



# INVESTIGATION OF FLAX YARNS FROM ITALIAN PAINTINGS USING AFM MECHANICAL CHARACTERIZATION

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## ART AND ENGINEERING: how much do they have in common?



Know the structure/chemistry  
 -> preserve the objects

Study of the ageing/degradation mechanisms

-> extreme environmental conditions, biological attacks...

The materials are mixed in a complex system

-> the compatibility between materials of different nature is often problematic



Know the structure/chemistry  
 -> materials with suitable and reproducible characteristics

Study of the ageing/degradation mechanisms

-> biocomposite must be long-lasting and with a predictable behaviour  
 -> ageing tests under controlled conditions

The materials are mixed in a complex system

-> compatibility between materials of different nature is often problematic

## CULTURAL HERITAGE: problems linked to their nature



Paintings on **flax**  
canvas

- Fragile
- The history of their conservation is unknown (art galleries, private collections, churches...)
- Test less destructive and invasive as possible
- Relationship with the institutions

**Atomic Force Microscopy in PF-QNM mode** -> small samples required, mechanical properties of single fibres, localization and identification of defects/fractures/particles

**Tensile tests**-> small sample required, quality index of the state of the fibre

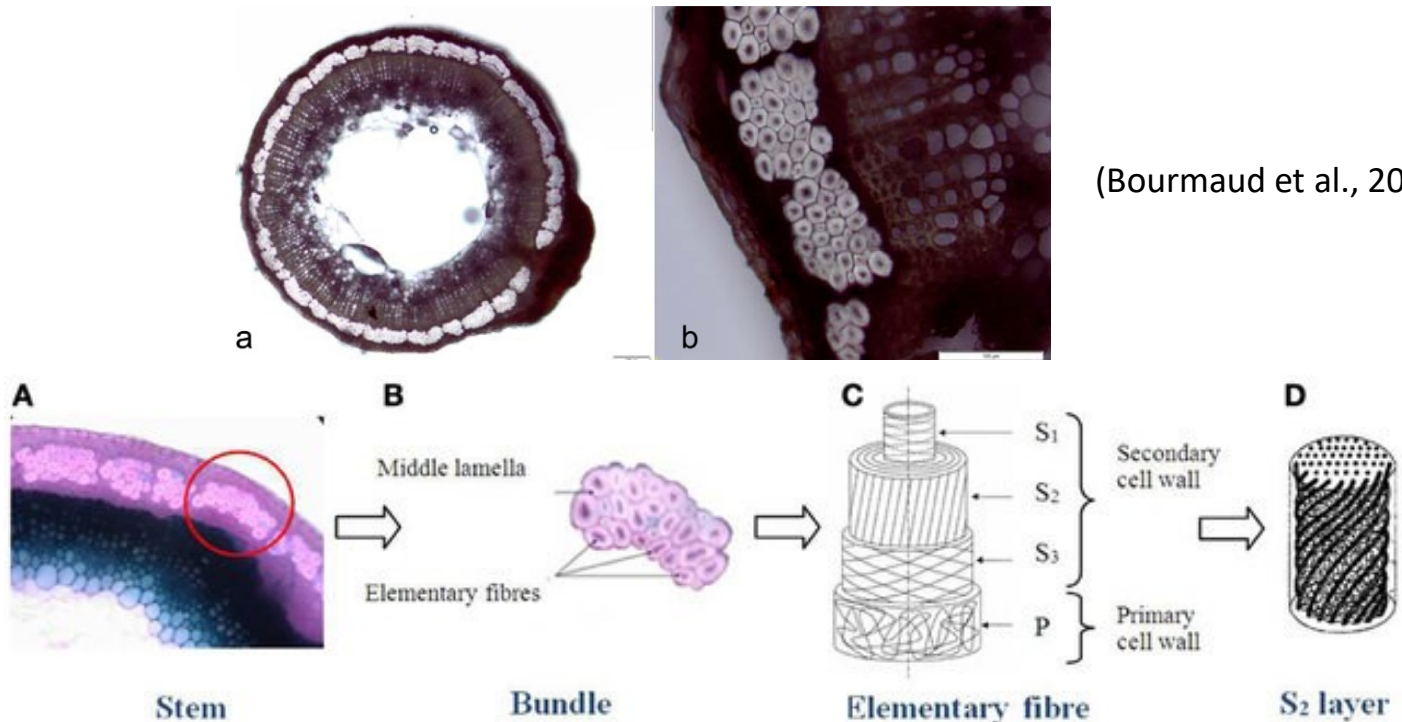
## THE STRUCTURE OF FLAX fibres



- Height of the plant around 100 cm
- Rapid growth (sowing in March, harvesting in July, retting process in August)

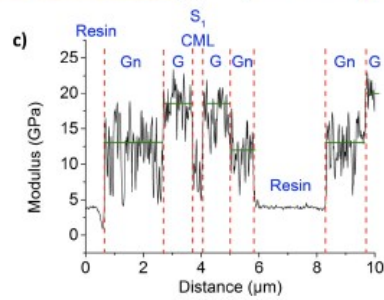
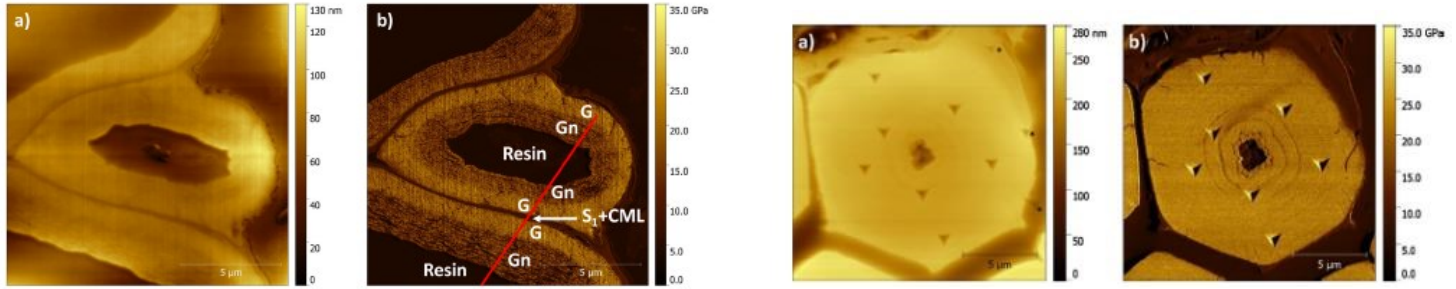


- Flax fibres extracted from the phloem region of the stem
- 10-40 elementary fibres grouped in a bundle. Elementary fibres have a hierarchical structure



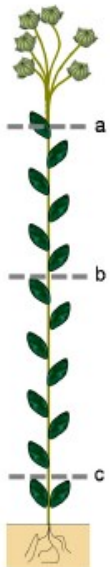
(Céline et al., 2014)

