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Peculiarities of using dumbbell specimens made of elastomeric materials subject to finite deformation in complex loading tests

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The features of experiments with dumbbell specimens performed to determine the mechanical properties of filled elastomers are analyzed. Emphasis is placed on testing materials that experience a complex loading history. Experiments such as cyclic loading with increasing amplitude of deformations and with nested stress/strain cycles are considered. It has been established that the rate of change of the stretch ratio in the central part (gauge section) of the specimen significantly differs from that calculated from the analysis of the positions of the grips. This difference manifests itself more prominently in the experiments with complex loading algorithms. The reason is that the viscoelastic properties and the Mullins effect of the filled elastomer are different in the central part of the specimen and at its ends near the grips. The presence of inhomogeneous deformation in the gauge section leads to additional difficulties associated with finding constants for constitutive equations. Besides, this phenomenon complicates a comparison of the mechanical properties of different materials, because detailed information on their behavior can be obtained in complex experiments only. So, experiments with straight specimens or rings with a rectangular cross-section offer more possibilities for studying the mechanical properties of filled elastomers than the experiments with dumbbell specimens.

Keywords: rubber material, dumbbell specimen, inhomogeneous strain rate field, ring-shaped specimen, complex loading history.

1. Introduction

Uniaxial loading experiments, even if they do not imply the study of the strength properties of elastomeric materials, are traditionally carried out on dumbbell-shaped specimens according to ISO 37, ASTM D412 and some others (DIN 53504 etc.) [1–3]. This approach suggests that for the same materials and under the same experimental conditions, the repeatability and the reproducibility of the results can be achieved within the limits of permissible errors. This also provides a more accurate estimate of the properties of materials and makes it easy to determine model parameters. However, in practice, good agreement for repeatability and/or reproducibility is difficult to obtain because of different factors, some of which mentioned in [1–3]. In our opinion, the strain rate must be the same and strictly and permanently controlled in all experiments. However, this is not the case for dumbbell specimens, since the rate of change of the stretch ratio in its central part is different from the strain rate near the grips, and the higher the speed of grips movement the more inhomogeneous the strain rate field along the entire length of the specimen.

It is known that, unlike simple uniaxial tensile tests, during complex cyclic experiments elastomers exhibit a viscoelastic behavior and soften according to the Mullins softening effect [4–11], which is typical of highly filled systems. In this study, the behavior of an elastomeric nanocomposite with high carbon black content was investigated in the experiments with complex loading paths. In the first case, the test program was cyclic loading with nested deformation cycles [5], and in the second, cyclic loading with increasing amplitude of deformations [4, 8, 9]. In experiments with nested cycles, it was found that after prolonged training of the material and at low rate of loading, there are discrepancies in the extension ratio of the specimen areas near the grips and its central part. Thus, the rate of deformation in the gauge section of the specimen is dependent on the viscoelastic behavior of the material. In the tests with increasing tension amplitude, the Mullins softening effect plays a significant role, and, with an increase in strain of the entire specimen, the discrepancy between the extension ratios observed in its central part and in the region near the grips increases significantly.

Analysis of the results obtained shows that dumbbell-shaped specimens should not be used in experiments with a complex loading history. An obvious way out in this situation is to use other forms of specimens, such as straight specimens or rings with a rectangular cross-section. Straight specimens are less preferred due to strong coming of the material out of grips and the possibility of their tearing there at high level of deformation. Tests on ring-shaped specimens make it possible to achieve uniform strain rates over the entire length of the specimen with high accuracy, but they should only be used in cases where the material is initially isotropic.

