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Determination of stress–strain characteristics of thin polymer films on cylindrical specimens

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Abstract. An experimental method (TWCS method) was applied to determine the flexural elastic modulus E and some other stress-deformation characteristics of a thin-walled circular cylindrical polymer shell at compression. A methodology of test sample preparation and fixation as well as means of the measurement of the load P and the respective displacement Δ were developed. Two modes of testing the equipment were used in which the sequences of measuring $P \to \Delta$ and $\Delta \to P$ were implemented. Repeatability of measurements was evaluated. Optimal geometric parameters of test samples and the range of determinable values of modulus E were defined. A procedure of the calculation of the relationship between the tensile stress σ and relative elastic tensile deformation ε in the most deformed area of the sample was developed. Applicability of the TWCS method for multiple assessments of changes of deformational characteristics by using a single sample was proved. It was shown that creep, creep recovery, and stress relaxation tests can be performed by the TWCS method.

Key words: polymers, thin films, cylindrical shell, compression, test methods, stress-deformation characteristics.

1. INTRODUCTION

Thin film (thickness less than 0.3 mm) is a shape of sample that is commonly used for the investigation of the structure and properties of polymeric materials [1]. There are a variety of practical uses of polymers as a thin film. It is the only form in which samples can be obtained by casting from polymer solutions, emulsions, or other dispersions [2,3]. Compared to thick samples, thin samples have less temperature and concentration gradients when the impact of the environment on the structure and properties of polymer material, including stress–strain characteristics, is studied. Many traditional methods used to evaluate these characteristics are actually not suitable for thin samples. Hence the feasibility to create a method that enables performing stress–strain measurements in the range of small values of relative deformation (where irreversible transformations of the polymer structure have not yet occurred) is of particular interest.

For that reason a method to determine the elastic modulus for solving the problem of compression (or tension) of a thin-walled circular cylindrical shell with a certain thickness, radius, and length (TWCS method) with regard to the geometrical and physical nonlinearity was recently proposed [4,5]. A simple procedure was worked out for the calculation of the flexural elastic modulus E and the relationship between the highest tensile stress σ and the relative elastic tensile deformation ε in the most deformed area of the sample from experimental relationships between the loading force P and the respective displacement Δ (Fig. 1).

The main objective of the present work was to develop simple testing devices and techniques capable of a reliable measuring of the respective P and Δ values with proper accuracy, which would allow gentle sample fixation for testing as well as easy detachment of the sample after testing without damage. Obviously repeated testing of the same sample is possible in case the actual value of deformation is reversible and small enough.

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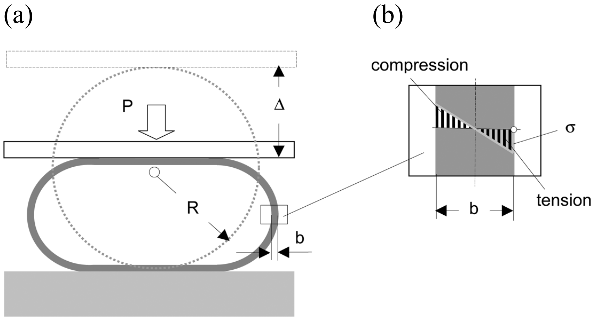

Fig. 1. Scheme of the compression of TWCS (a) and stress distribution in the most deformed area of the sample (b).

Another aim of this work was to expand the applicability of the TWCS method for the evaluation of some other stress–strain characteristics.

2. EXPERMENTS

2.1. Testing devices and procedure

The general testing scheme according to the TWCS method is shown in Fig. 2. The test sample was a strip of plane polymer film (I) with a definite length G, width L, and thickness b coiled in the shape of cylinder (II) with radius $R = G/2\pi$ and fastened in the measuring device (III). The sample was loaded between parallel plates athwart to the geometrical axis of the cylinder (IV). Force P and the corresponding displacement Δ were measured for a range of P and Δ values (see also Fig. 1), resulting in relationship $P(\Delta)$. Afterwards the sample was removed (V) and reverted to the initial plane shape (I).