# An existing protocol of PF-QNM for plant fibres analysis

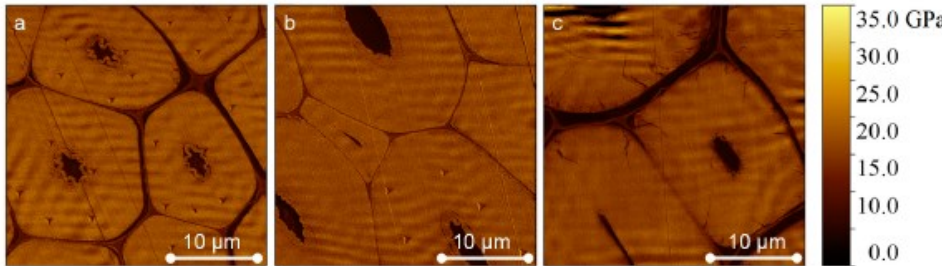


(Arnould et al. 2017)

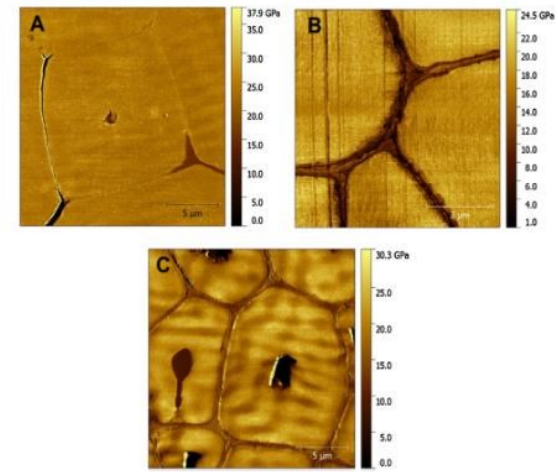
The AFM PF-QNM has been already used to study plant fibres and their layers !



<p><u>Average thickness</u> G : <math>6.1 \pm 1.1 \mu\text{m}</math> <u>Average modulus</u> G : <math>17.3 \pm 1.7 \text{ GPa}</math></p>	<p><u>Average thickness</u> G : <math>7.1 \pm 2.5 \mu\text{m}</math> <u>Average modulus</u> G : <math>18.4 \pm 1.3 \text{ GPa}</math></p>	<p><u>Average thickness</u> G : <math>8.2 \pm 1.5 \mu\text{m}</math> <u>Average modulus</u> G : <math>17.7 \pm 1.4 \text{ GPa}</math></p>
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(Goudenhooff et al. 2018)



(Bourmaud et al. 2019)

## AIM OF THIS STUDY

Investigate **degradations and ageing effects** on **ancient flax fibres**  
-> help to **predict some degradation mechanisms for other fields** where plant fibres are employed (engineering, biocomposites, design...)

Use the **AFM PF-QNM as a new tool** to investigate **cultural objects** with several advantages (small sample preparation, microsampling required, semi-destructive tests)

Contribution to a **global study on ageing** of the flax canvas support and how it can evolve

## Tommaso Sciacca, « Crocifissione » and « San Francesca Romana »

Dated 1765 - 1795 **sec. XVIII** Dated 1700 - 1799 **sec. XVIII**



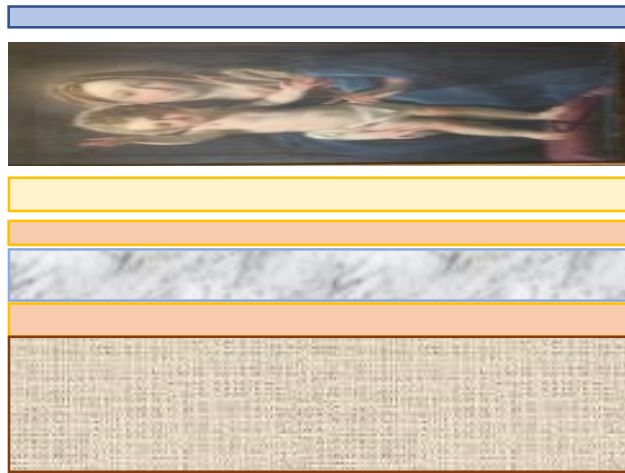
**Tommaso Sciacca** (Mazara del Vallo 1734-  
Lendinara 1795)->

- He worked in Palerm, Rome and Venice
- His master was Agostino Masucci but he became the pupil of Antonio Cavallucci
- He worked abroad (Polony)
- He had some important commissions as canvases and frescos for several churches

Antonio Cavallucci, *Levitation of Saint Thomas of Cori* (1786)



The influence of Antonio Cavallucci, is visible in the painting composition and cromaticity



varnish

pigmented layer

pigment preparation

animal glue

gypsum

animal glue

canvas (flax)

## Sec. XVI

*(Dunkerton & Spring, 1998)*

*(D'Anna, 1993)*

## The different layers of a painting

## Sec. XVI-XVII

*(D'Anna, 1993)*

varnish

pigmented layer

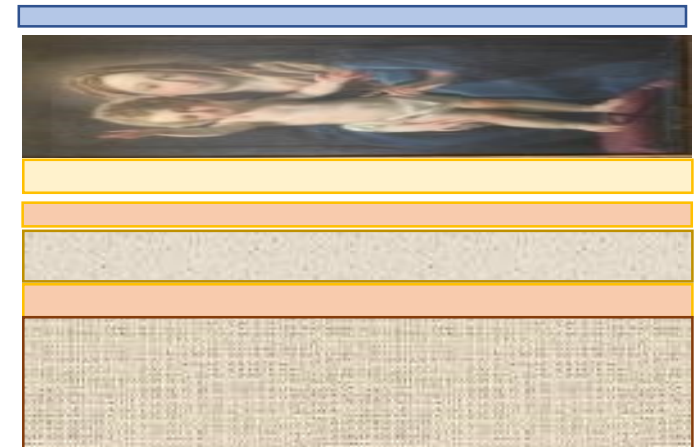
pigment preparation

animal glue

flour, oil, lead white

animal glue

canvas (flax)



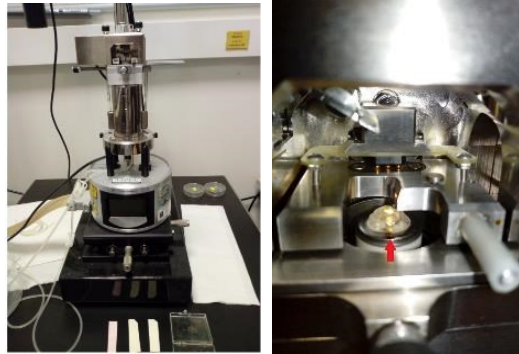
## Sec. XVII

Try to eliminate the glue layer to protect the back of the canvas and directly use the preparation on the flax canvas



## METHOD

Multimode 8 AFM  
Instrument  
(Bruker Corporation, Santa  
Barbara, USA)



Tip: RTESPA 525 (Bruker)

Parameters used

- PeakForce Frequency: 2 kHz
- Tip radius: 18-44 nm
- Spring constant: 130-200 N/m
- Peak force amplitude: 75 nm
- Scan speed: 8  $\mu\text{m/s}$  max.



