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The method of determination of material shear elasticity in the course of its solidification

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A method is proposed for investigation of solidification processes based on the shear modulus determination of the material being solidified. A procedure has been developed to calculate that quantity. The proposed method has been approved in experiment.

Предлагается метод исследования процессов затвердевания, в основу которого положено определение модуля сдвига твердеющего материала. Разработана методика расчёта этой величины. Проведена экспериментальная апробация предложенного метода.

The existing methods provide the study of either the initial solidification stage when the liquid phase predominates (see, e.g., [1–4]), or its final stage when the rigid phase is predominant (e.g., [5, 6]). This work proposes a method which allows to monitor continuously the solidification process at any liquid/rigid phase ratio.

The rigid phase rigidity modulus is known to exceed by several orders that of the liquid phase. This fact forms a basis for the proposed method. The authors propose to measure the solidifying system rigidity modulus in time, taking into account the fact that the immense rigidity modulus value of the rigid phase (as compared to the liquid one) will provide an accurate determination of the rigid phase amount in the system.

When measuring the rigidity modulus, the shape of the solidifying material sample is to be kept. The authors propose to solve the problem by putting the solidifying material into a tube made of another rigid material. That composite sample is being subjected to a twisting moment M (see Fig. 1). Let the following quantity be considered:

$$\eta = C + C_1 + C_0, \tag{1}$$

where C_0 is the the twisting rigidity of the pendulum without the sample: C_1, the empty tube twisting rigidity; C, the twisting rigidity of inner cylinder consisting of the solidifying material. The method concept is to determine separately the rigidity of filled (η) and empty ($C_1 + C_0$) tube as well as C_0 Such an experiment provides the C and C1 values. Using the known solution of elasticity theory problem on a composite cylinder twisting (see [7], for example), we get the formulas

$$C_1 = \frac{G_1\pi(R^4 - r^4)}{2l}. \tag{2}$$

$$C = \frac{G\pi r^4}{2l}, \tag{3}$$

making it possible to determine the rigidity moduli of the substance filling the tube and of the empty tube (C and C_1, respectively) proceeding from the measured C and C_1 values. The following notations are used in (2) and (3): l , the tube length; r and R, the tube inner and outer radii, respectively.

A twisting pendulum described in [8] was used to determine the twisting rigidity in experiments.

Let ν, ν_1 and ν_2 be the oscillation frequencies of the pendulum with composite sample, of that with the empty tube and of the sample-free pendulum. For those quantities, the following formulas are valid:

$$\nu^2 = \frac{1}{4\pi^2}\frac{\eta}{I}, \tag{4}$$

$$\nu_1^2 = \frac{1}{4\pi^2}\frac{\eta_1}{I}, \tag{5}$$

$$\nu_2^2 = \frac{1}{4\pi^2}\left(\frac{C_0}{I}\right), \tag{6}$$

where where I is the pendulum inertia moment.

As is seen from (3), the measurement error of the solidifying material rigidity modulus G will be defined by measurement errors of the system inertia moment I, radius r, the sample length l, and rigidity C. The latter may attain several (up to ten) per cent of the quantity value itself. As is seen from the experimental data presented below, the rigidity of a solidifying material filling the tube does not exceed 10% of the tube rigidity. In other words, C may have values equal to root-mean-square deviation of the C_1. That is why determination of the solidifying material rigidity modulus using formula (3) is problematical. Basing on the above, the authors propose the experimental data processing procedure which allows to avoid those difficulties.

The experiment shows that the oscillation frequency of the pendulum without the sample is time-independent. This is explained by the fact that the elastic element is a metal wire substantially free of time-dependent processes (creep, etc.). When the pendulum is studied in combination with the tube (that is made of a polymer), time-dependent processes occur in the latter due to stresses; in particular, the sample creeps under axial load. This creep is due to orientation of the polymer molecules along the tension direction, that results in changes of the tube elastic modulus and thus in the frequency change in time. The time dependence of the frequency is observed again when the filled tube is examined. That dependence is due to the above-mentioned polymer orientation as well as to variations in properties of the material filling the tube. The aim of this work is to get the time dependence of the rigidity modulus for the material filling the tube. It can be obtained by comparing the experimental time dependences for the filled and empty tube.

