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Comparative Research of the Dynamic Young's Module of Fibres

Abstract
This work presents an investigation of the dynamic Young's modulus for fibres of varied thickness performed on an Impulse Mechanical Spectrometer (IMS) and Dynamic Mechanical Analyser (DMA). The IMS was designed and built by the authors and the DMA instrument was produced by TA Instrument Co (USA). Because different methods are applied for determination of the dynamic Young's modulus it was necessary to perform a comparison analysis of the results received from the IMS and DMA, which are currently the most frequently used testers. This analysis shows that an IMS can be used when a DMA cannot be applied. The parameter set comprising the dynamic Young's modulus for longitudinal vibrations can find application in material engineering in industry. The DMA method does not require the application of mathematical formulas to determine Young's modulus but requires calibration as apposed to IMS. Measurements of the elasticity modulus were carried out under automatically stabilised climatic conditions. The compliance of the results obtained allows us to evaluate positively the performance of the Impulse Mechanical Spectrometer. Comparison of the results makes it possible to determine the measuring possibilities of the DMA and IMS and show the advantages of IMS over DMA.

Key words: *fibres, mechanical spectroscopy, dynamic modulus.*

■ Introduction

One of the basic parameters characterising the mechanical properties of fibres is the static [1, 2] and dynamic modulus of elasticity [3, 4].

The Impulse Mechanical Spectrometer (IMS) and Dynamic Mechanical Analyser (DMA) characterise materials by applying a sinusoidal stress and measuring the resultant sinusoidal strain. If the material is purely elastic, the phase difference between the stress and strain sine waves is zero degrees [5].

If the material is purely viscous, the phase difference is 90 degrees [5]. The most real materials are viscoelastic and, therefore, exhibit a phase difference between those extremes. This phase difference, together with the amplitudes of the stress and strain waves, is used to determine a variety of fundamental material parameters including the storage and loss modulus.

The techniques of measuring the dynamic Young's modulus began to be applied to resonance methods with displacements, to the propagation of waves, to an ultrasonic instrument and the method of using DMA [6 - 8]. However, these methods are applicable mainly to metal and ceramic products, and, to a limited extent, for textile filaments mainly in the form of bunch. The limits of using DMA for fibres are connected with the length and thickness of fibres. Due to the existence of reasons [9 - 13] as to the possibility of defining the dynamic module of elas-
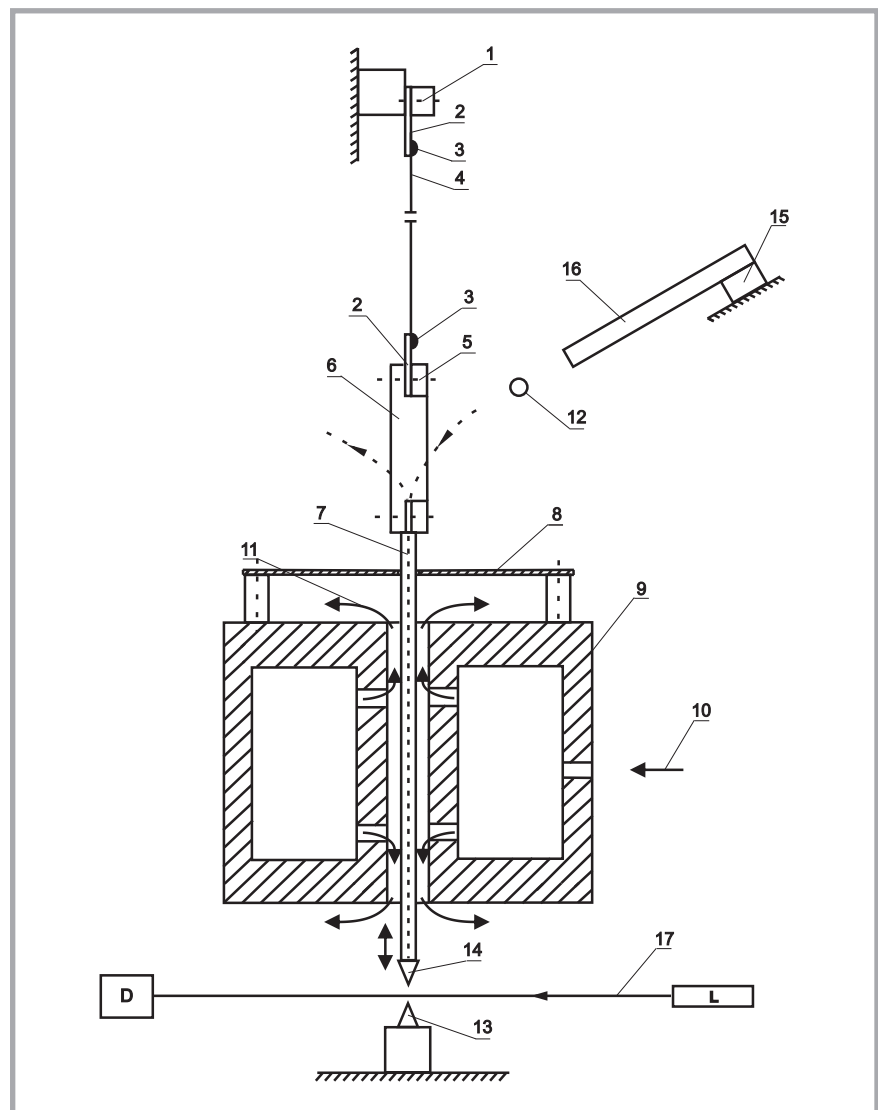