Flax  
yarn



Embedding  
(Epoxy low viscosity  
resin R1078, Agar  
Scientific)



Oven at 60°C for a  
night – polymerization  
of the block



Cut  
ultramicrotome  
(Diatom diamond knife)



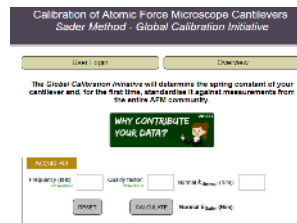
## Calibration

Sader method



- Spring Constant

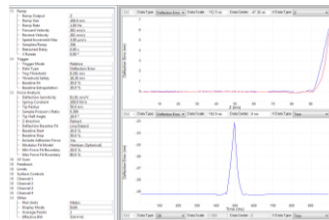
<https://sadermethod.org/>



Sapphire



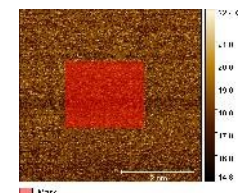
- Deflection sensitivity
- Sync distance QNM



HOPG



- Tip radius
- HOPG  $\rightarrow$  indentation  
modulus of 18 GPa

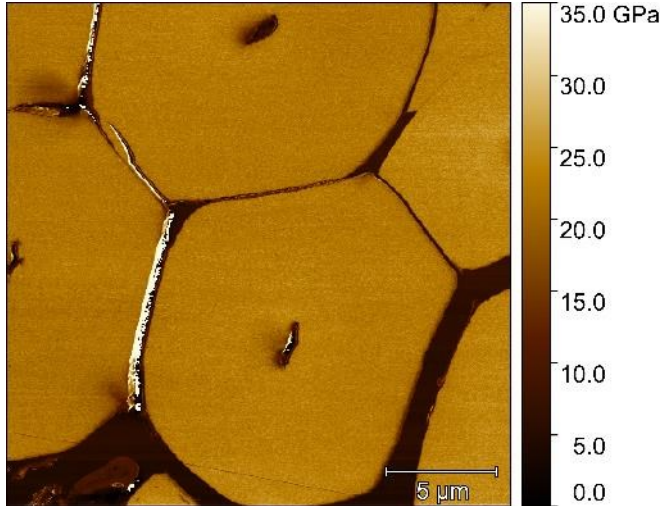


sample



Maximum load:  
200 nN

## RESULTS AND DISCUSSION

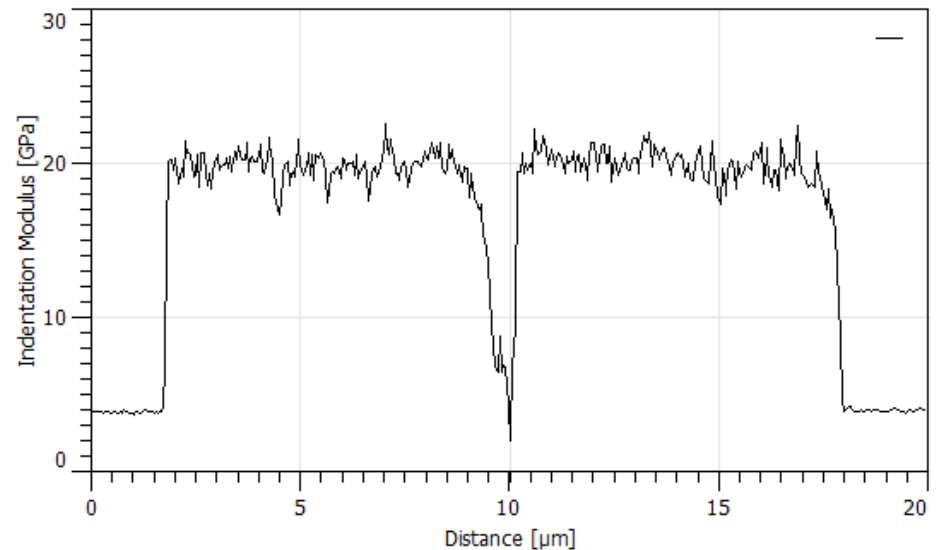
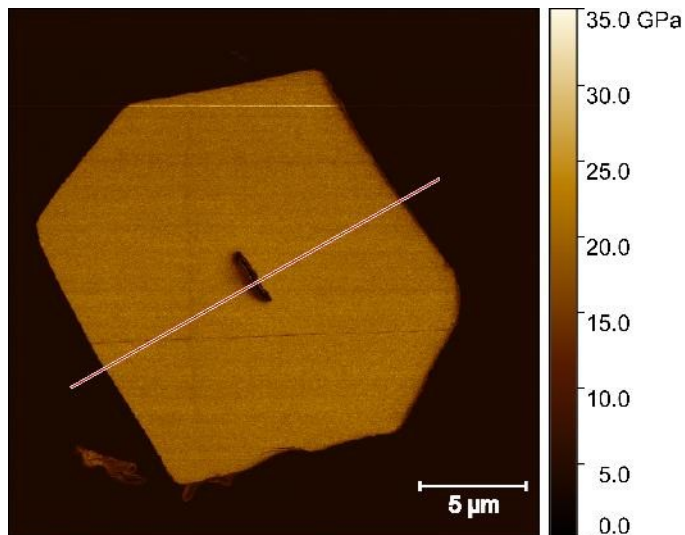


Flax fibres of high quality  
(Bolchoï variety, 2018)

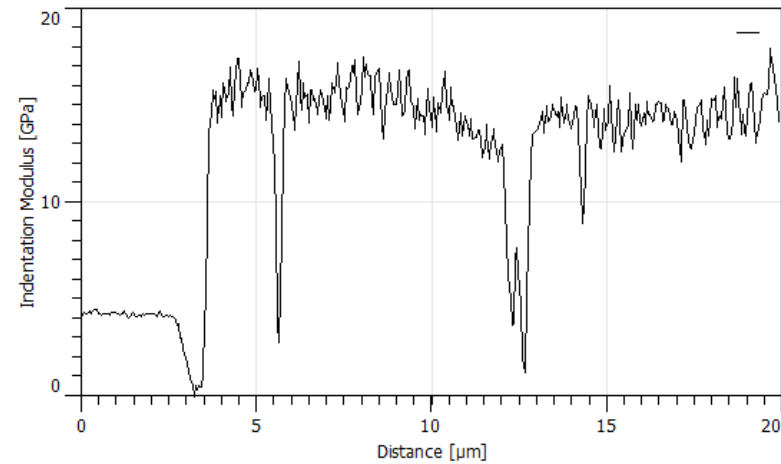
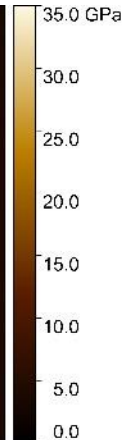
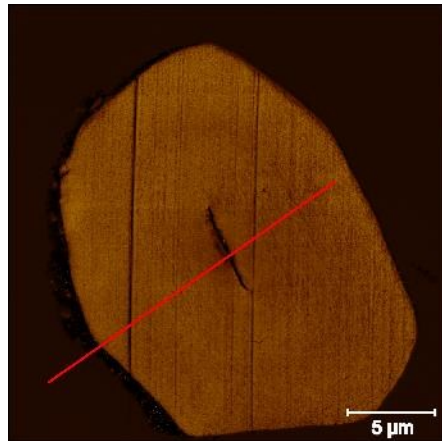


Modern fibres taken as **reference sample** to compare the mechanical properties of the ancient flax yarns

