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TWIST LEVEL INFLUENCE ON THE DYNAMIC MECHANICAL CHARACTERISTICS OF POLYAMIDE FILAMENT

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It is well known that twist improve the mechanical properties of short staple fiber yarns. Twist is often used to give continuous yarn integrity and force assembly of single fibers to behave as single unit. It is therefore required to know influence of twist level on selected mechanical properties of yarns. In this work the influence of twist level on the dynamical mechanical characteristics of polyamide (PA) filament yarn is investigated. The simple models valid for static modulus are adopted.

INTRODUCTION

The dynamic mechanical analysis (DMA) is commonly used to characterize a material in response to vibration forces. DMA enables investigation of stress (or deformation) oscillations parameters (frequency, waveform, amplitude) and temperature influence on the deformation (or stress) changes under selected mode of deformation (tensile, bending, compression etc.)The dynamic mechanical thermal spectrometer DMA DX04T developed by RMI Ltd. Czech Republic provides highly sensitive tool for reproducible measurements of fine dimensional changes during heating, cooling or even at extremely long isothermal measurements. It is well known that twist improve the mechanical properties of short staple fiber yarns. Twist is often used to give continuous yarn integrity and force assembly of single fibers to behave as single unit. It is therefore required to know influence of twist level on selected mechanical properties of yarns. In this work the influence of twist level on the dynamical mechanical characteristics of PA filament yarn ire investigated. The simple models valid for static modulus are adopted.

DYNAMICAL MECHANICAL ANALYSIS

The dynamic mechanical analysis [DMA] is commonly used to characterize a material in response to vibration forces. DMA enables investigation of stress (or deformation) oscillations parameters (frequency, waveform, amplitude) and temperature influence on the deformation (or stress) changes under selected mode of deformation (tensile, bending, compression etc.).

The viscoelastic stress/strain relations under oscillatory strain/stress conditions having frequency ω at a given temperature are in the form

$$\sigma(t) = \varepsilon^{o} (E'\sin \omega t + E''\cos \omega t) \qquad (1)$$

and

$$\varepsilon(t) = \sigma^{o}(D'\sin \omega t + D''\cos \omega t). \qquad (2)$$

The σ^0 is stress amplitude and ε^0 is deformation amplitude. The time-dependent properties of materials are characterized by storage and loss moduli E'_i and E''_i, and the storage and loss compliances D'_i and D''_i such that

$$E' = (\sigma^o/\varepsilon^o)\cos \delta,\ E'' = (\sigma^o/\varepsilon^o)\sin \delta, \qquad (3)$$

$$D' = (\varepsilon^o/\sigma^o)\cos \delta,\ D'' = (\varepsilon^o/\sigma^o)\sin \delta. \qquad (4)$$

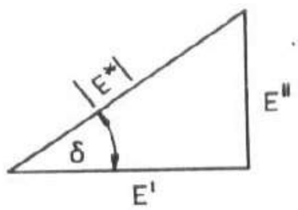
For anisotrotic materials are these quantities dependent on the direction. It is suitable to introduce complex modulus E^* and complex compliance D^*. The simple relations are valid

$$E^* = E' + i E'', \quad D^* = D' + i D''. \qquad (5)$$

The storage modulus is then equivalent to real part and loss modulus to the imaginary part of complex modulus. The same is valid for compliances. The elastic storage modulus E' is proportional to the energy fully recovered per cycle of deformation, and the imaginary component, E'' is proportional to the net energy dissipated per cycle in the form of heat. The dynamic storage modulus E' is the component which is in-phase with the applied strain and E'' is the component, which is 90^o out-of-phase. Tangent of phase angle $tg\,\delta$ (loss tangent, damping factor) is defined as

$$tg\,\delta = E''/ E', \quad tg\,\delta = D''/ D'. \qquad (6)$$

For solids, which are purely elastic, $tg\,\delta$ equals zero. Low damping materials such as metals and quartz conform fairly closely to the ideal while polymers have values of δ of the order of several degrees. From the expressing the complex modulus in complex plane (see Picture 1) it can be defined th following relations