Figure 1. *Scheme of impulse mechanical spectrometer (IMS); 1 - top clamp, 2 - plate, 3 - glue, 4 - fibre, 5 - lower clamp, 6 - connecting frame, 7 - bearing movable rod, 8 - air shield, 9 - air bearing, 10 - compressed air inlet, 11 - air outlet, 12 - steel ball, 13 - stationary edge, 14 - movable edge, 15 - electromagnet, 16 - glass tube, 17 - laser beam, L - laser, D - detector.*
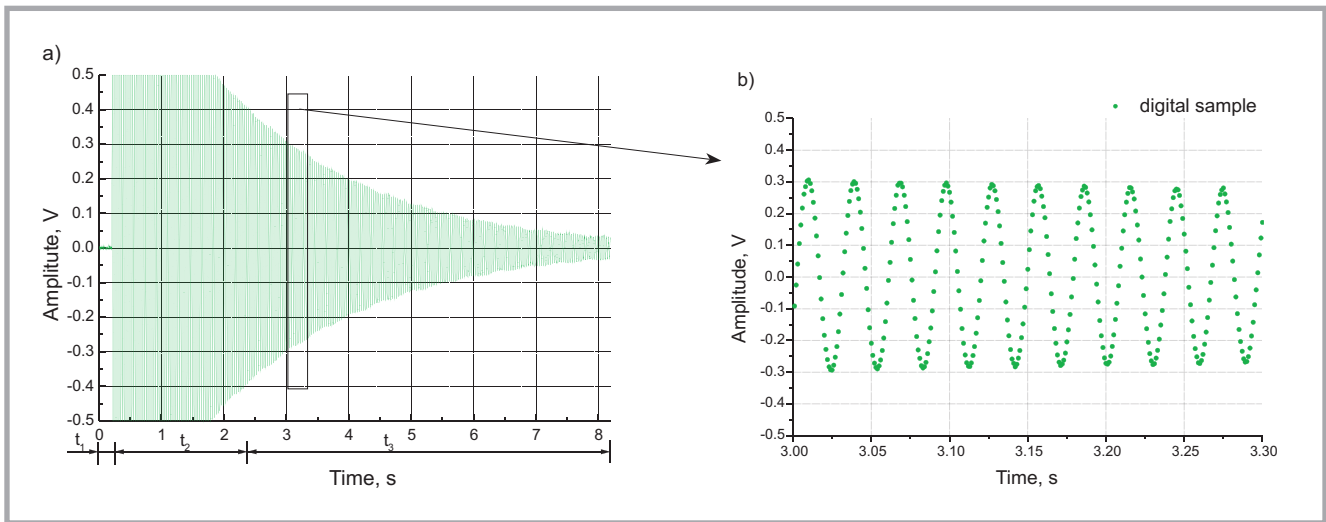
Figure 2. Amplitude of vibration versus time; a) total spectrum, b) zoom of selected part.

ticity using laser techniques, an impulse mechanical spectrometer for determining the mechanical Young's modulus under dynamic conditions was developed and produced.

Impulse mechanical spectrometer (IMS)

Existing methods of the examination of fibres using the diffraction of coherent light are static methods. The dynamic modulus is determined in the process of the longitudinal vibration of the elastic deformation of fibres. The monitoring of longitudinal vibration yields information about the various properties of samples. One way to determine the frequency of vibration is the determination of decaying oscillation in the time-domain. The optical method is successfully used as a dynamic testing method because of its high sensitivity, non-inertia, and non-contact. A description of the impulse mechanical spectrometer is presented in work [14].

