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# A NEW CYCLIC-LOADS MACHINE FOR THE MEASUREMENT OF MICRO-MECHANICAL PROPERTIES OF SINGLE FLAX FIBERS COMING FROM THE TURIN SHROUD

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**SUMMARY.** As a bibliographic research has shown the absence of machines of the type requested, it has been necessary to design, build and test a new cycling-loads machine capable to measure the micro-mechanical characteristics of flax fibers like Young modulus, tensile strength and the loss factor. The flax fibers in question have diameters of 5-25  $\mu\text{m}$  and lengths of 1-3 mm.

The requirements for the testing machine are to furnish the stress-strain parameters, with an uncertainty not greater than 10%, relative to different loading-unloading cycles measuring fibers from 1 mm to 30 mm long, with 1% strain, a resolution of the order of 0.1  $\mu\text{m}$  and capable to measure forces up to 0.5 N with a resolution better than 2  $\mu\text{N}$ .

The design problems have been solved by employing a mechanical lever displaced by a micrometric screw to impose the displacements and by using an analytical balance, properly calibrated to measure the corresponding forces. The single flax fibers have been glued on particular polyester bases build up for the purpose.

The machine has been used to date fibers coming from the Turin Shroud. To reach this purpose, proper calibration curves have been determined using a series of ancient flax textiles. The Turin Shroud fibers resulted of 400 AD with an uncertainty of  $\pm 400$  years at 95% confidence level, thus compatible with the epoch in which Jesus Christ lived in Palestine.

## 1 INTRODUCTION

This work is addressed to the measurement of stress-strain plots of single flax fibers of relatively small sizes in order to measure the mechanical properties of fibers coming from the TS (Turin Shroud) and not as usual [1, 2] of elementary fibers obtained directly from the stem of the flax plant.

By measurements using a vision system connected to an optical stereomicroscope, it resulted that the flax fibers coming from TS and from other ancient fabrics, or pieces of them, have a diameter in the range from 5 to 25  $\mu\text{m}$  and gage lengths between 0.75 and 1.65 mm.

Mechanical analysis methods based on Breaking Strength of fibers [1–6] are widely applied and ASTM standards [7, 8] specifies test conditions in reference to samples directly extracted from a plant. The literature proposes various experimental set-ups and measuring methods [9–13] to test mechanical properties of single and / or technical fibers but none of them fit for the characteristics of the fibers in the field of textiles dating.

Natural vegetable textiles undergo a spontaneous and irreversible degradation as a function of time; in particular the polymerization degree of cellulose diminishes with time due to an increase

of the amorphous cellulose. This variation influences the mechanical properties of the vegetable fibers that can be in some way related to a historical period.

The fact that breaking strength of usual fibers is much higher than that relative to very ancient fibers and the consequent impossibility to use standard tensile machines to test these ancient fibers pushed the authors to design and build a new machine [14,15] capable to measure loading cycles characterized by relatively low tensions (corresponding to forces of few  $\mu\text{N}$ ). This task has been reached with the employment of an analytical balance instead of a standard load cell.

In addition to this, the present study is addressed to the evaluation of various loading cycles and to the measurement of various mechanical parameters after these cycles. Loading cycles have been applied to single fibers in literature [3, 9, 16-18], but they have been only used to evaluate traditional parameters with no reference to the loss factor; know-how based on space structures [19–21] has been used in the present case to consider this important mechanical parameter too. Also Ref. [22] confirms the absence of bibliographic references regarding the viscous behavior of fibers.

Some dependence on parameters such as humidity [5, 22], fiber diameter [23–26], length and clamping method [7, 10, 23] have been also considered: on the basis of data reported in the literature, proper conforming coefficients have been used in this paper.

The cellulose degradation with time can be additionally accelerated by many environmental factors [27] such as: temperature, light, moisture, soil acidity, or attacks from lichens, molds and mites and rotting. These factors could obviously affect the mechanical properties of the fibers, but, as it results from recent works [14, 15], these effects are below the uncertainty of very few centuries if a proper pre-selection of the fibers to be posed under test is performed.