Picture 1. Modulus in complex plane

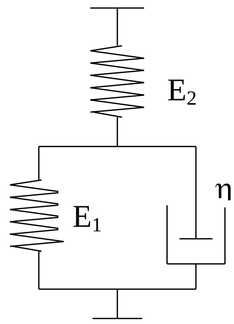
$$E = |E^*| = \sqrt{E_r^2 + E_i^2}\,,$$
$$E' = |E^*|\cos\delta, \qquad (7)$$
$$E'' = |E^*|\sin\delta = \eta^*\omega.)$$

Measure of dissipated energy is loss factor defined by the relation

$$tg\,\delta = E_i / E_r, \quad tg\delta = \eta^*\omega/ E_r, \qquad (8)$$

where η^* is viscosity of fibrous structure and ω is frequency of cyclic loading.

The dynamic moduli, compliances and loss tangent can be evaluated from formal viscoelastic models. Simple standard viscoelastic linear body (three element model having parallel arrangements of spring and Maxwell element) is shown in Picture 2.



Picture 2 Standard linear viscoelastic body

It is well known that stress relaxation o can be described by relation

$$E(t) = E_\infty + (Eo - E_\infty)*\exp(-t/\tau). \qquad (9)$$

The $E_\infty = E_1 + E_2$ is equilibrium modulus, $Eo = E_2$ is initial modulus and $\tau = \eta/E_2$ is relaxation time (these parameters corresponds with parameters of springs and dashpot standard viscoelastic body- see fig. 2). Corresponding relations for real and imaginary parts and loss tangent are in the form

$$E^{'}(\omega) = \frac{E_{\infty} + E_0(\omega\tau)^2}{1+(\omega\tau)^2},$$

$$E^{"}(\omega) = \frac{(\omega\tau)*(E_0 - E_{\infty})}{1+(\omega\tau)^2}, \quad (10)$$

$$tg\delta(\omega) = \frac{(\omega\tau)*(E_0 - E_{\infty})}{E_{\infty} + E_0(\omega\tau)^2}.$$

From these relations it can be deduced that:

- $tg\,\delta$ and E'' create peak at some frequency (frequency dependence id bell shaped)
- frequency dependence of E' is sigmoidally increasing function

For real materials with non-linear viscoelastic response are these dependencies more complicated.

By measuring the stress/strain amplitude ratio and phase angle at different frequencies of oscillation, the above material characteristics can be determined in the frequency domain. Subsequently, these data can be processed to compute the functions E(t) and D(t) in the time domain by performing inverse Fourier transformation.

Since it is impossible to obtain the complete range of frequencies required to evaluate the relaxation functions or creep compliances within reasonable time limits, dynamic testing of polymers is usually accelerated by expanding the experimental frequency range using the time-temperature or temperature-frequency equivalence principle [4]. The latter implies that a correlation can be established between the viscoelastic characteristics at a base temperature T_o and the respective material characteristics at a different temperature T through a parallel shift in the respective scale. For example, using this principle, the storage moduli E´ at any temperature T can be determined from the equation

$$E'(\omega,T) = E'(\omega a_T, T_o). \quad (11)$$

where a_T denotes the shift factor. The loss module, and the storage and loss compliances can be obtained from a similar relation. Note that the time-temperature equivalence is an intrinsic material property and must be established experimentally.

Because of the time-temperature equivalence in viscoelastic materials, the dynamic mechanical behaviour may be tested against frequency as well as against temperature, when the effect of longer time (lower frequency) is equivalent to that of higher temperature. Thus, the peaks frequency ω_P (for example defining T_g) of the dynamic mechanical characteristics (e.g. loss modulus) will be shifted to a lower temperature as the frequency is decreased. The frequency-temperature dependence is usually expressed by an Arrhenius type equation, as follows

$$\omega_P = A\exp(-\frac{\Delta E}{RT}), \quad (12)$$

where ΔE is the activation energy for relaxation, corresponding to the energy barrier for polymer chain movement from one location to another and A is pre-exponential factor (corresponding to the activation entropy).