Two diverse modes to obtain $P(\Delta)$ relationships were implemented in respective devices. According to mode I, the sample was gradually loaded by adding discrete calibrated weight to reach a certain load P. The respective Δ value was measured after each recurrent addition of weight. The sequence $P \rightarrow \Delta$ was carried out. The test is schematically depicted in Fig. 3.

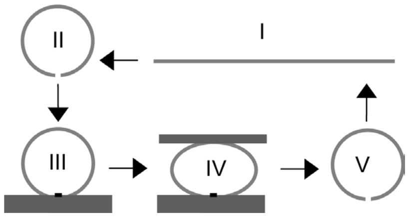

Fig. 2. Sample testing scheme according to the TWCS method: I – test sample, a strip of polymer film; II – sample coiled in the shape of cylinder; III – sample fastened in the measuring device; IV – sample loaded between parallel plates; V – removed sample.

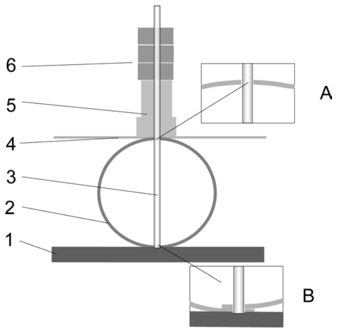

Fig. 3. Scheme of the test device, mode I: 1, base; 2, sample; 3, bar; 4, deforming plate; 5, guide; 6, weights. View of the upper (A) and the lower (B) part of the sample with holes.

To ensure precisely centred loading, the sample (with pre-cut holes) was placed on a smooth bar (to avoid friction). The deforming plate with a guide and weights was placed on the same bar. Any non-contact device can be used for measuring the displacement Δ. A cathetometer KM-8 was used in our work (measurement accuracy ±0.001 mm) [6]. The use of relevant laser equipment seems advisable [7].

Mode II provides gradual moving of the deforming plate to reach a certain value of displacement Δ and measurement of force P after each enlargement of Δ. The sequence $\Delta \rightarrow P$ was carried out.

The respective test device is schematically illustrated in Fig. 4. The sample was fixed on a ground plate by a special lock for the gentlest mounting. Displacement Δ was provided by lowering the rigid plate, which was fixed firmly to the movable part attached to a thumbscrew. The Δ value can be measured by any suitable device with an accuracy not less than ±0.02 mm. In our case we adapted a digital slide gauge with the measuring accuracy of ±0.01 mm. Electronic balances that allow measuring forces with an accuracy of not less than ±0.01 g can be successfully used for the determina-

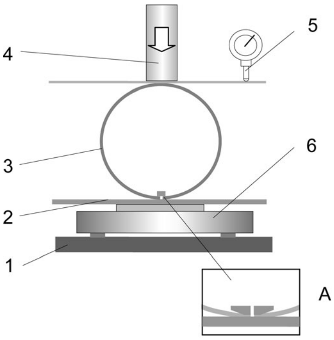

Fig. 4. Scheme of the test device, mode II: 1, base; 2, ground plate; 3, sample; 4, movable part with deforming plate; 5, displacement measuring device; 6, force meter. View (A) of the lower part of the sample with a lock.

tion of the force P. We used an electronic balance Precisa BC2200C.

Loading (mode I) or deformation (mode II) must be done at a rate corresponding to the quasistatic conditions. According to mode I, each next weight was gently applied manually within ~1 s. The respective Δ value should be measured after certain time (~2 s). The quasi-equilibrium Δ value corresponding to the total P value is reached already during this time, but noticeable creep has not yet occurred.

With mode II each Δ level was reached likewise within 1–2 s. The respective P value was determined after 1–2 s. The quasi-equilibrium P value, corresponding to the total Δ value, had already been reached during that time, but noticeable stress relaxation had not yet occurred.