2. Mechanical properties of specimens in experiments with a constant rate of grips separation

In order to model the structures produced from viscoelastic materials, it is necessary to use constitutive equations able to take account of mechanical behavior of these materials. Experiments are required to select a viscoelastic model and find the constants included in it. It is also necessary to compare the experimental data obtained for different materials and construct convenient algorithms for finding the constants in the constitutive equations at prescribed deformation rate. All rubber testing machines provide necessary modes of grip movement. But with dumbbell specimens, problems arise when analyzing the mechanical behavior of rubbers. They are associated with the fact that there is a significant increase in the width of the specimen in front of the grips. In these areas, non-uniform stress fields appear, and they differ significantly from the stresses in the gauge section. This leads to a nonlinear rate of deformation of the central part of the specimen under conditions when grips move at a constant speed.

There is also a phenomenon that complicates the analysis of the mechanical properties of filled elastomers. The viscoelastic properties of a material appear in different ways at different rates of deformation. And this is especially pronounced at large strains. The material softening process (Mullins effect) also takes place in filled elastomers. The Mullins effect manifests itself much brighter in the central part of the specimen than near the grips. Moreover, the degree of material softening depends on the rubber compound. Therefore, the features of deformation of the specimen gauge length cannot be foreseen in advance. But softening of the material can lead to a noticeable change in the stress-strain state in the manufactured products, as shown in the article [10]. Modern equipment makes it possible to obtain information on the extension ratio of the specimen gauge length. One can track the positions of the reference marks using video or contact extensometers. But it is difficult to ensure the required rate of deformation of the central part by controlling the displacement of the grips in the tensile testing machine.

Let us consider how the stretch ratio of the specimen changes in simple tests and under complex loading conditions. In what follows, we estimate the stretch ratios λ_g, determined by viewing the positions of the grips, and λ, by tracking two marks of the specimen gauge section, as the ratio of distances:

$$\lambda_g = \frac{L_g}{L_{g0}},\ \lambda = \frac{l}{l_0},$$

where l and l_0 is the distance between the reference marks on the specimen gauge section at current and reference instants of time, and L_g and L_{g0} is the distance between the grips at current and reference instants of time. The experiments described in this paper were carried out on the dumbbell specimens manufactured according to ISO 37 Type 2 standard (Fig. 1a) and on the rings with rectangular cross-section (Fig. 1b). Note that the result of investigations with ring-shaped specimens is given at the end of the article. In the material under study, SBR-1500 elastomer was used as a matrix and carbon black N220 as a filler in amount of 50 phr. The deformation of the material in the specimen gauge section was monitored by tracking two marks with a videoXtens video extensometer.

The results of the experiments, in which the grips travel with a constant speed, are shown in Fig. 2 and Fig. 3. The dependence of the stretch ratios λ/λ_g shown in Fig. 2 was determined at a constant rate found by analyzing the positions of the grips $d\lambda_g/dt = 0.5$ min^{-1}. The difference in the strain rate of the gauge length from the constant test speed is the higher the greater the displacement rate of the grips taken for the experiment (Fig. 3). It is seen that, even in a simple tensile test, the dependence of the parameters (λ/λ_g and $d\lambda_g/dt$) on λ is nonlinear. In this case, nonlinearity is difficult to predict in advance, since both the viscoelastic properties of material and the softening effect depend on the specific composition of the material. More problems arise when the mechanical properties of rubbers are studied in complex deformation modes.

3. Mechanical properties of specimens in the experiments with nested deformation cycles

Simple experiments with polymers give little information on its mechanical properties. Based on the results of these tests, researchers can obtain only a curve that represents the dependence of stress on the extension ratio at a prescribed rate of grips separation, or only a stress relaxation curve

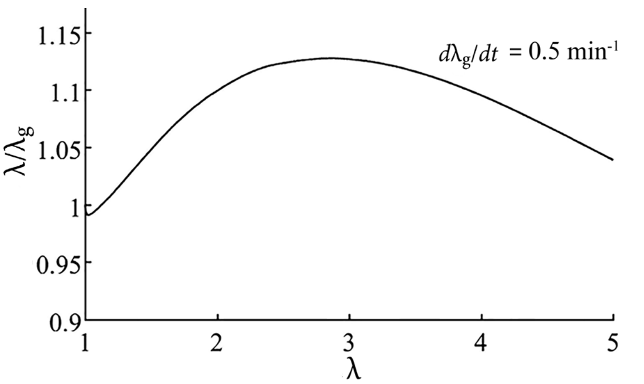

Fig. 2. Relationship between the stretch ratio λ of the gauge section and the stretch ratio λ_g determined by viewing the positions of the grips.