The dynamic storage modulus, loss modulus and damping factor [loss tangent] are simply obtainable from DMA measurements. The dynamic storage modulus is a measure of the material stability to store energy and is commonly used for an indication of inter-atomic potential. Loss tangent is one of the most sensitive measures of atomic motion, particularly suitable for the evaluation of structural relaxation activated by a thermal process.

For yarns is loss tangent connected with the dissipation of frictional energy.

INFLUENCE OF TWIST ON THE MODULUS

In 1907 Gegauff proposed a simple analysis to correlate the twist angle of yarn with the yarn modulus. From the simple helix model of yarn is twist angle defined as [1]

$$tg\alpha = \pi DZ, \quad (13)$$

where R is yarn diameter, α is helix angle and Z is number of twists. From helix geometry is yarn modulus E_y simple function of fiber modulus E_f in the form

$$E_y = E_f \cos^2\alpha = \frac{E_f}{1+\pi^2 D^2 Z^2}. \quad (14)$$

The yarn diameter id approximately expressed as

$$D = \sqrt{\frac{4T}{\pi\rho\mu}}, \quad (15)$$

where ρ is fiber density, T is yarn fineness, μ is yarn mean packing density (for filament yarns is $\mu \approx 0{,}7$). Filament yarn modulus is then equal to

$$E_y = \frac{\mu\rho}{\mu\rho + 4\pi T Z^2}. \quad (16)$$

By using of well known Koechlin equation $Z = \alpha_k\sqrt{T}$ is yarn modulus approximately given by equation

$$E_y = \frac{\mu\rho}{\mu\rho + 4\pi Z^4/\alpha_k} = \frac{A_1}{A_1 + A_2 Z^4}, \quad (17)$$

where α_k is twist coefficient. In the case of this simple model validity leads the dependence of 1/E(Z) on Z^4 to the linear form $1/E(Z) = 1 + A_2Z^4/A_1$.

However, the practically measured yarn modulus obeys a stronger dependence on twist level. White et. all proposed more complicated analysis based on the continuum mechanics and including of interfilament friction and transverse forces as well [6]. Their final equation has the form.

$$E_y = E_f\left(\frac{1}{4} + \frac{9C}{4} + \frac{3C}{(1-C)}\ln\sqrt{C}\right), \quad (18)$$

where $C = \cos^2\alpha$. This simple model leads to the underestimation of yarn modulus. In the work [2] they introduced anisotropy ratio R equal to longitudinal modulus E_f divided by shear modulus of filament. The yarn modulus is expressed as

$$E_y = E_f \frac{\left[\left(\frac{3C+1}{2RC}\right) + \frac{(1-R)^2}{R^3\tan^2\alpha} - \ln\left(\frac{(1-R)C+R}{C}\right)\right]}{\left(\frac{1+R^2}{R^2}\right)}. \quad (19)$$

These models show that higher twist leads to the lowering of yarn modulus. In this work is the simple model used for the case of dynamic modules.

TERMOMECHANICAL ANALYZER TMA CX/04

The dynamic mechanical thermal spectrometer DMA DX04T was developed by RMI Ltd. Czech Republic to provide highly sensitive tool for reproducible measurements of fine dimensional changes during heating, cooling or even at extremely long isothermal measurements. DMA DX04T offers seven deformation modes including: compression, tension, three-point bending, single cantilever, dual cantilever, cylinder-cylinder [annular pumping] and shear.