In the literature, the evaluation of the probable age of a cellulose-based handwork is normally carried out by using the radiocarbon dating, a relatively expensive and destructive method, not always easily applicable. On the other hand, Museums have not always the necessity to know the date of samples with accuracy higher than a century to allocate them to a particular historical period. It is in this context that alternative dating methods, primarily non destructive and also cheaper, can be useful; a recent example [28] is the dating of single fibers coming from ancient flax textiles using vibrational spectroscopy and in particular Raman and FT-IR methods.

## 2 DESIGN OF THE MICRO-CYCLING MACHINE

After a bibliographic analysis, also accounting for the limited budget, a first solution (Figure 1) based on strain gages has been considered, which has three principal components: a displacement generator with a measurement gauge, a fiber tabbing system and a force measurement block. In addition to this solution many other have been considered [14].

The design problems came out when considering the specification for the fibers under test. In fact the force measurement system must have a resolution better of  $2 \mu\text{N}$  and a range of at least  $0.5 \text{ N}$  which implies a dynamic range of at least 250000 points, not easy to handle with strain gauges especially when forces are about of few micronewtons.

As even the most costly loading cells based on strain gauges resulted to have problems in reaching the necessary resolution, a solution based on analytical balances has been preferred. The final solution is reported in Figures 2 and 3, where the relatively small displacements posed in the requirements have been obtained using a proper mechanical lever capable to reduce the imposed displacement of a nominal factor from 5 up to 100.

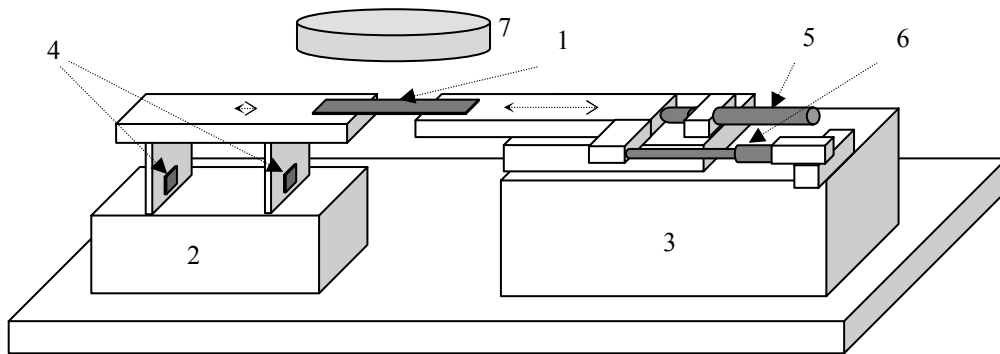


Figure 1. First design of the micro-cycling machine. The flax fiber tabbed on a proper sheet (1) connects the block (2) for force measurement to block (3) for displacement imposition and measurement. Block (2) consists on a base on which two flexible plates are clamped. On them some strain gauges measure the force applied to the upper plate. Block (3) consists on a base on which it is both mounted a micrometric screw (5), that moves the fiber under test, and a displacement gauge like a LVDT (6) to measure the corresponding displacements. A microscope connected with a camera (7) controls the fiber behavior.