2.2. Determination of the flexural elastic modulus *E*

The modulus was calculated from the experimental relationship $P(\Delta)$ [1]. According to [8], the dimensionless parameters α that characterize the relative displacement and β that describe the load, were used:

$$\alpha = \Delta / 2R, \tag{1}$$

$$\beta = PR^2 / EJ, \tag{2}$$

where:

E – flexural elastic modulus of the material,
$J = Lb^3/12$ – moment of inertia of the rectangular cross-section of the sample,
R – radius of the cylinder,
L – width of the sample,
b – thickness of the sample.

The value of E was determined according to a unified loading diagram [1,2]. The following calculation algorithm was used: 7–8 points from the experimental curve $P(\alpha)$ corresponding to $\alpha = 0.2-0.8$ were recalculated to the dimensionless function $\beta(\alpha)$. Afterwards the modulus E_α values were determined for each α value by comparison of experimental points with the corresponding points on the unified loading diagram with the equation:

$$E_\alpha = PR^2 / \beta J. \tag{3}$$

A special calculation program (in Excel) was created where geometrical parameters of the sample as well as experimental function $P(\alpha)$ are entered, and E_α values corresponding to certain α values were calculated. The average value of the modulus $\bar{E}$ was calculated as the arithmetical mean of seven–eight E_α values for the range of $\alpha = 0.2-0.8$. The value $\bar{E}$ is the flexural modulus of the investigated material.

Samples of several industrially produced polymer films with $R = 15$, 20, 25 mm and $L = 20$, 40, 60 mm were tested. Average modulus values $\bar{E}_{\mathrm{av}}$ and relative deviations from the average value $\Delta E = (E_{\max} - E_{\min})/\bar{E}$ for 4–6 parallel samples are shown in Table 1.

The value of the greatest relative tensile deformation ε in the most deformed area of the sample (see Fig. 1) can be calculated by using the unified relationship $\varepsilon_{\mathrm{red}}(\alpha)$ [2], where $\varepsilon_{\mathrm{red}}$ is reduced relative deformation:

$$\varepsilon_{\mathrm{red}} = \varepsilon(R/b). \tag{4}$$

The respective value of the tensile stress σ was determined according to Hooke's law for shell:

$$\sigma = \varepsilon_{\mathrm{red}} \frac{b}{R} \frac{E}{(1-\nu^2)}, \tag{5}$$

where ν – Poisson's ratio.

Accordingly the experimentally obtained relationships $P(\alpha)$ can be easily converted to the respective $\sigma(\varepsilon)$ relations.

2.3. Testing a single sample

One of the important goals of the present work was to ascertain the suitability of the TWCS method for repeated multiple assessments of changes of deformational characteristics by using a single sample. It is correct only if the actual tensile deformation ε is small and reversible, and therefore does not cause any permanent changes in the material structure.

The deformation ε, which corresponds to a certain α value, depends on the geometrical characteristics of the sample. It decreases with the growth of the specimen's radius R and with the decrease of the sample thickness b (see Eq. (4)).

For comparison, the calculated reachable ε values for various R and b at $\alpha = 0.5$ are summarized in Table 2. As can be seen, the respective ε values are very small.

It is important to emphasize that the values of displacement Δ are very large compared with the corres-

Table 1. Values of average modulus $\bar{E}_{\mathrm{av}}$ and relative deviation from the average value ΔE for some polymer films

Material	b, mm	$\bar{E}_{\mathrm{av}}$, GPa	ΔE, %
PVC–Z*	0.2 ± 0.01	2.7	2–4
PVC–S*	0.2 ± 0.01	2.3	2–5
PVC–C*	0.1 ± 0.01	2.7	2–5
PP**	0.3 ± 0.01	1.9	2–4
PVA***	0.14 ± 0.03	9.8	3–6

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* Polyvinylchloride cover films; ** polypropylene cover films; *** polyvinylalcohol film casted from water solution.