Experimental

Materials

Studies were carried out on fibres of varied thickness:
- Polyester fibre of diameter d_n = 154.4 μm (designation PET150), the Rhodia company (France),
- Polyester fibre of diameter d_n = 200 μm (designation PET200), the Rhodia company (France).

The fibres were chosen because their thickness allowed to perform the test on both the IMS and DMA.

Methods

A scheme of the impulse mechanical spectrometer is shown on *Figure 1*.

One end of the sample of fibre 4 examined was fixed to the plate 2 by glue 3 in top clamp 1 and another one was mounted in lower clamp 5 to bearing rod 7 with a connecting frame. Fibre 4 with additional weights 6 and 7 was introduced in the vibration by an impulse force as a result of the impact of decreasing steel ball 12. The vibration of bearing movable rod 7 changed the distance between edges 13 and 14. Laser beam 17 from laser L passes through the slit and generates a diffraction pattern. Changing this slit affected the diffraction image and, specifically, the light intensity in the zero fringe, where detector D, transforming the optical signal into an electrical signal, was placed. This signal was amplified, digitalised by a 12-bit A/D converter, recorded in computer memory and processed by custom software.

The set-up for the dropping of a metallic nodule consisted of electromagnet 15 and glass tube 16. At the flow of current through the electromagnet, the ball attracted by the electromagnet was placed at the top of the tube. The loss of electrical current caused a decrease in the glass tube. The direction of ball 12 was specified by tube 16 so that the ball kept connecting frame 6 and after reflection not intersect laser beam 17. Air bearing 9 should have a lack of resistance in the direction selected, which was possible when there was an air bag between the moving surfaces. In *Figure 1* the inlet of compressed air is denoted by 10 and

the outlet by 11. Air shield 8 damped the mechanical turbulence when air flows through the bearing.

The initial load of the fibre applied was not bigger than 0.5 cN/tex.

After completing the initial setup there was a series of ten measurements for each test sample.

Measurements were carried out for the fibres, with the length of samples being around 50 mm. For each type of fibre 5 samples were tested.

In the time of the measurements the mechanical loss spectrum obtained was sometimes with mechanical and electrical disturbance. In this situation new measurement was necessary. The mechanical disturbance of vibrations are caused by the torsion oscillations of the frames when air flows through the bearing, imperfect impact of the ball (sometimes the ball does not impact the center of the frame) and when the laser beam is cut by the ball. The electrical disturbance depends on electromagnetic pulses and electric hum.

The time-domain spectrum (*Figure 2*) is analysed by mathematical functions of the form $g(t)$ [15, 16] with parameters: A, β, n, ω, φ.

$$g(t) = A \cdot e^{-(\beta t)^n} \cdot \sin(\omega t + \varphi) \quad (1)$$

where:
A – amplitude in V,
β – damping coefficient in s^{-1},
ω – resonant frequency in s^{-1},
φ – phase in rad,
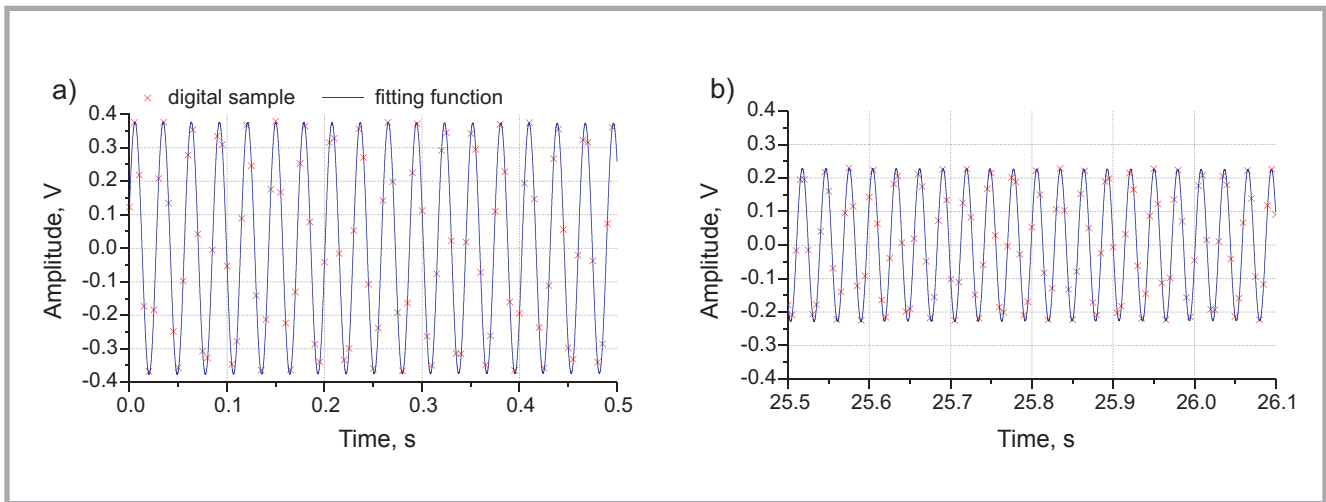n – parameter,
t – time in s.