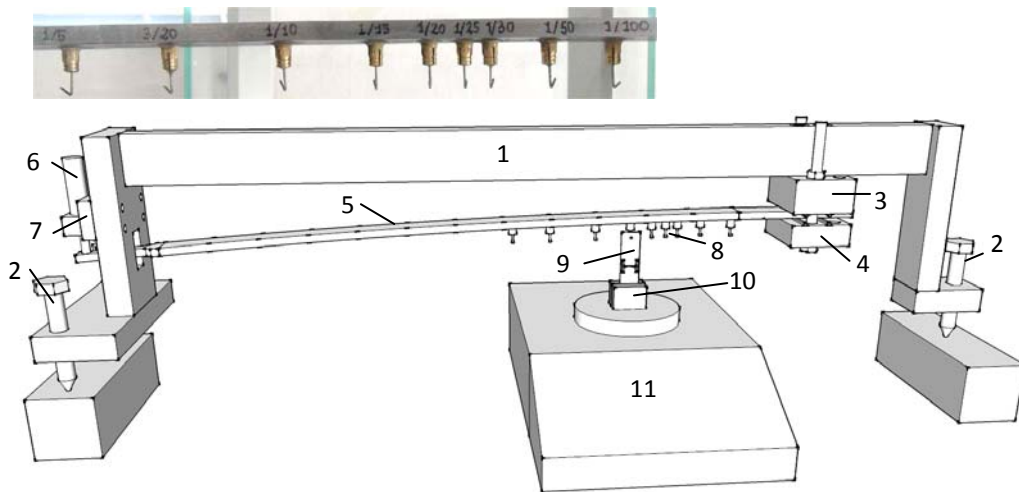


Figure 2. Sketch of the micro-cycling machine built. 1 frame is horizontally regulated by screws 2; 3,4 clamping blocks for the displacement-reducer cantilever beam 5; 6 micrometric screw gauge (mounted on coupling plate 7), that moves beam 5. To one of the hooks 8 (on the top left a detail of the hooks having reduction factors from 1/5 to 1/100) is hanged a proper sheet 9 tabbing the fiber under test. The lower part of 9 is glued with two iron blocks 10 that lay on the plate of the analytical balance 11.

The micro-cycling machine has been built in agreement with the design criteria reported here

below and satisfying the condition for the measured data to have a design uncertainty better than 10%:

- a. to measure cycles in correspondence of stresses ranging from zero to  $2/3$  of  $\sigma_R$  (breaking strength) with resolution better than about 100 measurement points per cycle;
- b. to measure the elongation of single fibers up to 1% with a resolution of  $0.1 \mu\text{m}$ , having serviceable lengths from 1 mm to 30 mm;
- c. to measure forces  $F$  from 0 to 0.5 N, with a resolution of  $2 \mu\text{N}$ .

The fiber seen through a stereo-microscope has been clamped on a special polyester mask using cyanoacrylate glue. The mask is then hanged on the hook corresponding to a nominal reduction factor of  $1/20$  in the micro-cycling machine and it touches the plate of the analytical balance by means of two iron blocks of predefined mass (see Figure 3).



Figure 3. On the left, photo of the new micro-cycling machine. On the middle, the special polyester mask in which the flax fiber is clamped (evidenced by the arrow). On the right the tabbed fiber clamped on two iron masses and leaned on the plate of the analytical balance after having broken the two lateral strips using an iron wire as heating source.

The problem relative to the clamp of the flax fiber is not simple because a proper support should be used. As it is not simple to build such clamps of micrometer size, a simpler solution has been preferred in the present study: the fiber has been simply glued (cyanoacrylate) on a proper support designed for the purpose. The gage length and the diameter of the tabbed fiber have therefore been successively measured using a vision system connected to a stereo-microscope.

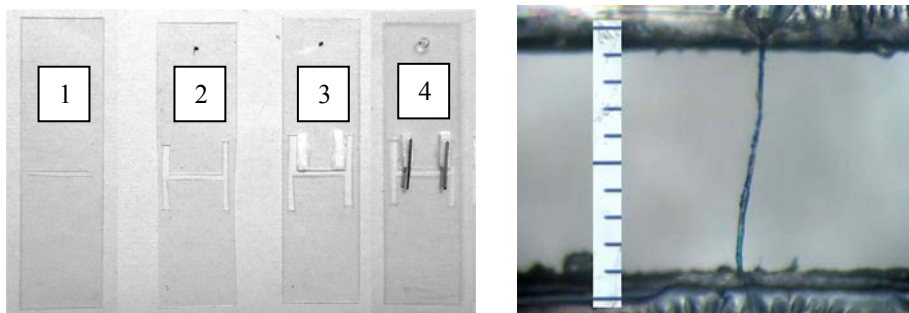


Figure 4. On the left four phases of the polyester strip construction for flax fiber tabbing. On the right a detail of a flax fiber glued on the strip (sample A.07, see Ref [15], gage length 0.85 mm, diameter  $14.1 \mu\text{m}$ ); the non-straight position of the fiber with some possibility to elongate is voluntary to avoid premature ruptures during handling.