Table 2. Calculated ε values for various R and b values at $\alpha = 0.5$

b, mm	R, mm		
	15	20	30
0.1	0.027	0.020	0.014
0.2	0.054	0.041	0.027
0.3	0.081	0.061	0.041

ponding ε values, and therefore are easily measurable with adequate accuracy. The values of the displacement Δ corresponding to the ε value for $b = 0.1$ mm were compared with the absolute value of the elongation Δl of standard samples with a length of 25 mm in tensile test (see Table 3). As can be seen, Δ values are nearly two orders higher.

The fact that the deformation of a thin polymer shell is characterized by great displacements and relatively low respective deformations is an important advantage of the TWCS method.

It was of special interest to compare the repeatability of measurements of several parallel samples with repeated measurements of the same sample. It turned out that in the latter case repeatability was about one order higher. For instance, the testing of four parallel PVC samples showed that $\Delta E = 2.7\%$, while the testing of one and the same sample four times one by one showed that $\Delta E = 0.28\%$. Apparently repeatability is determined more by differences in the structure and geometrical parameters of samples than by differences in manipulations with the sample. This brings the perspective to introduce the testing of a "single" sample significantly nearer.

The testing of a single sample means that the sample is exposed to a specific external factor (temperature, radiation, environment, and others) for a certain time, tested, and then reverted. This cycle can be repeated.

A single sample was used to determine the dependence of the elastic modulus of PVA film on the moisture content in the material. It is known that water acts as a PVA plasticizer [9]. A dry sample (vacuum dried at 60 °C) was tested and then kept for a certain time in a desiccant (which provides ~55% of relative humidity) where it absorbed moisture. Then the sample was weighed, its moisture content was determined, and it was tested again. This cycle was repeated till the moisture content reached equilibrium. The experimentally determined dependence of the sample's moisture content and modulus on the exposure time is shown in Fig. 5a,b. The derived dependence of the modulus on the moisture content is shown in Fig. 5c.

Table 3. Calculated values of ε and respective Δ and Δl values for different R at $b = 0.1$ mm and $\alpha = 0.5$

	R, mm		
	15	20	30
ε	0.027	0.020	0.014
Δ, mm	15	20	30
Δl, mm	0.68	0.500	0.35
$\Delta/\Delta l$	22	40	86

2.4. Determination of relative change of modulus

As it was already mentioned, measurements of the load P for at least 6–8 α values (in the range $\alpha = 0.2–0.8$) are necessary to determine elastic modulus. It was interesting for practical use to check whether the relative change of modulus was equal to the relative change of the load P at a certain single α value. For this purpose the load P_α values, which corresponded to α values of 0.3, 0.4, and 0.5, were selected from the measurement series of PVA film with different moisture content (Fig. 6a).

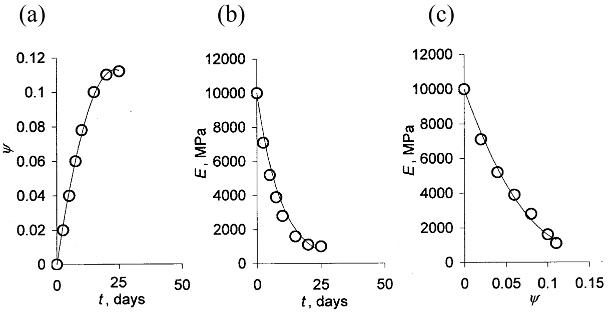


Fig. 5. Influence of PVA film exposure time t at 55% relative humidity on the water weight fraction ψ(a) and the elastic modulus E (b). Dependence of the elastic modulus E of PVA film on the water weight fraction ψ(c).