≈20 GPa

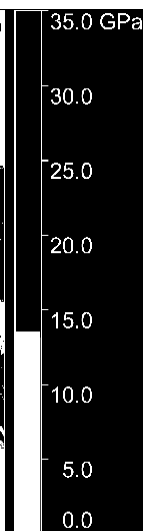
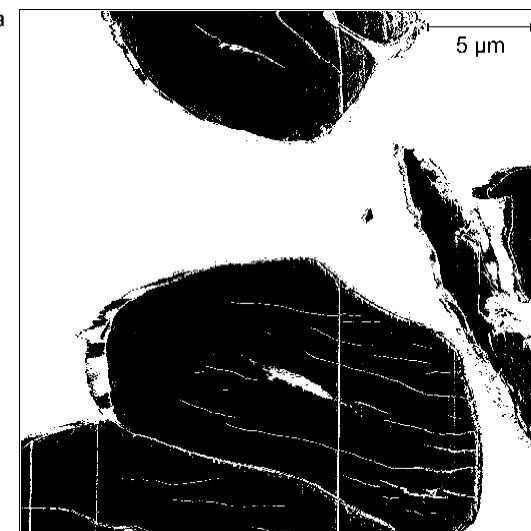
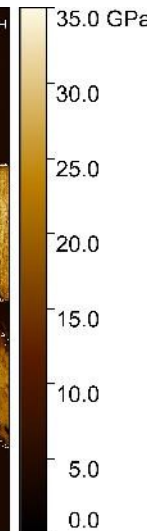


# SAMP 1 - « San Cristoforo »



Indentation modulus lower than modern flax fibres

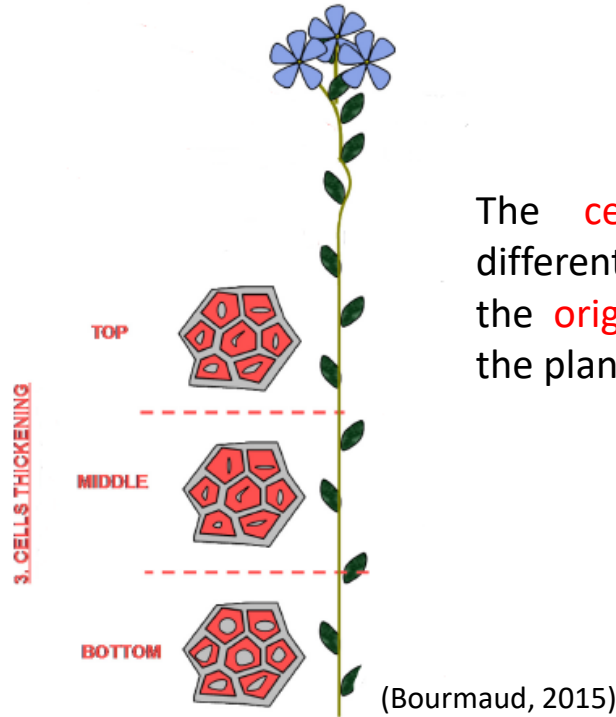
Fractures in the cell wall



# SAMP 1 - « San Cristoforo »

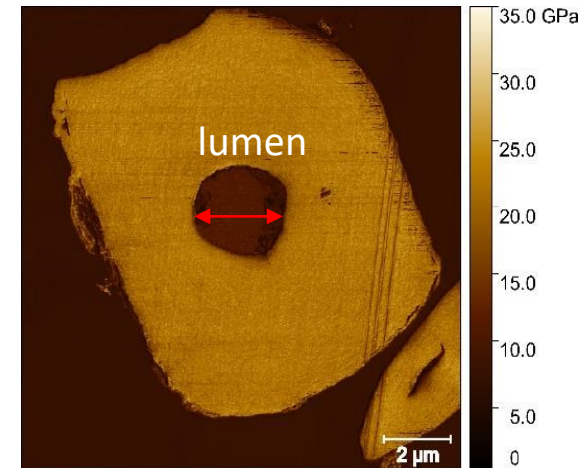


## CELLS DEVELOPMENT

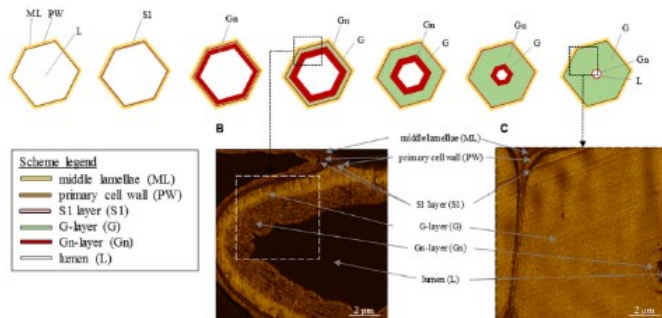


The cell wall is filled differently depending on the origin of the fibres in the plant

## SAMP 1



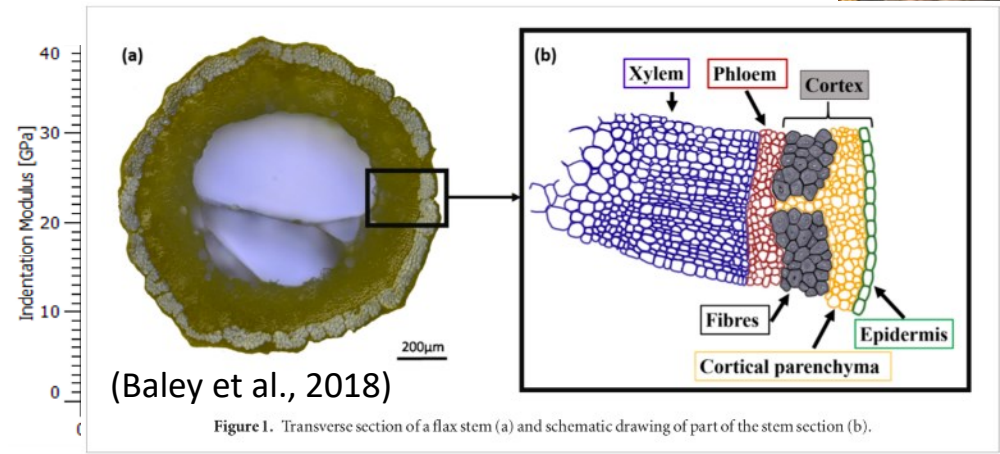
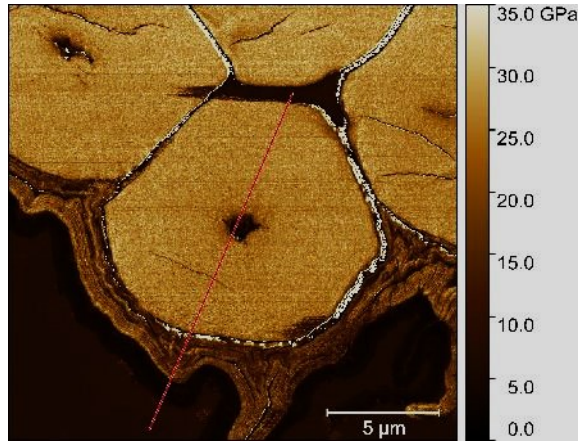
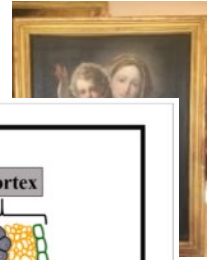
Lumen with high diameter



(Goudenhooff et al., 2019)