The polyester sheet (Figure 4) has been built with a proper slit, one millimeter in sizes, capable to support the two ends of the flax fiber under test. The two strips at the edges have been built to stiffen the whole system thus preventing premature fiber ruptures during handling. When mounted on the machine, these two strips are broken using an heating sources (a white-hot iron wire) thus letting the flax fiber free to move in the vertical direction. The four iron bars glued on the strip are put to avoid possible rotations of the upper part of the strip. To avoid friction between strip and bars a small piece of paper has been put in the two.

The flax fiber is glued not in a straight position, but with some possibility to elongate in order to avoid premature ruptures during handling. The initial gap is well evident in the resulting stress-strain plots and therefore it can be easily eliminated.

### 3 MACHINE TESTING

#### 3.1 Calibration

Due to the fact that the designed clamp is obviously not perfect, the flexible cantilever displacement-reducer beam, designed on the base of a clamp model, has been calibrated to find the reduction factors of the 9 hooks. The results are reported in Table 1 where the calibration parameters of the micro-cycling machine for a nominal reduction factor  $\rho$  of 1/20 (always used in the present analysis and for which it turned out to correspond to a measured reduction of 1/18.9) are summarized.

*Table 1. On the left the measured reduction factor  $\rho$  of the hooks mounted on the displacement-reduction beam. On the right the calibration parameters of the micro-cycling machine related to the nominal factor of 1/20 (measured of 1/18.9) that has been used to perform the tensile tests.*

Nominal $\rho$		Measured reduction factors $\rho$	
1/5	(0,200)	0,197 $\pm$ 1,3 %	(1/5,1)
3/20	(0,150)	0,152 $\pm$ 1,0 %	(1/6,6)
1/10	(0,100)	0,102 $\pm$ 1,2 %	(1/9,7)
1/15	(0,067)	0,068 $\pm$ 1,6 %	(1/14,6)
<b>1/20</b>	<b>(0,050)</b>	<b>0,0528 <math>\pm</math> 1,7 %</b>	<b>(1/18,9)</b>
1/25	(0,040)	0,0427 $\pm$ 1,9 %	(1/23,4)
1/30	(0,033)	0,0367 $\pm$ 2,3 %	(1/27,2)
1/50	(0,020)	0,0235 $\pm$ 3,7 %	(1/42,6)
1/10	(0,010)	0,013 $\pm$ 8,5 %	(1/79,1)

Force	Displacement	
Standard uncertainty	Range	Standard uncertainty
$\pm 0.5$ mg	0 – 50 $\mu\text{m}$	$\pm 2.5$ $\mu\text{m}$
	50 – 300 $\mu\text{m}$	$\pm 3.0$ $\mu\text{m}$
	300 – 400 $\mu\text{m}$	$\pm 3.5$ $\mu\text{m}$
0 – 0.50 N	400 – 500 $\mu\text{m}$	$\pm 4.3$ $\mu\text{m}$
	500 – 600 $\mu\text{m}$	$\pm 5.0$ $\mu\text{m}$
	600 – 700 $\mu\text{m}$	$\pm 5.5$ $\mu\text{m}$
	700 – 792 $\mu\text{m}$	$\pm 6.5$ $\mu\text{m}$

The analytical balance is used in a manner not covered by the constructor. It is well known that the compensation circuit of an analytical balance can be instable if an additional spring (the fiber under test) is added, but this problem can be avoided either using a proper calibration based on the acquisition data after a predetermined time interval (solution adopted for these test) or using higher-cost analytical balances capable to properly filter the instability produced by the additional spring. Thus a calibration has been done with the purpose to verify that the readings provided by the analytical balance are within the standard design uncertainty [29] and that its response is linear also under various elastic properties (in fact the fibers to be tested have different geometry and

stiffness).