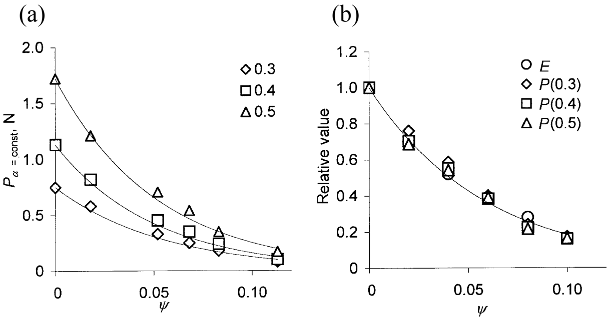


Fig. 6. Relationship between the deforming force at a certain relative displacement $P_{\alpha = \text{const}}$ and water weight fraction ψ(a); dependence of the relative values of the modulus E and $P_{\alpha = \text{const}}$ on the water weight fraction ψ(b).

As can be seen in Fig. 6b, the relative values of the modulus E/E_0 and the load $P_\alpha/P_{\alpha 0}$ practically coincide (E_0 and $P_{\alpha 0}$ designate the respective values for dry samples, and E and P_α the respective values for wet ones). So it is acceptable to determine experimentally only the initial value of modulus E_0 and to calculate the current values of modulus E from the ratio $E = E_0(P_\alpha / P_{\alpha 0})$. This significantly reduces the labour intensity of the experiment.

2.5. Experimental determination of creep and stress relaxation relationships

The TWCS method can be modified for studies of long-term stress–strain characteristics of polymer: creep (permanent deformation of material under stress) and stress relaxation (relieve of stress under constant strain) [10]. By using the test mode I, the time dependence of α was measured at a permanent load P (creep):

$$\alpha(t)\big|_{P=\text{const}}. \tag{6}$$

After a certain time t load P was removed and creep recovery was measured:

$$\alpha(t)\big|_{P=0}. \tag{7}$$

If necessary, both relationships can be recalculated to respective $\varepsilon(t)\big|_{\sigma=\text{const}}$ and $\varepsilon(t)\big|_{\sigma=0}$ relationships (see 2.2). Examples of creep and creep recovery curves of polypropylene (PP) samples are shown in Fig. 7.

Test mode II can be used to determine the stress relaxation relationship. The time dependence of the load P is measured at a fixed deformation α:

$$P(t)\big|_{\alpha=\text{const}}. \tag{8}$$

The relationship can be recalculated to the respective $\sigma(t)\big|_{\varepsilon=\text{const}}$. Stress relaxation curves of PP samples are demonstrated in Fig. 8.

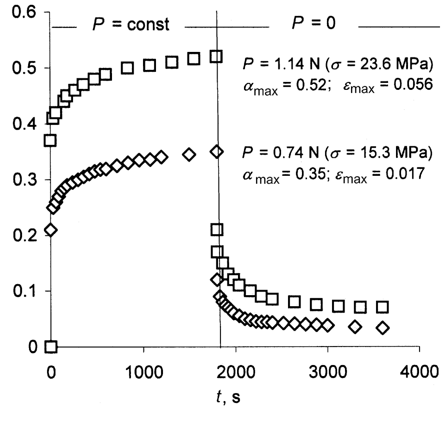
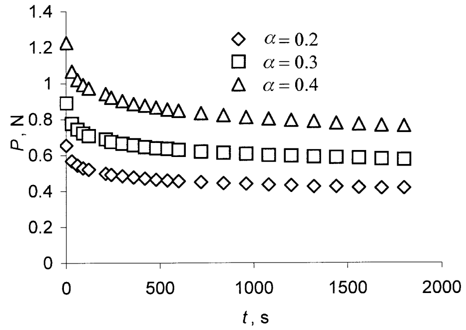


Fig. 7. Creep $\alpha(t)|_{P=\text{const}}$ and creep recovery $\alpha(t)|_{P=0}$ curves at two different values of P = const (PP sample: $R = 25$ mm, $L = 40$ mm, $b = 0.3 \pm 0.01$ mm).