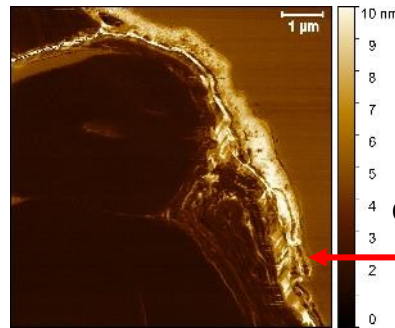
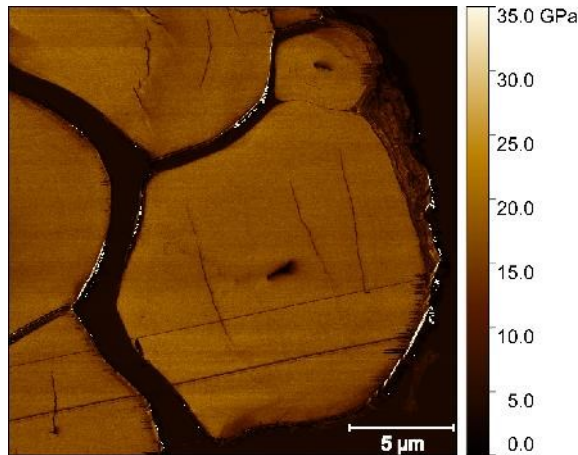
With the maturity of the fibre, the size of the lumen is reduced by the deposition of new layers

# SAMP 2 - « Madonna col bambino »



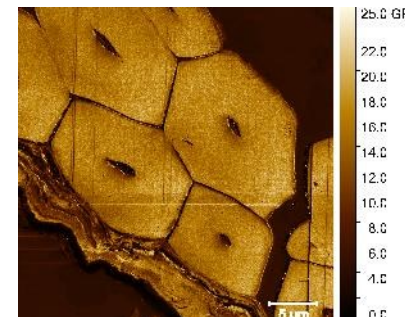
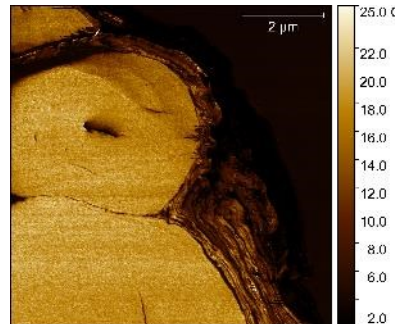
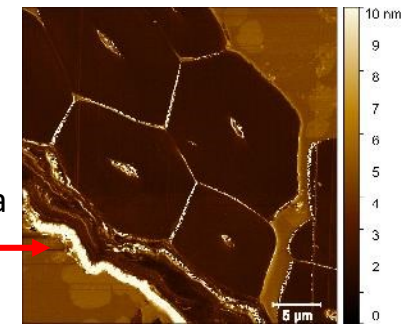
SAMP 2

Recent flax yarn (2019)

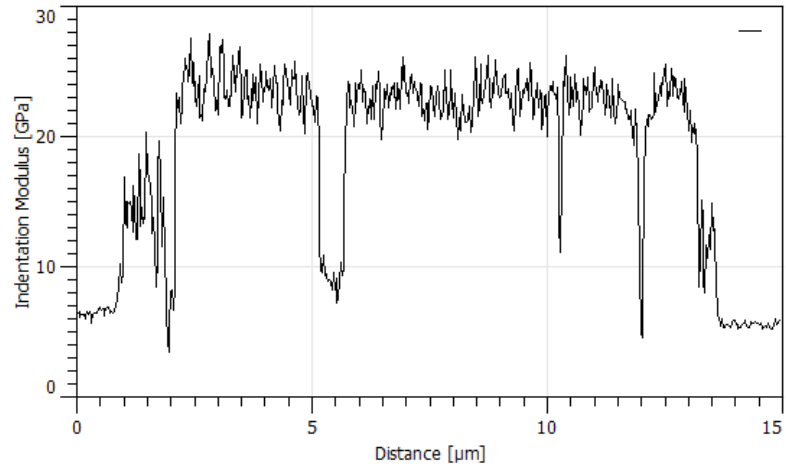
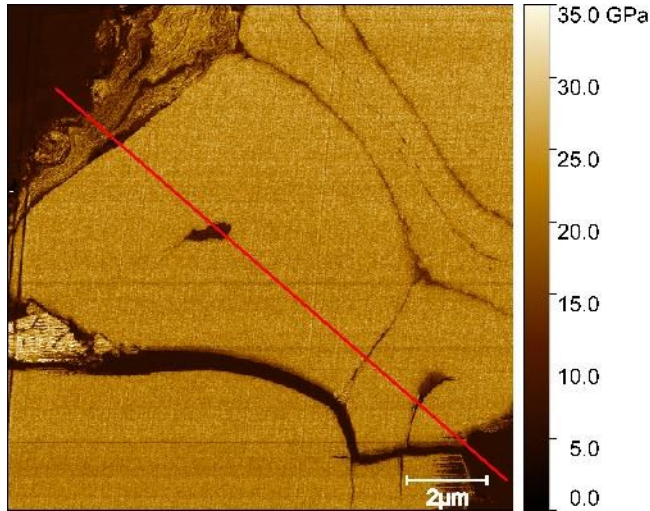


deformation

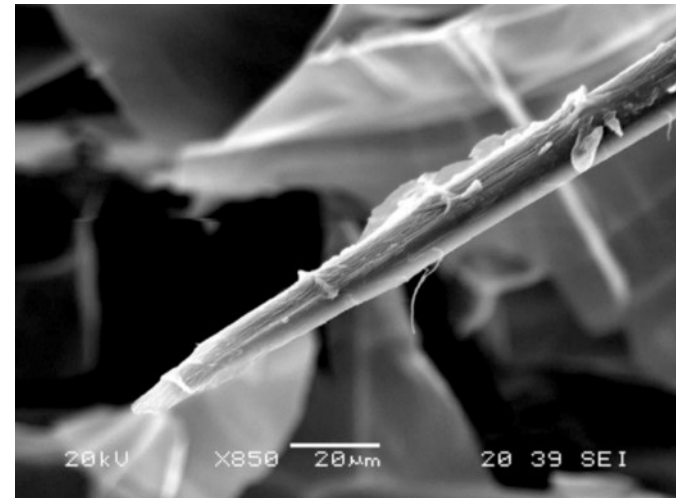
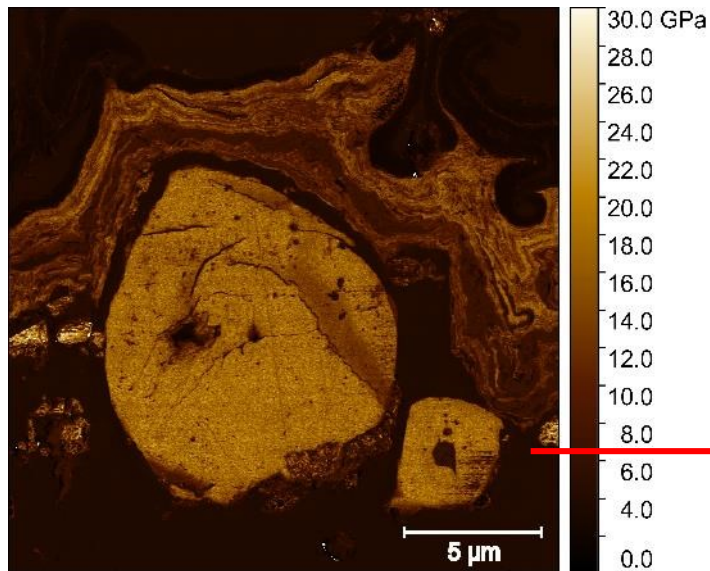
Rests of phloem/  
cortical parenchyma



