed ~2 mg) was separated from a fibre cake and merged in distilled water in a beaker. A piece of aluminium was placed on top of the beaker to prevent water vapour to escape, and then the beaker was placed in an ultrasonic agitation (Branson 1510 ultrasonic cleaner) and agitated for 1,5 hours (increasing fibres breakage was observed with prolonged agitation time).

With two pair of tweezers, single fibres were pulled out from the fibre lump taken from the water. The wood fibres were dried and selected using a stereo microscope (Zeiss Stereo

Discovery.V20 Stereo Microscope). Fibres that were bent, contained more than one fibre or had other defects were discarded.

A frame of aluminum foil was designed, (Fig. 6) to hold the fibre and be fixed onto the tensile device to carry out the tensile test on single fibres. One drop of super glue was placed on both sides of the hole of the aluminium frame to adhere the fibre.

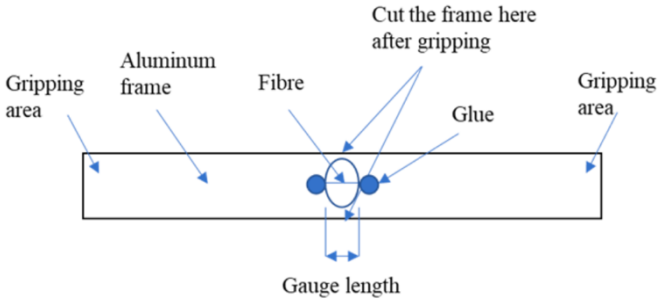


Fig. 6: *Aluminum frame for single fibre specimen for axial tensile testing.*

When the glue had dried, the aluminium frame was placed under the stereo microscope. The gauge length was measured on each single fibre test sample (Fig. 7). If found bent, twisted or misaligned (under sample preparation), the fibres were discarded. Then the tensile test samples were conditioned for 0%, 50%, and 100% relative humidity at 20 °C for 72 hours.

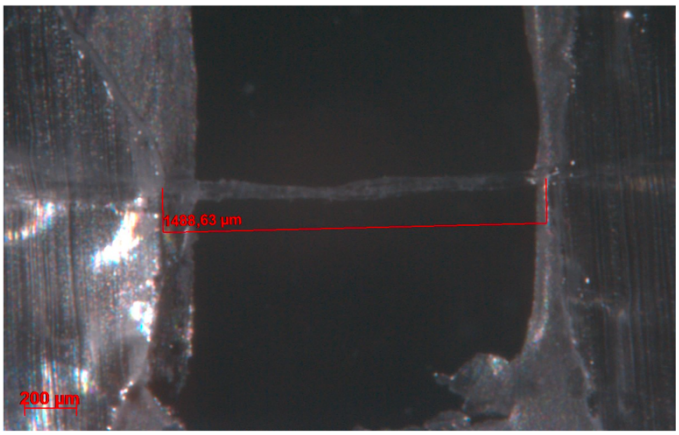


Fig. 7: *Measure the gauge length of a single fibre tensile test sample using the stereo microscope.*

5) Single fibre tensile testing

The Kammrath and Weiss Materials Testing Module was used to carry out the tensile tests. PC-controlled microprocessor hardware (DDS4) and a software package (MDS) was used to collect data from the tensile tests. ASTM standard (C1557) on fibre tensile testing was followed.

The tensile tests were conducted by using the tensile machine under a constant speed of displacement of 5 µm/s at room temperature. Before the test could start, the aluminum frame was cut on the sides next to the fibre so that the frame was only connected through the fibre. During the tensile test, time, displacement and force were monitored simultaneously as well as recorded with the help of the program MDS. When the test was done, e.g., after the fibre had been pulled apart, the aluminium frame was saved for measuring the cross-section using SEM (Fig. 8).