The stiffness of the movable load support which carries the load pan of the analytical balance turned out to be sufficiently high to produce a negligible loading effect; in fact its stiffness is  $1.0 \pm 0.2$  MN/m that is about 100 times higher than the mean stiffness shown by the modern flax fiber tested (it is of the order of 10 kN/m). The corresponding loading error resulted at the most of 1% that is sufficiently smaller than the design uncertainty.

Using four strain gauges glued near the support of the flexible beam, the output of the analytical balance, under different stiffness conditions has been checked. The experimental results obtained confirm that the response of the analytical balance is linear and the values given are acceptable if they are read within a limited time (3 seconds), as it has been done, so the force uncertainty is not greater than  $\pm 5$   $\mu$ N even if in presence of drift.

### 3.2 Fiber normalization

A flax fiber can be represented by an irregular hexagonal cross-section cylinder with a central hollow duct (lumen). see Ref. [1, 9, 22], but in the present work a flax fiber can be more simply modeled as follows: a cylinder without voids, of circular cross section, geometrically uniform along its length, homogeneous and isotropic subjected to a uniform tension during test that is parallel to its longitudinal axis. Its Young Modulus can vary from 1 GPa to 100 GPa. This simplified model can produce a standard uncertainty [29] lower than 5% and this uncertainty can be neglected with respect to the design uncertainty of 10% of the test machine.

It has been observed that the mechanical parameters of the single flax fibers also depend on diameter, length and environmental humidity of the laboratory. It has been called by the authors "normalization" the affine transformation regarding the measured stresses  $\sigma$  of flax fibers (while the strain has not been changed). Thanks to this, the dispersion of the data reported in the resulting plots has therefore been reduced of also about 20%. For this reason the measured stress  $\sigma$  of flax fibers, having various sizes, have been normalized with respect to the standard stress  $\sigma_s$  relative to a standard fiber [14] characterized by:

- length L between the two clamps: 1.0 mm;
- diameter d: 15  $\mu$ m;
- relative humidity H: 50%,

considering the influencing factors taken from Ref. [22]. The standard stress  $\sigma_s$  applied to each fiber has been evaluated as:

$$\sigma_s = K_L K_d K_H \sigma \quad (1)$$

where  $K_L$ ,  $K_d$  and  $K_H$  are respectively the length, diameter and humidity coefficients defined as follows:

$$K_L = \frac{1479}{1500 - 20.93L} \quad (2)$$

$$K_d = \frac{67.62}{89.41 - 1.452d} \quad (3)$$

$$K_H = \frac{743.0}{580.4 - 3.250H} \quad (4)$$

where L [ $\mu$ m], d [ $\mu$ m], are respectively the measured fiber length and diameter, and H [%] is the

laboratory humidity.

The equation of the length coefficient  $K_L$  is obtained interpolating the data reported in the plot of Ref. [22, p. 19], assuming a linear behavior in the length range 0,5 – 15 mm with the same slope of the line that fits the first two points of the technical fibers' series and with constant term fixed at 1.5 GPa, slightly below that of the “standard decorticated elementary fibres”, because the fibers tested are extracted from ancient fabrics, not directly from the stem.

The equation of the diameter coefficient  $K_d$  (range 4 – 40  $\mu\text{m}$ ) is that of the “fitted line” interpolating the data reported in the plot of Ref. [22, p. III]. The equation of the humidity coefficient  $K_H$  is obtained from the plot of Ref. [22, p. 28], assuming the slope corresponding to the line of 3.5-mm-length in range 30-65%. The gage lengths of the samples studied are actually less than 3.5 mm (they are between 0.75 and 1.65 mm), but no diagrams for such sort length are available in the literature.