This apparatus brings a new concept in compact desktop analyzers. Force motor generates very precise deformation with sinusoidal or any others waveform. Real generated waveform of force and real corresponding deformation are measured by special tensometer and displacement sensor. Whole force and deformation spectrum is processed by DMA control unit and transferred via 10 Mb Ethernet link to the PC. All data are recorded and evaluated by PC by using of programs under Windows, including new concept of data processing based on Fast Fourier Transformation.

In the TMA CX-04 the sample is placed on the movable sample tray connected with displacement sensor, which measures dimensional changes of the sample. This construction design improves temperature stability and decreases sample holder deformation.

The most important part of the TMA instrument is displacement sensor. A completely new concept of displacement sensor different from classical LVDT [Linear Variable Differential Transformer] ensures linearity [better than 0.1% μm resolution], low noise [typically 0.02 μm without signal filtering], good temperature stability [typically 0.05 μm/^{0}C] and small drift [0.1 μm/day].

A novel electronic system is used to control load generating force motor, which can be expanded to flexible high performance dynamic operation. Loading up to 1000 mN [1mN steps] may be programmed in two modes: constant load and cycle load. By means of precise electronic and computer control it is possible to change loading during the measurement according to specified program.

A new synthetic oscillation mode is based on generation of force waveform, which is synthesis of different frequencies. Following FFT data evaluation allow to measure multiple frequencies at the same time. A high number of frequencies from 0.0001 to 100 Hz can be measured in combination with classic frequency sweep scan method at shorter time than on standard DMA analyzers. The combination with strain sweep scan mode is also available.

To achieve good temperature spread into the sample the advanced furnace with turbulent moving atmosphere is used. Various inert gasses [nitrogen, helium etc.] may be utilized. The three thermocouples are available for high precise measurement of the temperature of long samples.

Sample dimensions for tension mode are: maximum length. = 46 mm [30 mm active], maximum width. = 12 mm and thickness up to 5 mm.

EXPERIMENTAL PART

The PA multifilament continuous yarn with fineness 11.tex having integrity twist 20 [1/m] has been used. Multifilament yarn is composed from 32 filaments. The twist was imparted by using of the Zweigle D312 apparatus. Samples have been prepared with 20, 800 a 1600 twist [m^{-1}]. The pre loading by the 40 cN was used. Static modules ES were measured on the TIRATEST tensile machine

The dynamic measurements were realized on the dynamic mechanical thermal spectrometer DMA DX04T in the tensile mode. The temperature was kept constant at 25^{o}C. The frequencies 1, 5 and 10 Hz were selected.

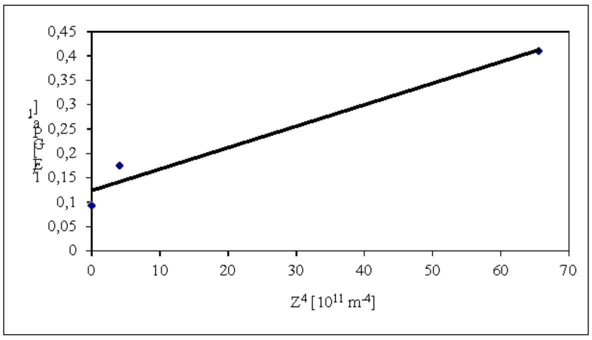
RESULTS AND DISCUSSION

The static E modulus for selected twist level, corresponding standard deviation s and variation coefficients v are given in the tab. 1.

Table 1

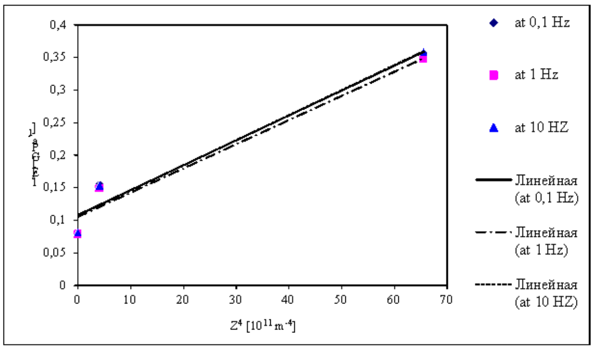
Twist [m^{-1}]	E [GPa]	s [GPa]	v [%]
fibers(0)	12,931	2,290	17,71
20	10,824	0,365	3,38
800	5,726	0,401	0 07
1600	2,440	0,343	14,06

The dependence of reciprocal modulus on the Z^4 is shown in the Picture 3.