Fig. 1. Specimens used for studying the viscoelastic behavior of rubbers: a dumbbell-shaped specimen (a), a ring-shaped specimen mounted on two lubricated cylindrical rods and passing through the guides (b). The gauge lengths of the specimens are intervals of length l; L_g is the distance between the grips.

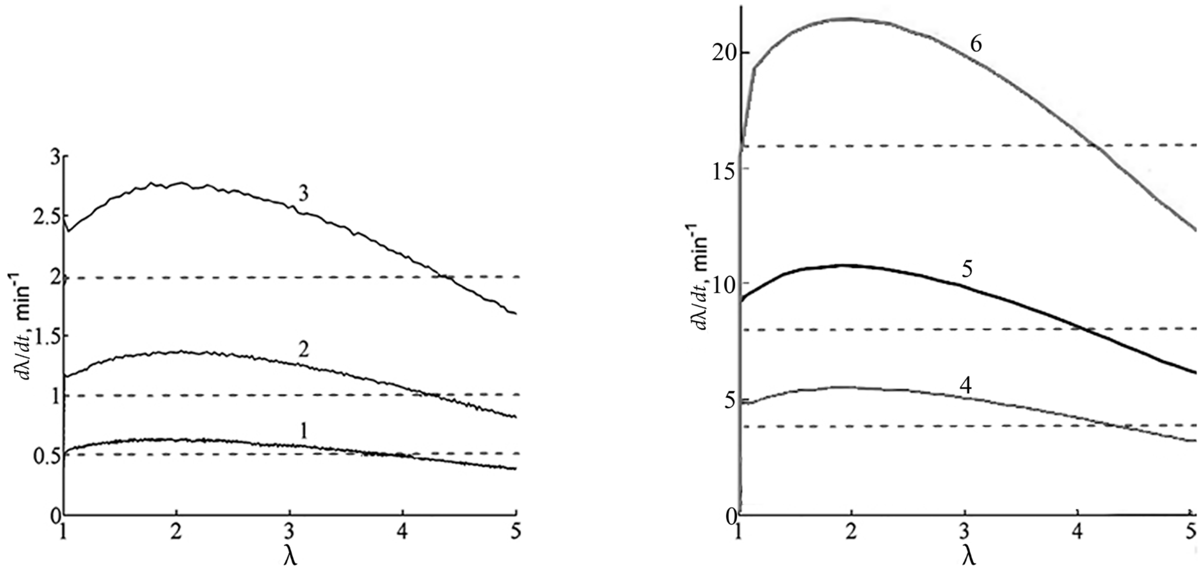

Fig. 3. Strain rate $d\lambda/dt$ of the gauge section at different λ. Dashed lines represent the rate of grips separation: 1 — $d\lambda_g/dt = 0.5$ min^{-1}, 2 — $d\lambda_g/dt = 1$ min^{-1}, 3 — $d\lambda_g/dt = 2$ min^{-1}, 4 — $d\lambda_g/dt = 4$ min^{-1}, 5 — $d\lambda_g/dt = 8$ min^{-1}, 6 — $d\lambda_g/dt = 16$ min^{-1}.

at a given elongation; it is also possible to obtain a creep curve in a separate experiment. Testing under complex strain history provides much more data. In order to obtain such characteristics as material softening, hysteresis losses, and residual deformation, the following cyclic loading modes are usually applied: multiple cyclic loading [6,7,11], cyclic tension with increasing amplitude [4,8,9] and with increasing amplitude, including material compression [12]. Multi-step relaxation tests during loading and unloading of specimens [8–10] are also of interest as a way to determine the material behavior excluding time-dependent processes.