### 3.3 Parameters under test

From the stress-strain plots, the following mechanical parameters have been evaluated for all the linen fabrics, see Ref. [15]:

- Breaking Strength  $\sigma_r$ .
- Young Modulus  $E_f$  relative to the last part of the increasing loading cycles. It is the classical modulus defined by ASTM as “Initial Modulus”, Ref. [7, §12.5]. The stress-strain curve of flax fibers is not linear because to both its plasticity and its “Packing Effects”, see Refs. [19, 20], caused by the micro-fibrils rotation of the “Secondary Cell Wall – S2”, see Ref. [9], with loading increasing, that progressively stiffen the fiber. The final part of the stress-strain plot seems therefore more representative because in this area the mentioned effects are reduced.
- Young Modulus  $E_i$  relative to the first part of the decreasing loading cycles. Being the curve not linear as described above, this initial part looks representative because the fiber seems here more “packed” and less subjected to creep.
- Loss Factor  $\eta_d$  relative to the last complete loading cycle representing the dissipated energy. It is defined as:

$$\eta = \frac{D}{2\pi U} \quad (5)$$

where D and U are respectively the dissipated and stored energies during a whole loading cycle.

- Loss Factor  $\eta_i$  relative to an inverse loading cycle. This is evaluated in reference to the last unloading phase of the cycle coupled with the last loading cycle that produced the fiber breaking. This parameter is more significant than the previous one if the loading cycles are not too much as in the case of premature breaking of the fiber under examination.

## 4 RESULTS

Once tested the micro-cycling machine, a mechanical property of flax fibers and their corresponding ages has been correlated for the determination of a two-way relationship. This goal was reached performing stress-strain measurements in 350 loading-unloading cycles of 85 different flax fibers belonging to samples of 12 different historical and modern flax fabrics, see Ref. [14], dating back to up to more than five thousand years ago, obtaining approximately 16,000 manual acquisitions.



These measurements have been made within the more general procedure described here below, necessary to obtain the calibration curves to be used for further dating.

1. To search for a sufficient number of samples of ancient flax fabric from various periods, but characterized by a known origin.
2. To search for a mechanical properties of the flax fabric sensitive to aging. For example the fiber's strain at break was a measured parameter not sensitive to ageing.
3. Determination, if any, of a mathematical relationship between the measured mechanical property and the date of the sample under test.
4. Determination and quantification of the systematic effects, generally of environmental type, capable to influence the relationship established in the previous step.
5. Determination of a bi-univocal mathematical relationship corrected by systematic effects.
6. Selection of the sample of unknown date under test and preliminary analysis using a series of parallel techniques able to highlight the suitability of the sample to be subjected to dating (otherwise, the sample must be discarded).
7. Measurement of the unknown date of the flax sample using the relationships just defined, with obvious assignment of the corresponding measurement uncertainty [29].

Once determined the following mechanical parameters sensitive to the historical age of the sample, Breaking Strength  $\sigma_r$ , Young Modulus E and Loss Factor  $\eta$ , the corresponding two-way relationships have been determined. Due to both the non-linearity of the loading-unloading cycles and to the non negligible creep/stick-slip effects present in the first loading cycles, some parameters have been evaluated as described in Section 3.3.