# SAMP 4 - « Crocifissione »



Flax fibre extremity

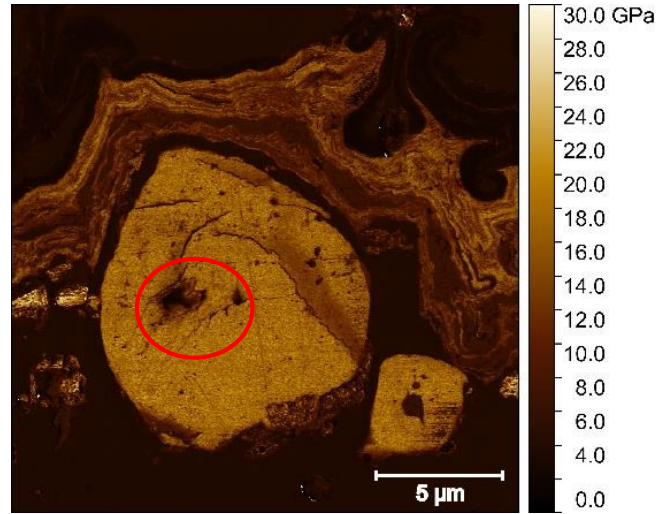


(Baley et al., 2018)

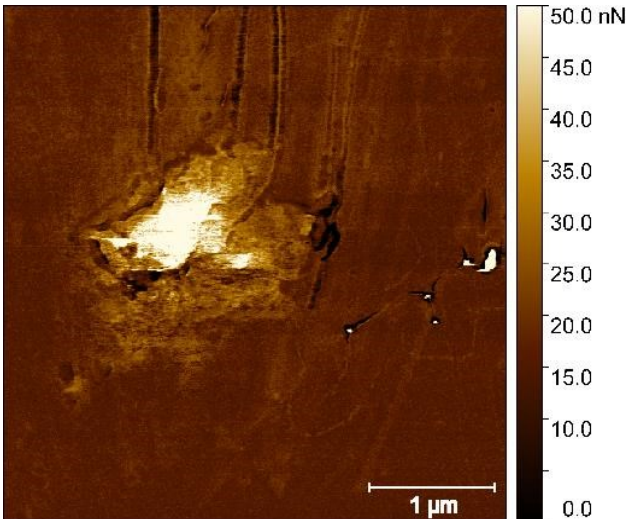
# HYPOTESIS: biological attack



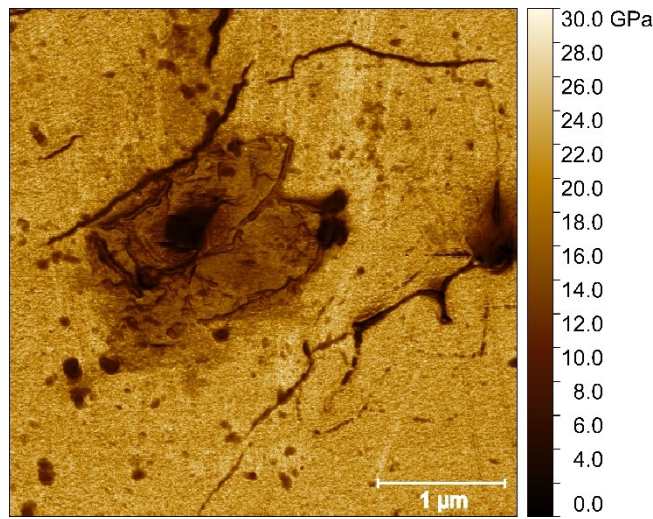
Fungal hyphae penetrates in the secondary cell wall and reach the lumen where a rapid proliferation occurs (Peacock, 2003)



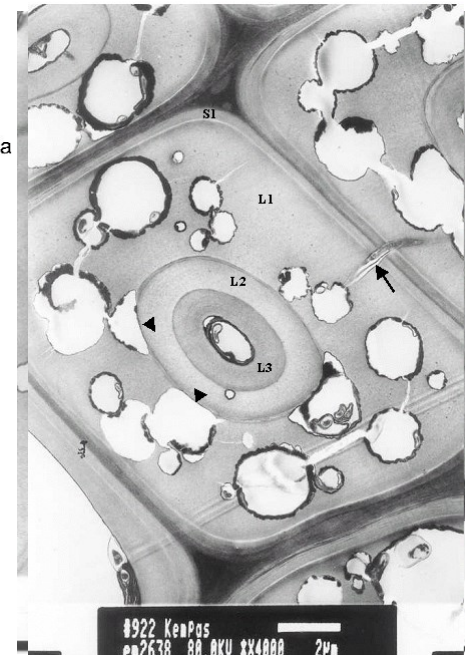
adhesion ↓ ↓ ↓ ↓



modulus



(Singh et al., 2004)



# SAMP 6 - « San Francesca Romana »



(Oriola et al.2014;  
Peacock, 2003)