In our recent paper [5], we have proposed an algorithm for performing the experiment in which a material specimen undergoes deformation with nested cycles and a time delay at each change in loading direction (see Table 1 and Fig. 4). The advantages of using this algorithm are as follows:

1. There is no need to carry out multiple different experiments on different specimens to get data for analyzing the features of the mechanical behavior of the material. Large quantity of information can be obtained immediately after examining only one specimen. This enables savings in experimental time and savings in the material used to manufacture specimens.

2. It is easy to identify the losses caused by material softening (Mullins effect) by comparing the first and second cycles of specimen deformation. Analysis of the viscoelastic behavior of the softened material is performed in the second and subsequent cycles.

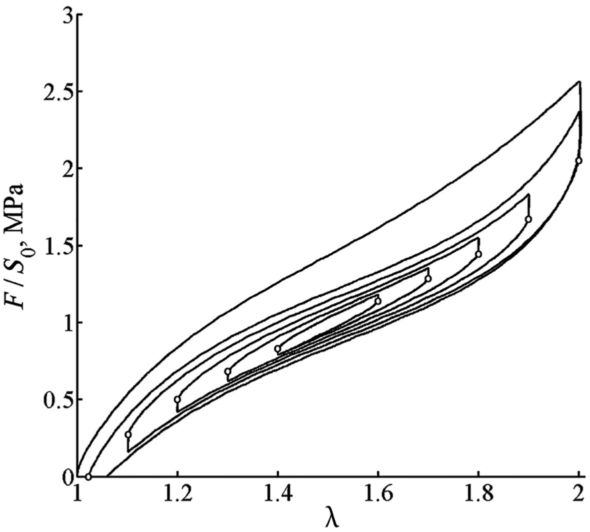

Fig. 4. Plot of force vs. stretch ratio calculated by tracking reference marks in the experiment with nested deformation cycles. Small circles on the curve indicate the points that represent the equilibrium state of the material.

3. The nested stress/strain cycles, starting from the second one, demonstrate the peculiarities of the deformation curves of the specimen for different displacement rates of the grips, which remain unchanged until the moment when the mode of grips movement changes.

4. The experiment provides data on stress relaxation at different stretch ratios of the softened material, observed at time intervals when the grips remain stationary.

Table 1. Algorithm for material specimen deformation in uniaxial test with nested cycles.

Cycle number	Specimen loading rate $d\lambda_g/dt$, min^{-1}	Maximum stretch ratio per cycle λ	Time delay, min	Specimen unloading rate $d\lambda_g/dt$, min^{-1}	Minimum stretch ratio per cycle λ	Time delay, min
1	1	2	20	1	Primary state	20
2	1	2	10	0.5	1.1	10
3	0.5	1.9	10	0.25	1.2	10
4	0.25	1.8	10	0.125	1.3	10
5	0.125	1.7	10	0.06	1.4	10
6	0.06	1.6	10	0.06	1.4	

5. If the intervals when the grips remain stationary are long enough, then at the final section of stress relaxation the equilibrium state of the material can be fixed. By connecting the obtained equilibrium points on the graph with the approximation curve, an equilibrium stress-strain curve is plotted, as if the material is slowly loaded. Unfortunately, it is unreasonable to stop moving of the grips over a long period because this will increase the time of the experiment significantly. Therefore, the grip stops usually do not exceed 10 minutes. This yields the approximate values of stresses and strains for filled elastomer in an equilibrium state. The accuracy can be improved when the stress relaxation time in all states with stationary grips increases to 20 or 30 minutes.