#### 4.1 Mechanical Multi-Parametric Dating Method (MMPDM)

For each of the five parameters in question, the following calibration curves have been determined (see Ref [15]) with the corresponding Pearson's correlation coefficient R:

$$\sigma_r = 139.14 e^{0.0009678x} \pm 300 \text{ years}; \quad R = 0.94 \quad (6)$$

$$E_f = 6.2219 e^{0.00070938x} \pm 400 \text{ years}; \quad R = 0.91 \quad (7)$$

$$E_i = 8.1758 e^{0.00060588x} \pm 500 \text{ years}; \quad R = 0.91 \quad (8)$$

$$\eta_d = 7.5376 - 0.0013721x \pm 200 \text{ years}; \quad R = 0.95 \quad (9)$$

$$\eta_i = 4.2604 - 0.0011458x \pm 400 \text{ years}; \quad R = 0.90 \quad (10)$$

The dates, resulting from the five parameters in question, have been combined together by means of the so called Mechanical Multi-Parametric Dating Method (MMPDM) to obtain the historic data of the sample under test. Basing on the experience gained during testing, the authors propose the following weighted mean of the five results in which the historic data  $y_m$  of the sample in question derives from the significance level of the single parameter considered (see Ref. [15]):

$$y_m = \frac{2y_{\sigma_r} + y_{E_f} + 3y_{E_i} + y_{\eta_d} + 3y_{\eta_i}}{10} \quad (11)$$

The resulting uncertainty of the historical dates deriving from the measured mechanical parameters has a standard uncertainty [29] of about 200 years.

It must be observed that another parameter related to the reliability of the mechanical dating is the compatibility of the resulting dates obtained from Eqs. 6-10 because they are relative to properties of the cellulose that are subjected to opposite bias if the linen fibers are subjected to environmental factors capable to change the mechanical behavior.

If proper shrewdness is taken into account by means of a preliminary selection of the samples also based on microscopic visual inspection, the bias due to environmental effects can be reduced and therefore a rough mechanical dating is possible. The data dispersion reported in the correlation coefficients of Eqs. 6-10 seems greatly due to environmental factors that obviously increase the variability of the mechanical behavior of the linen fibers, but this dispersion is not so high to prevent a rough dating. Future detailed analysis of the influence of the environmental factors could then reduce the detected dispersion hopefully making more competitive this method.

Exposition to humidity seems an environmental factor not very influent in the analysis if a proper textile selection is done, as previously discussed. This because it seems that humidity highly damages the chemical structure, the color and the mechanical behavior of these fibers that therefore are quite simple to recognize and discharge during a preliminary selection.

Exposition of flax textiles to relatively high temperature (200 °C for some hundreds of seconds) can alter the mechanical properties of the fibers thus producing bias of some hundreds years coupled with a more brownish coloration. Therefore a darker color of the textile can be the sign of an exposition to heat sources. If instead the color of the textile is not appreciably different from the original one, it can be supposed that the bias produced by heat source can be less than one century.

#### 4.2 An application to the Turin Shroud

The MMPDM has been applied to flax fibers sampled from the back of the TS in correspondence of the buttock area during the 1978 campaign of tests (vacuumed dusts, Filter h [30]). Eight different TS fibers have been recognized as such by the first author among others using a petrographic microscope and a particular technique based on cross-polarization. TS fibers in fact present a greater number of transversal defects (kink bands [10]) than other more recent flax fibers and it is therefore relatively easy to detect them.

The mean mechanical parameters detected for the eight TS fibers and the corresponding ages using Eqs. 6-10 are reported in Table 2.

Rounding the result to the century with an uncertainty evaluated at 95% confidence level, according with the MMPDM, the date of the TS is 400 AD  $\pm$ 400 years.

*Table 2: mean values of the mechanical parameters of the eight TS fibers tested with the new micro-cycling machine and corresponding ages with the corresponding standard uncertainties.*

Mech. Par.	$\sigma_r$ [MPa]	$E_f$ [GPa]	$E_i$ [GPa]	$\eta_d$ %	$\eta_i$ %	MMPDM
Mean Value	243.22	6.28	10.78	8.2	3.6	-
Date BC	577±336	14±418	456±538	-510±194	564±384	372±214

## 5 CONCLUSION

A new cycling-loads machine has been designed, built and calibrated for the measurement of micro-mechanical properties of single flax fibers of relatively small sizes. In fact the length of 1-3 mm and the lower breaking strength of ancient flax fibers prevented the use of commercial machines of this kind.