(a)



(b)

	α		
	0.2	0.3	0.4
P_0, N	0.65	0.90	1.22
σ_0, MPa	0.055	0.074	0.102
P_f, N	0.41	0.59	0.77
σ_f, MPa	0.035	0.048	0.064

Fig. 8. Example of stress relaxation curves: $P(t)|_{\alpha=\text{const}}$; at $\alpha = 0.2$, 0.3, and 0.4 (PP sample: $R = 25$ mm, $L = 40$ mm, $b = 0.3 \pm 0.01$ mm) (a); respective initial values: P_0 (σ_0 calculated) and final values P_f (σ_f calculated).

2.6. Optimal geometric parameters of test samples and the range of determinable values of modulus E

Experience shows that acceptable results can be obtained for samples with a relatively wide range of geometric parameters: $b = 0.1$–0.3 mm, $R = 15$–45 mm, and $L = 10$–40 mm.

Selection of optimal geometric parameters is determined to some extent by the modulus E value of the material. If the E value is less than 600 MPa, the cylindrical specimen will noticeably flatten, even when $R < 15$ mm. Consequently, this value should be considered as the lower limit of the E value that can be determined by the TWCS method. The upper limit of the E value has not been firmly established. We succeeded in testing samples with E close to 10^4 MPa. High modulus polymers ($E > 10^4$ MPa) show mostly small elongation at break. There is concern that respective samples may fracture while preparing to test.

The probable thickness of the sample is basically determined by the manufacturing technology of polymer film. Our practice shows that for polymer with $E = 10^3$–10^4 MPa, the value of the thickness b should desirably be within 0.1–0.3 mm.

Other preferable sizes of the sample are determined by several considerations. In order to save the material it would be advantageous to reduce the radius R of the sample (the value of R determines the length of the sample: $2\pi R$) as well as the sample width L. However, with decreasing R value the accuracy of α measurement essentially decreases, whereas samples with $L < 15$ mm notably sway. Therefore the following optimal geometric characteristics of samples for testing by the TWCS method can be recommended: $R = 20$–30 mm, $L = 20$–30 mm, $b = 0.1$–0.3 mm.

3. CONCLUSIONS

1. Two modes of equipment applicable for measuring the load P and the respective displacement Δ at the compression of a thin-walled circular cylindrical polymer shell (TWCS method) with the aim to determine the flexural elastic modulus E and some other stress-deformation characteristics were developed and applied.
2. The TWCS method is suitable for repeated multiple assessment of changes of deformational characteristics with using a single sample in the case when the reachable tensile deformation ε is small enough, reversible, and does not cause any permanent changes of the material structure.
3. The TWCS method can be adapted for performing creep, creep recovery, and stress relaxation tests.

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Õhukeste polümeerkilede pinge-deformatsioonikarakteristikute määramine silindrilistel katsekehadel

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Polümeerkilede painde-elastsusmooduli E ja mõnede teiste pinge-deformatsioonikarakteristikute määramiseks kasutati õhukeseseinaliste silindrite (TWCS) meetodit. Arendati edasi katsekeha ettevalmistamise ja kinnitamise ning rakendatava jõu P ja vastava nihke Δ mõõtmise metoodikaid, kasutades kaht erinevat katseseadet, vastavalt jõu ning nihke laotamiseks. Hinnati mõõtmistulemuste korratavust. Selgitati välja katsekehade optimaalsed geomeetrilised parameetrid ja väärtuste vahemik, milles saab moodulit määrata. Arendati edasi arvutusmetoodikat tõmbepinge ja suhtelise elastse tõmbedeformatsiooni seose leidmiseks katsekeha kõige enam deformeeritud piirkonnas. Näidati, et TWCS-meetodit saab kasutada deformatsioonikarakteristikute korduvaks määramiseks samal katsekehal ja samuti roome- ning pingerelaksatsioonikatseteks.