6. A comparison of the states of the material after the period of its rest at the beginning of the cycles gives information about the magnitude of residual deformations or indicates their absence. Certainly, the period of rest should be long enough, but even at a 10-minutes rest period there is a chance to know whether residual strains are negligibly small, or they need to be studied in more detail. This result could be an indication of irreversible changes or incomplete creep processes in the material.

An example of using the above algorithm for conducting the experiment with nested deformation cycles is shown in Fig. 4. Throughout the paper, the following notation is used: F is the force acting on the specimen, and S_0 indicates the initial cross-section of the specimen gauge section. The plot shows the loading (stretching) and unloading areas of the specimen at a constant displacement rate of the grips. At the stretch ratio $\lambda = 2$, the grips stopped moving and a 20-minute delay was set. After unloading, a 20-minute delay was set for the initial deformation cycle. This ensured accurate identification of equilibrium points at irreversible losses that simultaneously occurred in the material due to its viscoelastic nature and softening. Besides, this made it possible to learn whether the material is able to restore its initial state or whether residual strains may occur in it. For other cycles, a 10-minute delay was set. Since the vertical sections (curves of stress relaxation) are of ordinary shape, so we dropped them from the detailed consideration. However, these sections should be considered in order to verify the accuracy of the chosen mathematical model and to assess the correctness of model parameters. The circles indicate the points at which the state of the material can be approximately considered an equilibrium state. The quantitative features of the algorithm proposed are presented in Table 1.

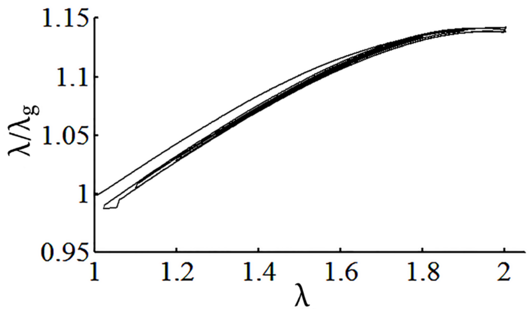

Fig. 6. Relationship between the stretch ratio values determined by tracking reference marks and by analyzing the positions of grips at different λ in the experiment with nested deformation cycles.

The use of the algorithm for studying the mechanical behavior of a filled elastomer in the experiment with nested stress/strain cycles is shown in Fig. 5. Analysis of the graphs presented in Fig. 5a and b indicates that the strain rate determined within the gauge section of the specimen differ from that obtained near the grips. This is especially pronounced during the first cycle, when the viscoelastic behavior of the material is accompanied by a softening effect; in other cycles, this difference becomes less significant. This statement is supported by the relationship between the stretch ratios given in Fig. 6. These differences should be taken into account when searching for constants in the constitutive equations of the medium. To investigate the influence of the Mullins effect at different material stretch ratios, it is necessary to carry out a separate experiment that will be discussed below in this article.

4. Mechanical properties of specimens in the experiments with increasing amplitude of deformations

The experiment with increasing deformation cycles provides important information on the effect of material softening on the stretch ratio of the material in the specimen gauge section. In contrast to the experiment with nested stress/strain cycles, this test enables getting data on the mechanical behavior of specimens in the case when the softening degree increases at each cycle. Figure 7 shows the plot of force versus stretch ratio determined by analyzing the positions of the grips. At each