This work has been performed with the aim to define an alternative mechanical method for textiles dating and the MMPDM defined after the study of various parameters linked to stress-strain measurement during various loading cycles allowed to assign a date to the sample in question with a standard uncertainty of two centuries. The parameters sensitive to ageing are: breaking strength, two Young Modules and two Loss Factors.

The bias due to environmental factors like humidity and temperature can highly alter the resulting date of the flax sample in question of also of many centuries. It has been therefore reduced using a pre-selection of the fibers under analysis. The standard uncertainty of two centuries reached at this initial stage could be reduced in the next future if a greater number of historical samples will be analyzed and proper calibration curves will be defined as a function of the climatic conditions in which the sample was conserved.

The MMPDM has been applied to flax fibers coming from the back side of the TS (Turin Shroud), picked up during the 1978 campaign in correspondence of the buttocks area [30]. These fibers furnish a rounded mechanical date of the TS of 400 AD  $\pm$ 400 years at 95% confidence level that is compatible with the epoch in which Jesus Christ lived in Palestine. This result is also compatible with parallel dating made on the TS using Raman and FT-IR analysis [28], but it is not compatible with 1988 radiocarbon result [31] that determined a Medieval period. This incongruence, that, in agreement with various studies, seems due to a systematic effect caused by environmental contamination, is the aim of further studies also connected with the not yet explainable body image formation, because this last could have produced an increment of C14 isotopes in the TS flax.

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## **ABSTRACT / summary**

# **A NEW CYCLIC-LOADS MACHINE FOR THE MEASUREMENT OF MICRO-MECHANICAL PROPERTIES OF SINGLE FLAX FIBERS COMING FROM THE TURIN SHROUD**

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The aim of the present study is to measure the micro-mechanical characteristics of flax fibers coming from the TS (Turin Shroud) [1]. In particular various parameters like Young modulus, tensile strength and the loss factor [2] have been considered from the analysis of various cyclic loads. As a bibliographic research showed the absence of machines able to fulfill the requirements necessary to perform the tests, it has been necessary to design, build and test a new machine. The flax fibers in question have a diameter in the range from 5  $\mu\text{m}$  to 20  $\mu\text{m}$  and lengths also lower than 3 mm.

The necessary requirements for the testing machine are the following: to built a practically zero cost machine in the laboratory of the Department of Industrial Engineering capable to furnish the stress-strain parameters relative to different loading-unloading cycles; to measure fibers from 1 mm to 30 mm long, and 1% stain with a resolution of the order of 0.1  $\mu\text{m}$ ; to measure forces up to 0.5 N with a resolution better than 2 N. For the aim of the study, the design uncertainty has been defined to be not greater than 10%.

The design problems have been solved by employing a mechanical lever displaced by a micrometric screw to impose the displacements and by using an analytical balance, properly calibrated the measure the corresponding forces. The manual reading, that can be automated in the future, furnishes the stress-strain values as a function of time that allows to build the loading cycles up to the breaking strength of the fiber under test. Using a stereo-microscope, the single flax fibers have been glued on particular polyester bases build up for the purpose.

Various parameters relative to the TS have been considered for the mechanical characterization but the following five have been revealed more interesting: a) the breaking strength; b) the mean Young modulus; c) the final Young modulus; d) the loss factor of direct cycles; d) the loss factor of inverse cycles. The results relative to the TS fibers are reported in plots showing the relative uncertainty and are compared with other results relative to recent fibers. The lower breaking strength (243 MPa instead of 1000 MPa of recent fibers) the lower Young modulus, both mean and final (6.3 and 10.8 GPa respectively instead of 25 and 32 GPa of recent fibers) and the higher loss factor, both direct and inverse (8% and

4% respectively instead of 5% and 2% of recent fibers) lead to think that the TS fibers are very old, of 400 BC  $\pm$ 400 years [3].

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