Figure 3. *Graphical chart: a) beginning of the chart , b) end of the chart. It is the effect of the impulse force.*

Initial values of the parameters were analysed by the graphic method. By putting dozens of periods of mechanical loss spectra and the fitting function on one chart we can change the parameters (A, β, n, ω, φ), to the imposition of two charts. After the introduction to the program of initial values of the parameter received we obtained an exact fitting. The quality of this fitting was analysed graphically by comparing the mechanical loss spectra in one chart. In addition, the least square method was used. The graphical chart is shown in ***Figure 3***. If n is not equal to one then the decay of the amplitude is not exponential.

When the fitted value of parameter n is equal to one, then the fitting function can be described and the decay of the amplitude is exponential[15, 16]:

$$g(t) = A \cdot e^{-(\beta t)} \cdot \sin(\omega t + \varphi) \quad (2)$$

The application of the function of form (2) means that the Kelvin-Voigt model is the mathematical model of the test object [17]. Parameters β and ω received make it possible to calculate Young's modulus E, and the dimensionless logarithmic decrement of damping. In the calculation the mass of fibres is omitted because it is insignificantly small over the weight of the load.

$$\sigma = \frac{F}{S}, \quad \varepsilon = \frac{\Delta L}{L}, \quad k = \frac{F}{\Delta L}, \quad (3)$$

$$\omega_0^2 = \frac{k}{m}, \quad \omega^2 = \omega_0^2 - \beta^2$$

where:
σ – stress in Pa,
F – force in N,
S – cross section of the fibre, $S = \pi d_n^2/4$, in m^2,

ε – relative elongation,
L – length of the sample in m,
k – coefficient of elasticity in N/m,
m – mass of the load in kg.

Table 1. *Dynamic modulus of PET 150.*

Parameters	PET 150					
	Sample					Total
	1	2	3	4	5	
Average values of dynamic modulus of elasticity E_{IMS}, MPa	9915.5	9932.5	10893.6	9887.6	9988.1	10123.5
Coefficient of variation $v_{E_{IMS}}$, %	0.10	0.10	0.20	0.10	0.10	3.90
Relative random error of average values $u_{E_{IMS}}$, %	0.07	0.07	0.14	0.07	0.07	1.11
Relative uncertainty $U'_{E_{IMS}}$, %	3.81	4.23	3.73	3.80	3.68	3.80

Table 2. *Dynamic modulus of PET 200.*

Parameters	PET 200					
	Sample					Total
	1	2	3	4	5	
Average values of dynamic modulus of elasticity E_{IMS}, MPa	10594.7	10680.2	11044.6	11127.6	11036.9	10896.8
Coefficient of variation $v_{E_{IMS}}$, %	0.10	0.05	0.08	0.20	0.30	2.00
Relative random error of average values $u_{E_{IMS}}$, %	0.07	0.04	0.06	0.14	0.21	0.57
Relative uncertainty $U'_{E_{IMS}}$, %	3.90	3.90	3.90	3.90	3.90	3.90

The dynamic Young's modulus and logarithmic decrement of damping δ were calculated from the following dependencies [17, 18]:

$$U_{E_{IMS}} = \sqrt{\left|\frac{\partial E_{IMS}}{\partial m} \cdot U_m\right|^2 + \left|\frac{\partial E_{IMS}}{\partial L} \cdot U_L\right|^2 + \left|\frac{\partial E_{IMS}}{\partial d_n} \cdot U_d\right|^2 + \left|\frac{\partial E_{IMS}}{\partial \omega} \cdot U_\omega\right|^2 + \left|\frac{\partial E_{IMS}}{\partial \beta} \cdot U_\beta\right|^2}$$

$$U_{E_{IMS}} = \sqrt{\left|\frac{4L}{\pi d_n^2}(\omega^2 + \beta^2) \cdot U_m\right|^2 + \left|\frac{4m}{\pi d_n^2}(\omega^2 + \beta^2) \cdot U_L\right|^2 + \left|m\frac{8L}{\pi d_n^3}(\omega^2 + \beta^2) \cdot U_d\right|^2 + \left|m\frac{8L}{\pi d_n^2}\omega \cdot U_\omega\right|^2 + \left|m\frac{8L}{\pi d_n^2}\beta \cdot U_\beta\right|^2}$$