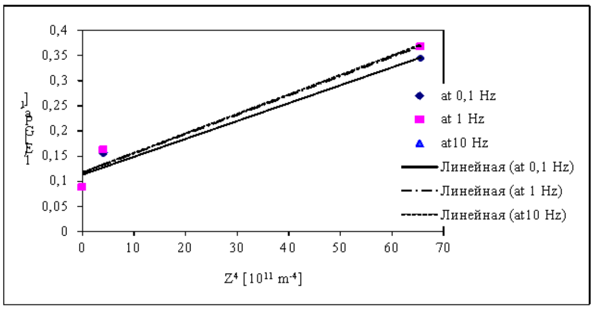


Picture 3: The dependence of reciprocal static modulus on the Z^4

The corresponding graphs for expressing the dependence of dynamic modulus on the twist are given in Picture 4 and 5.



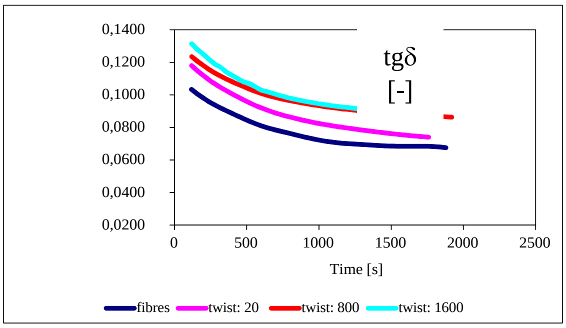
Picture 4: The dependence of reciprocal dynamic modulus on the Z^4 for load amplitude 1000 – 2000 mN



Picture 5: The dependence of reciprocal dynamic modulus on the Z^4 for load amplitude 200 – 400 mN

It is clearly visible that trends conform to linearity but due to limited number of points it is not useful to compute parameters of these lines.

Time dependences of loss tangent is shown in the Picture 6.



Picture 6: The time dependence of loss tangent of PA with various twist level for load amplitude 1000 – 2000 mN

From fig. 6 is clear that loss factor is minimal for loose filaments and is increasing function of twist level. The reason of these phenomena is increasing of number of contacts between filaments due to twist mainly. In dynamical mode of deformation are contacts responsible for inter-filament friction and corresponding energy loss. This behavior is in accordance with definition of loss factor and ratio of real and imaginary modulus.

CONCLUSION

It was evaluated that dependence of static and dynamic modulus on the twist level can be explained by simple geometrical model. The loss factor is measure of number of contacts between filaments and therefore is increasing function of twist. The described DMA apparatus is suitable for measurement of fibers. There is still open question if the twist of filaments has influence on the location of peaks on tg δ spectrum.

ACKNOWLEDGEMENTS

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BIBLIOGRAPHY

1. *Gegauff C.* Bull Soc Ind Mulhouse 77,153 (1907)
2. *Y. Rao, R.J. Farris* J.Appl. Polym. Sci. 77, 1938,(2000)
3. *A. R. Bunsel* Fibre Reinforcements for CompositM materials, ,Elsevier, chap 6 (1988)
4. *A.G. Mc Crum, G. Williams* Anelastic and Dielectric Effects in Polymeric Solids, (1991)
5. *M. Meloun, J. Militký and M. Forina* Chemometrics in Analytical Chemistry vol. II, Interactive Model Building and Testing on IBM PC, Ellis Horwood, Chichester (1994)
6. *White J.L., Cheng C.,C., Spruiell J. E.* J.Appl. Polym. Symp. 27, 275, (1975)

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