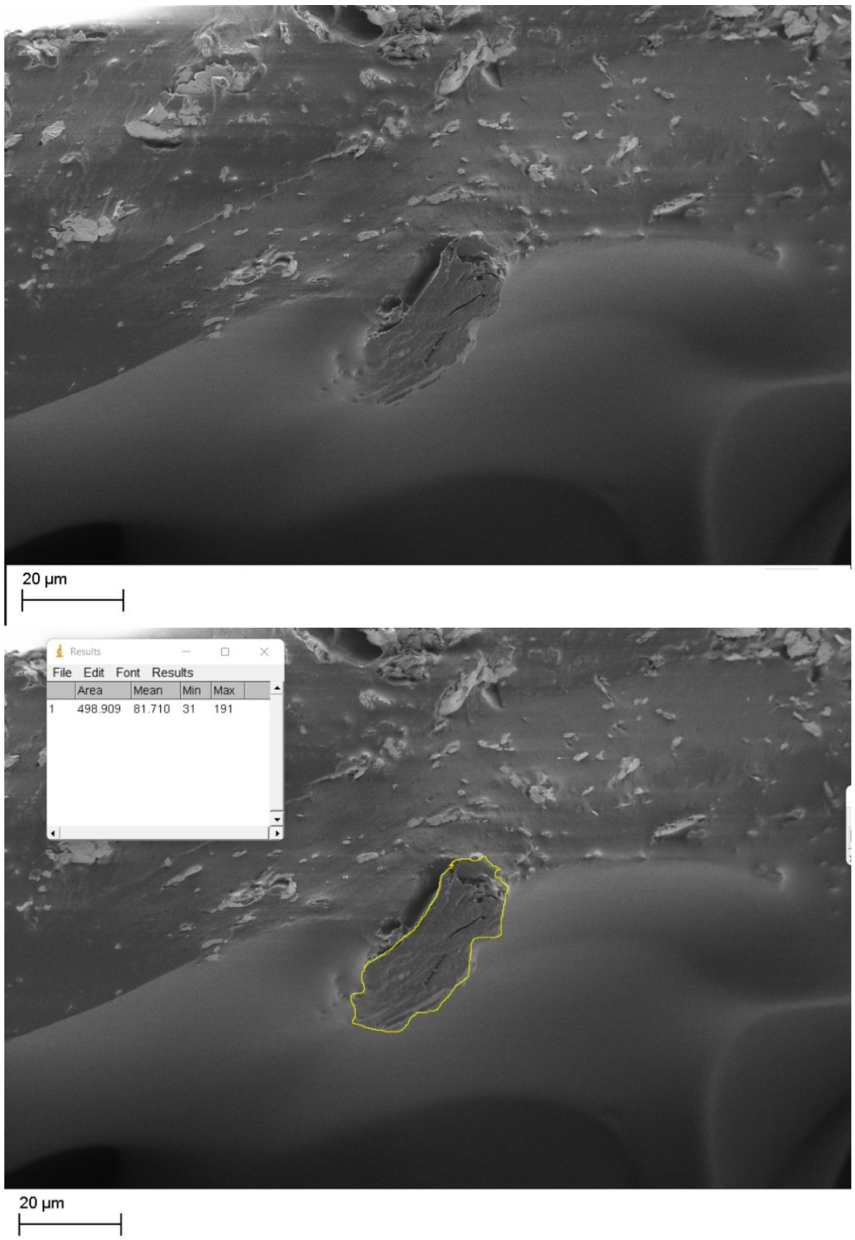


Fig. 8 *Scanning electron microscopy micrograph showing a fibres cross-section (above); and using ImageJ to define the cross-section area and to obtain the cross-section area of the fibre.*

6) *Tensile properties of single wood fibres conditioned at 0%, 50%, and 100% relative humidity*

For each humidity condition at least 22 fibres were successfully tested. Some representative results were shown in Fig. 9. Note the strain were calculated, considering the measured system compliance. The tensile properties of the single fibres in three different humidity conditions is summarised in Table 1.