(6)

Equation 6.

$$E_{IMS} = \frac{\sigma}{\varepsilon} = \frac{F\,L}{S\,\Delta L} = k\,\frac{L}{S} =$$
$$= m\,\frac{L4}{\pi d_n^2}(\omega^2 + \beta^2) \qquad (4)$$

$$\delta = \frac{2\pi\beta}{\omega} \qquad (5)$$

For example PET 150, assuming values m = 0.09055 kg, d_n = 154.4 μm, L = 0.036 m, ω = 2πf = 238.64 Hz and ß = 1.748 Hz, we can get values of the dynamic Young's modulus E_{IMS} = at a level of 9915.5 MPa.

The uncertainty of the measurements was calculated from the following relationships:

■ the uncertainty of complex from **Equation 6** [19]:
■ the relative uncertainty of complex from **Equation 7**:

$$U'_{E_{IMS}} = \frac{U_{E_{IMS}}}{E_{IMS}}100 \qquad (7)$$

The results of the study are presented in **Tables 1** & **2**.

Dynamic mechanical analysis (DMA)

Dynamic mechanical analysis (DMA) measures the modulus and damping properties of materials which are deformed under periodic stress. DMA can be used to evaluate a wide variety of material types (polymers, composites ceramics and other materials). It is particularly useful for evaluating polymeric materials which exhibit time, frequency and temperature effects on mechanical properties because of their viscoelastic nature. In DMA, dynamic mechanical properties, such as the dynamic modulus in polymers are measured as a function of temperature and frequency. IMS works on resonance frequency while DMA uses driven oscillation. A DMA device does not allow the examination of samples with a length exceeding 30 mm, therefore it does not exactly match the values of the lengths used in the study on the impulse mechanical spectrometer. However, it saved the condition concerning the value of the preliminary stress.

The sample is mounted in the clamp (**Figure 4**), one part of which is stationary while the other is movable and connected to the drive motor. Thus the motor directly affects the deformation of the sample.

The drive motor is used to deliver force or stress to the moving drive block. This motor is non-contact in nature, in that the fixed motor assembly is not physically in contact with the movable drive block. The optical encoder measures the resulting displacement of the moving drive block. For smooth, noise-free and continuous delivery of force, the moving drive block is suspended by an air bearing.

The DMA (**Figure 5**) instrument contains all the necessary electronics and software needed to perform experiments and store the results. There is a flash disk in the cabinet that saves parameters vital to system operation if power is interrupted. The touch screen enhances the overall ease and displays valuable information during set-up, calibrations and experimentation. It has control features such as the ability to setup, start, stop or reject experiments.

■ Experimental

Tests were performed only for thick fibres because, despite the assurances of the manufacturer, it is only possible to examine fibres with diameters ranging from 20 to 800 μm. There are some problems in clamping thin fibres, because the effect of samples sliding is observed. Therefore measurements were carried out for filaments PET 150 & PET 200. Changes in the dynamic Young's modulus were recorded for the frequency range 0.01 - 160 Hz (**Figure 6**).
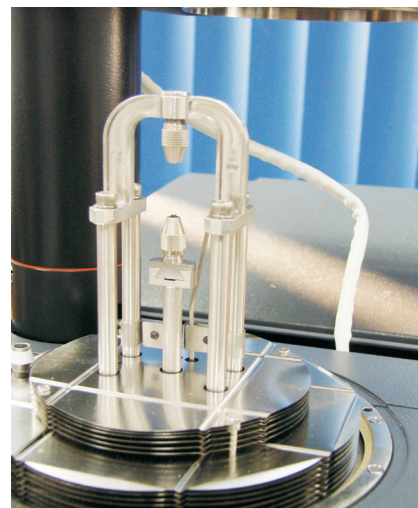

Figure 4. *Tension clamp (fibers) [20].*

Figure 5. *General view of Dynamic Mechanical Analysis Q800 (DMA) [20].*

TA Instrument Co. states that the accuracy of modulus determination is 1% [20].