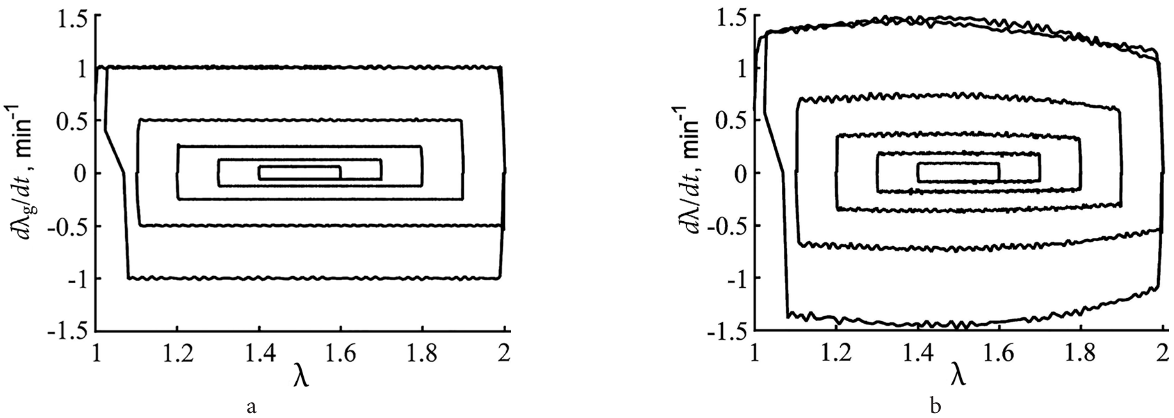

Fig. 5. Rates of change of the material stretch ratio in the different specimen areas subject to deformation at a constant rate of grips travel. The stretch ratios are determined by viewing the positions of the grips (a) and by tracking the reference marks (b) of the specimen.

new cycle, the maximum stretch ratio increased by $\Delta\lambda = 0.5$. Each time, the maximum extension ratio was achieved in the cycle, and the grips stopped moving for 10 minutes. This made it possible to observe stress relaxation at the corresponding extension ratio λ. After unloading, the grips stopped moving for 10 minutes at each cycle so that we could observe structure recovery and got values of residual strains. The rate of change of the material stretch ratio induced by the grips was $d\lambda_g / dt = 0.25$ min^{-1} under stretching conditions, and $d\lambda_g / dt = -0.25$ min^{-1} under unloading conditions.

The results of the experiment are shown in Fig. 8. Based on these data, we can conclude that at large deformations there is a significant difference in the experimental results for the same material. A noticeable increase in the strain rate $d\lambda / dt$ at the end of the cycle at large stretch ratios λ is an unexpected event. This can be attributed to the fact that at the end of the cycle one can observe additional material softening which develops differently in the central part of the specimen and in the specimen area near the grips. The same complex pattern arises when we analyze the relationship between the stretch ratios determined by tracking the marks and those obtained near the grips (Fig. 9). The significant difference between the stretch ratios λ and λ_g suggests that it is more effective to perform the above mentioned experiment with specimens of different shape, e.g., ring-shaped specimens, rather than with dumbbell specimens.

5. Mechanical properties of the material in the experiments carried out on ring-shaped specimens

In the experiment, we used ring-shaped specimens, the fastening scheme of which is given in Fig. 1b. The rings are cut from a 2 mm thick plate and mounted on the rods in the form of round cylinders 5 mm in diameter. These rods are the grips for stretching specimens and they are lubricated with silicone grease to facilitate slippage. In the initial cycle, the ring with a rectangular cross-section was straightened so that its behavior corresponds to the behavior of straight specimens under uniaxial loading. To reduce the size of the area where the rings were straightened, we used guides through which the specimen passed practically without friction, but this limited their ability to take a round shape (Fig. 1b). An example of stretching a ring-shaped specimen at $d\lambda_g / dt = 1$ min^{-1} is shown in Fig. 10. As expected, the stretch rate of the material, determined by tracking marks turned out to be the same as that caused by the grips. This happens at any algorithm predicting specimen deformations.