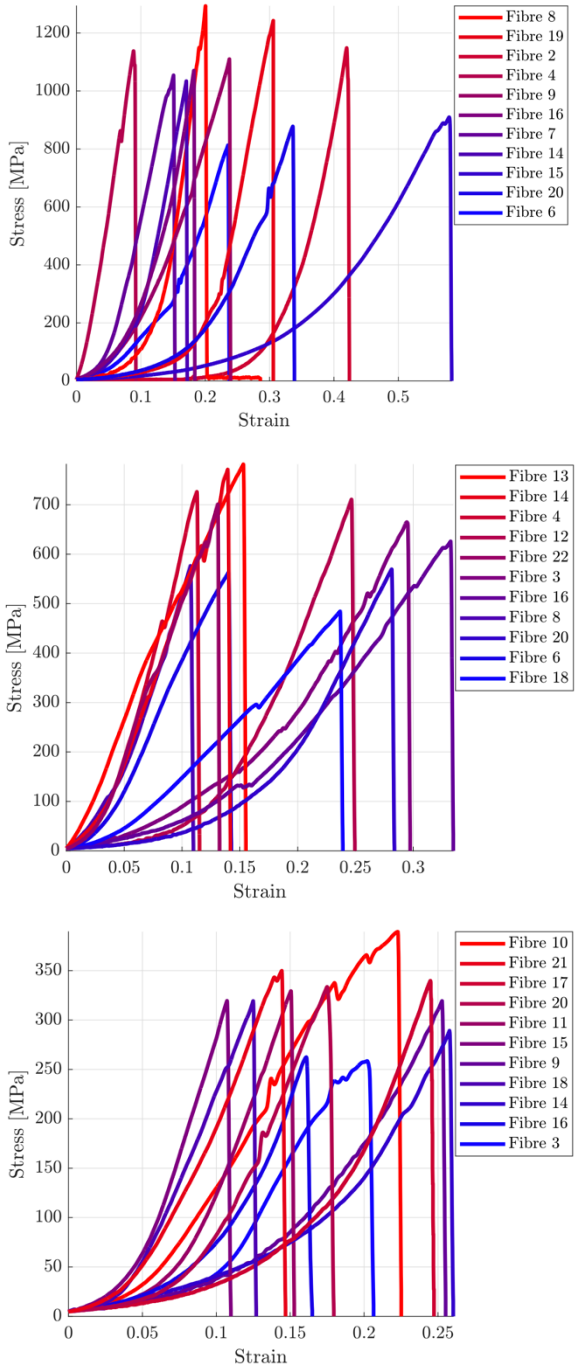


Fig. 9 *Stress strain curves single fibres conditioned at 0% (above), 50% (middle), and 100% (bottom). Note the big difference in the range of the strain axis.*

Table 1: *Ultimate tensile strength with standard deviation for fibres conditioned in the different relative humidity level.*

Condition of the fibres (relative humidity)	*Ultimate tensile strength (MPa)*
0%	*864±237*
50%	*506±166*
100%	*227±113*

Table 2: *Young's modulus with standard deviation for fibres conditioned in the different relative humidity level.*

Condition of the fibres (relative humidity)	*Young's Modulus (GPa)*
0%	*9.08±3.36*
50%	*4.78±2.31*
100%	*2.83±1.5*

7) Weibull statistics

The Weibull distribution is usually used to model mechanical properties that are widely dispersed and to measure the variation of the strength of natural fibres. Fig. 10 shows the Weibull statistics graph for the different humidity conditions.

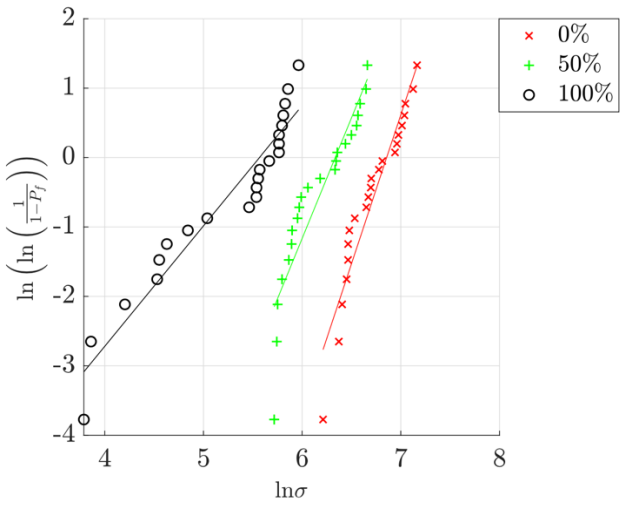


Fig. 10 *Weibull statistics graph of fibres differently humidity conditioned at 0%, 50% and 100% .*

Stress strain curves single fibres conditioned at 0% (above), 50% (middle), and 100% (bottom). Note the big difference in the range of the strain axis. The calculated normalization root-mean-square deviation (NRMSD) are relatively small in all cases between 0.068 –0.0923.

Concluding remarks

We have developed a micromechanical tensile testing method, which can reproducibly test short single natural fibers such as wood fibres, revealing the nature variation of the wood fibres, and potentially the hygroscopic (when absorbing water vapor) swelling of the fibres and the consequent changes in the mechanical properties.