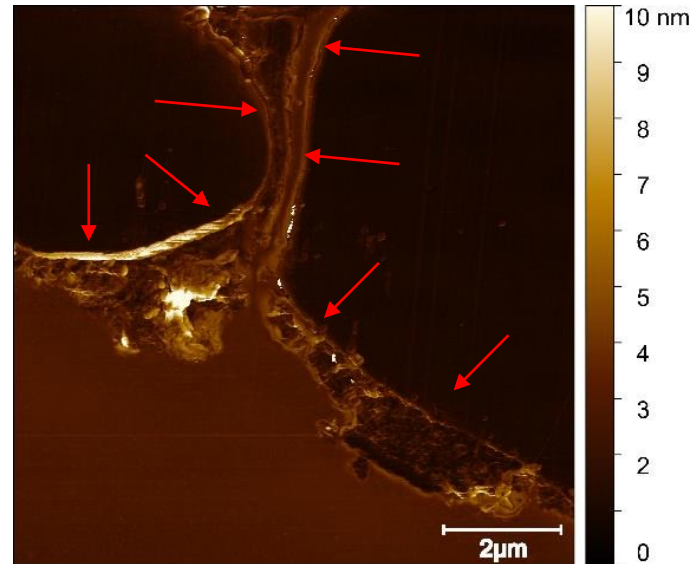
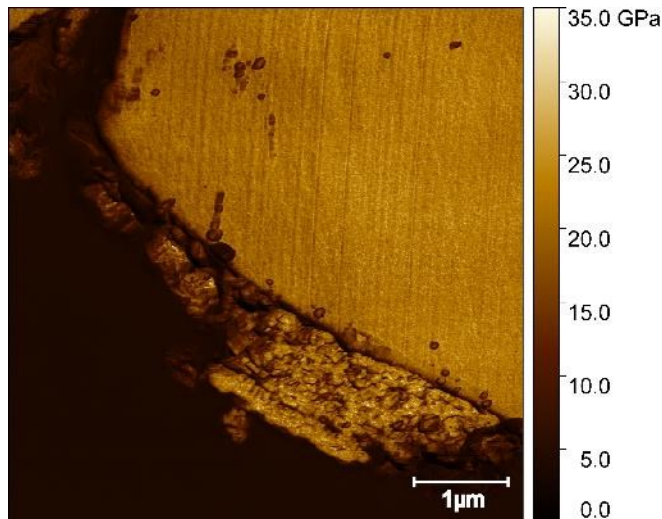
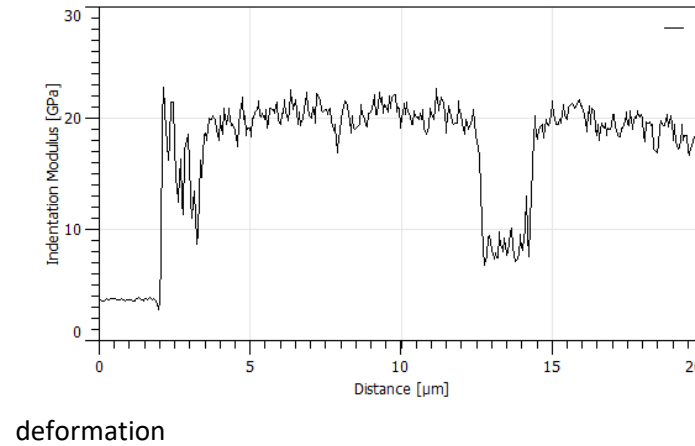
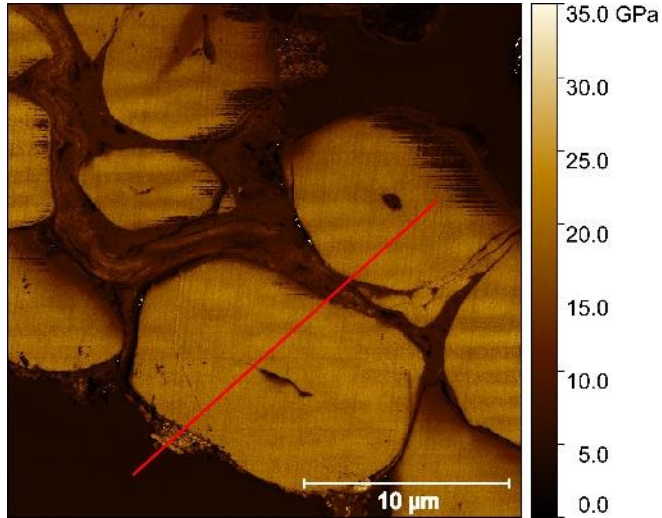
Acid conditions  
promote acid  
hydrolysis of  
cellulosic fibres



animal glue pH 5-7



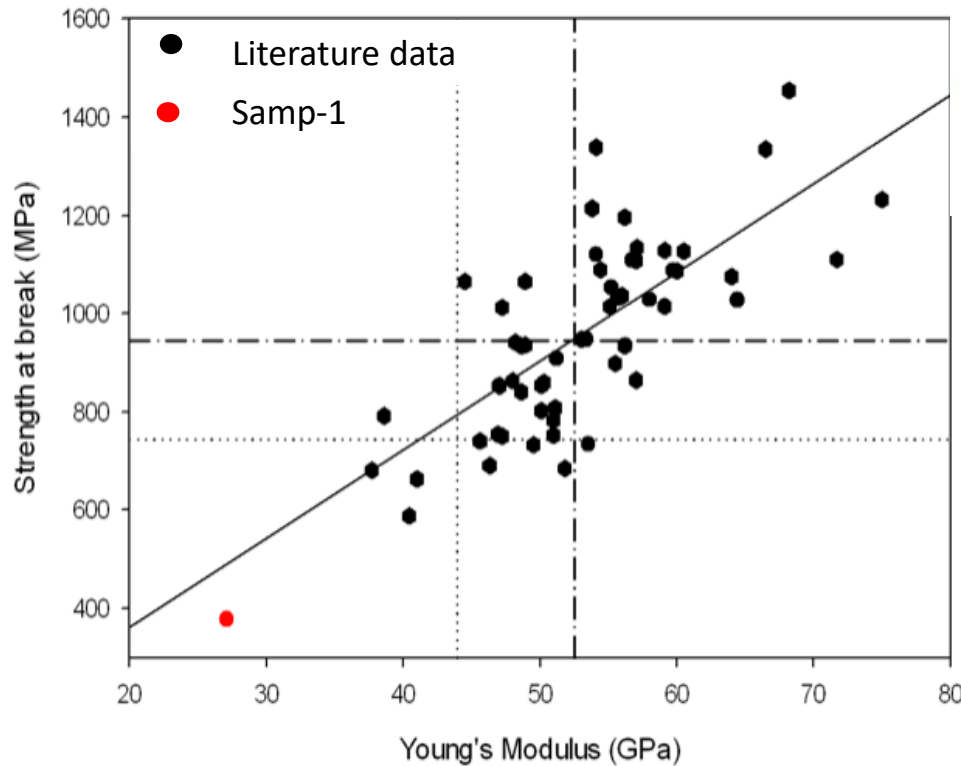
Study of the  
interface between  
the layers and the  
flax yarns



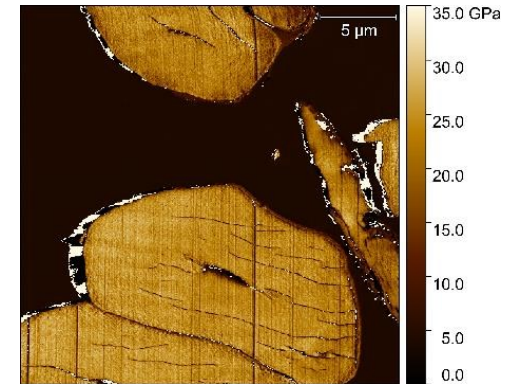
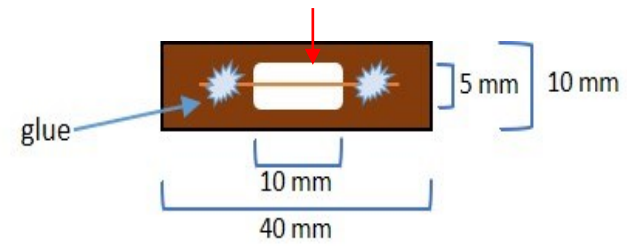


# TENSILE TEST ON ELEMENTARY FIBRES (SAMPLE 1)

(Baley & Bourmaud, 2014)

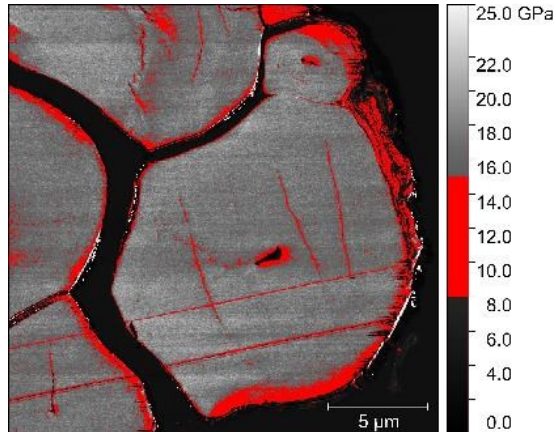


Elementary flax fibre on paper support



**Indentation modulus** of ancient samples is **comparable with the indentation modulus of recent flax fibres** at the cell level, but **Young's modulus and the strength at break are lower** at the elementary fibre level