The number of tests was n = 5 for PET 150 and n = 7 for PET 200. From the charts values of the modules were received for the average value of the frequency f at which the module on the impulse mechanical spectrometer was read out.
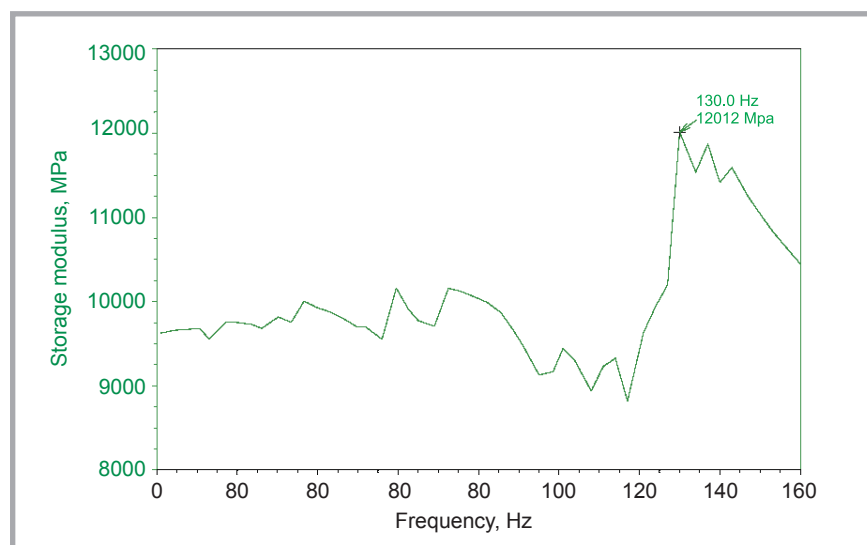

Figure 6. *Changes in the dynamic Young's modulus for filament PET 150 for the frequency range 0.01 - 160 Hz.*

Table 3. *Dynamic modulus.*

Parameters	Fibres	
	PET 150	PET 200
Average values of dynamic modulus of elasticity E_{DMA}, MPa	9984.6	11028.4
Coefficient of variation $v_{E_{DMA}}$, %	3.90	4.70
Relative random error of average values $u'_{E_{DMA}}$, %	4.84	5.83

The results of the measurements and calculations of variation indicators are given in **Table 3**.

Results and discussion

Statistical analysis of the comparative values of dynamic Young's modules obtained by means of the impulse mechanical spectrometer and DMA device was conducted using the two-tailed Student's t-test for samples of unequal variations at significance level α = 0.05.

The results of the analysis are presented in **Table 4**.

Table 4. *Results of statistical analysis.*

Statistics t	Fibres	
	PET 150	PET 200
Calculated	0.754	0.662
Limited	2.571	2.447

Comparison of the values of the dynamic modulus obtained from the new IMS and those received from the DMA device showed that they are very similar.

For filaments PET 150 and PET 200 statistically no differences between the average values from both instruments were found.

Analysing the values of the modules determined on the impulse mechanical spectrometer for the cycles of individual samples (**Tables 1** & **2**), it can be concluded that the value of preliminary stress applied does not exceed the limit of the proportionality fibre test. Variation in modulus values in each cycle and for all samples of fibres tested is very low, which indicates that there was no permanent deformation which would significantly change the values of modulus elasticity.

Conclusions

On the basis of the results of the research obtained the following conclusions can be drawn:

1. Values of the dynamic modulus from the impulse mechanical spectrometer developed are compatible with those from DMA.
2. The advantage of the system developed is the possibility of testing samples of thin and long fibres, which is not possible with DMA.
3. The DMA method does not require mathematical formulas to determine Young's modulus, but calibration of the DMA instrument must be performed, while the IMS leverages mentioned before require mathematical formulas and do not need calibration.
4. The method proposed for determining the dynamic Young's module provides a very interesting and very useful alternative to an expensive and not always accurate instrument.
5. The parameter set comprising the dynamic Young's modulus for longitudinal vibrations can find application in material engineering in industry.
6. The construction of IMS allows to determine the dynamic Young's modulus for textile fibres with both individual and bunches of fibre for one frequency of free vibration of floating. Changing the length of the sample allows to obtain a different frequency of free vibration. DMA measurements of the dynamic Young's modulus were tested in the range 0.01 - 160 Hz.

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