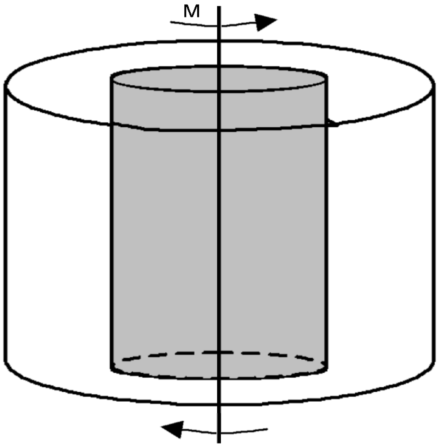

Fig. 1. Diagram of sample loading.

We will be interested in time dependence of the rigidity modulus G. Let G_0 be the G value at the initial time instant t_0, considering G as the rigidity modulus values in subsequent time instants. The pairs of variables G_{10} and G_1, ν_{10} and ν_1, ν_0 and ν will get an analogous interpretation.

Let us introduce the following quantities,

$$\xi = \frac{\nu^2 - \nu_2^2}{\nu_0^2 - \nu_2^2} = \frac{C_1 + C}{C_{1_0} + C_0}, \tag{7}$$

$$\xi_1 = \frac{\nu_1^2 - \nu_2^2}{\nu_{10}^2 - \nu_2^2} = \frac{G_1}{G_{10}}, \tag{8}$$

for filled and empty tube, respectively.

Substituting to formula (17) the corresponding rigidity values, we get

$$\xi = \frac{\frac{\pi(R^4 - r^4)}{2}G_1 + \frac{\pi r^4}{2}G}{\frac{\pi(R^4 - r^4)}{2}G_{10} + \frac{\pi r^4}{2}G_0} \tag{9}$$

Comparing the experimental values of oscillation frequencies ν_0 and ν_{1_0} for empty and filled tubes at initial time moment, we can conclude that $G_0 = 0$. In this case, formula (9) takes the form

$$\xi = \frac{\frac{\pi(R^4 - r^4)}{2}G_1 + \frac{\pi r^2}{2}G}{\frac{\pi(R^4 - r^4)}{2}G_{1_0}} = \frac{G_1}{G_{1_0}} + \frac{r^4}{R^4 - r^4}\frac{G}{G_{1_0}} \tag{10}$$

Comparing (10) and (8), we get

$$\frac{\xi}{\xi_1} = 1 + \frac{r^4}{R^4 - r^4}\frac{G}{G_1}. \tag{11}$$

From the latter expression follows

$$G = \frac{\xi - \xi_1}{\xi_1}\frac{R^4 - r^4}{r^4}G_1. \tag{12}$$

The formula (12) was used to calculate the rigidity modulus of a solidifying material.

The solidification process of gelatin aqueous solution in the course of time was used as an example to approve the proposed method. Numerous works were aimed at investigation of that system (see, e.g., [9, 10]). There are only few works where the shear rigidity of the system has been considered, although only at the initial solidification stage [11, 12].

Fig. 2 shows the experimental time dependence rigidity modulus obtained for 12.2 % aqueous gelatin solution. The tube rigidity modulus was $G_1 = 5 \cdot 10^7$ N/m^2. As is seen from the diagram, the gelatin solution rigidity modulus G varies within the range of $0.04G_1$ to $0.14G_1$. As it can be seen from the error values indicated in the diagram evidence that, in spite of a low value of rigidity modulus G (as compared to G_1), the proposed method permits to single out reliably the solidifying material rigidity modulus out of the total modulus which characterizes the composite cylinder as a whole.

The proposed method has been patented in Ukraine [11].

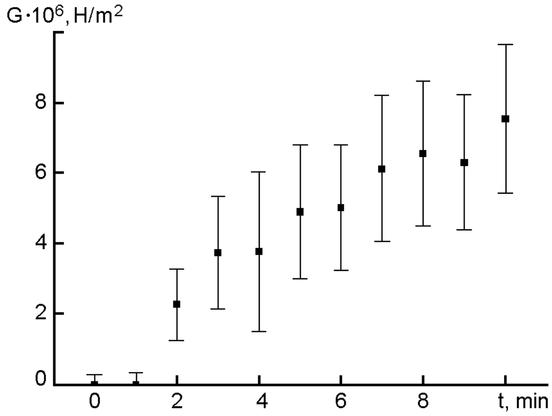

Fig. 2. Gelatin aqueous solution. Time dependence of rigidity modulus for gelatin aqueous solution.

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Метод визначення зсувної пружності матеріалу в процесі його твердіння

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Запропоновано метод дослідження процесів твердіння, в основу якого покладено визначення модуля зсуву матеріалу, що твердіє. Розроблено методику розрахунку цієї величини. Проведено експериментальну апробацію запропонованого методу.