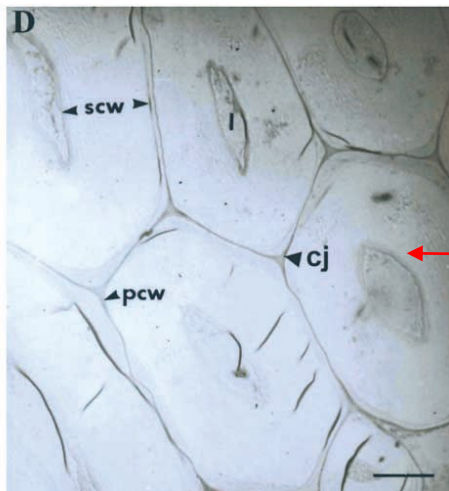
# FRACTURES



1) Flax fibres are sensible to the humidity (swelling)->  
**can humidity be the main cause of these fractures?**

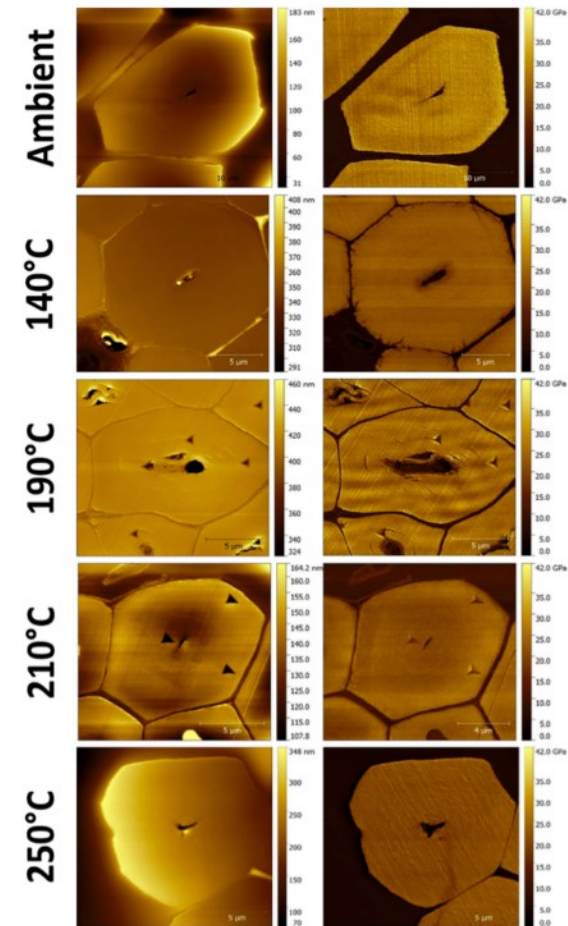
2) **Microbial deterioration** can induce cracks and transversal segmentations  
 (Peacock, 2003)

(His et al., 2001)



3) Fractures similar to the aged samples -> **fractures produced during the sample preparation?**

Flax fibres treated at different high temperatures do not show fractures



(Siniscalco et al., 2018)

## CONCLUSIONS

Ancient paintings are investigated with the AFM PF-QNM technique and degradation mechanisms are highlighted

Some ancient flax fibres show a high indentation modulus comparable to the recent fibres but at the fibre level their tensile mechanical properties are low.

**BUT:**

**Modern and ancient flax fibres are from different varieties, different maturity, growing conditions and retting/fibre extraction process**

## FUTURE ANALYSIS

Use the **recent flax yarn** and make several **humidity cycles** (10-100% RH) to **induce fractures**

**Link the results** with complementary analysis performed by the **University of Camerino** (Raman spectroscopy, x-ray diffraction, cross-sections...)

# THANK YOU FOR YOUR ATTENTION

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*A. R. T. & Co. Srl* - Spin-off company of the University of Camerino, Ascoli Piceno, Italy

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## References

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## Links

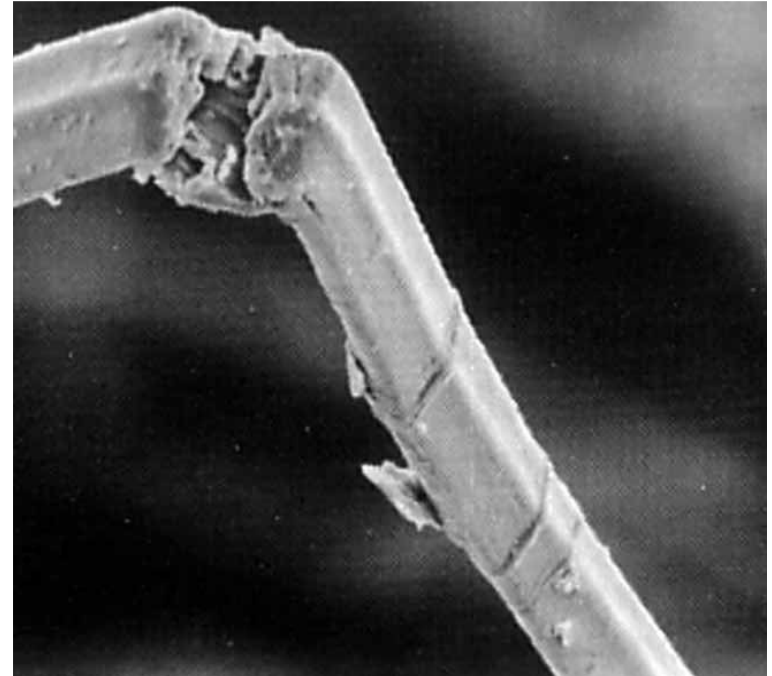
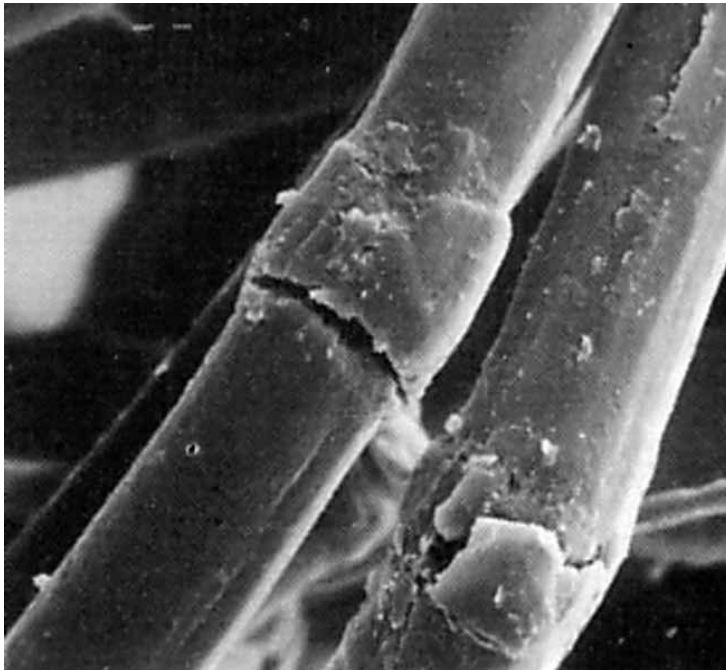
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<http://www.istitutoeuroarabo.it/DM/tommaso-maria-sciacca-fatto-pittor-dalla-natura/print/>

# Why indentation modulus and tensile test show different results?

Fractures noted around the kink-bands in ancient flax fibres



From: Martuscelli, E.; Paideia, 2006