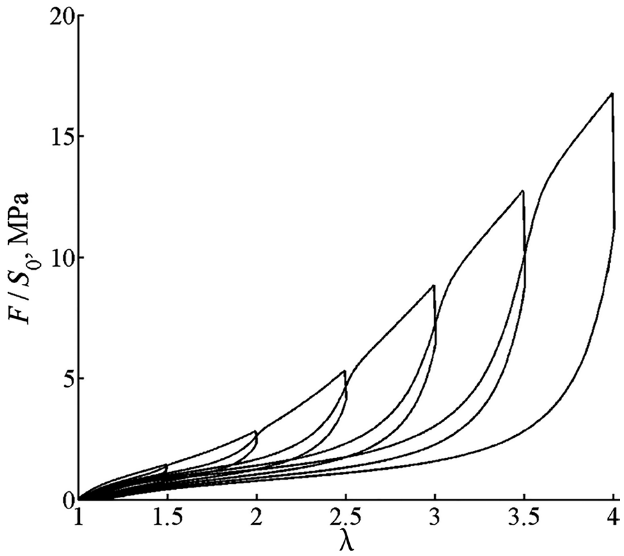

Fig. 7. Cyclic stretching of the specimen in the experiments with increasing amplitude and with time delays at maximum and minimum stretch ratio values.

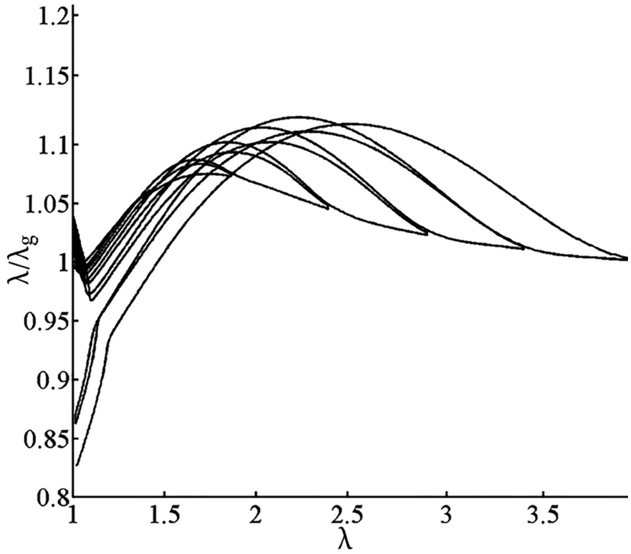

Fig. 9. Relationship between the stretch ratio values determined by tracking reference marks and by analyzing the positions of grips at different λ in the experiment with increasing amplitude.

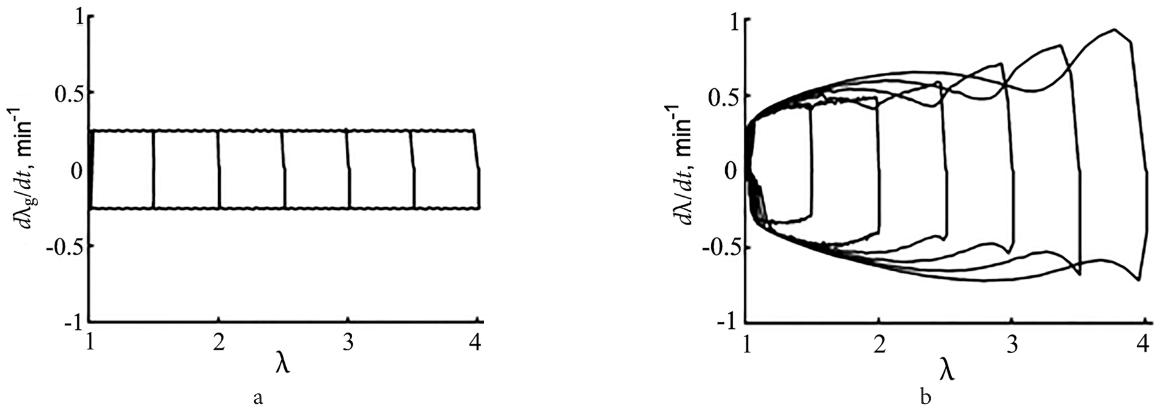

Fig. 8. Rates of change of the material stretch ratios at constant rate of grips displacement. The stretch ratios are determined by viewing the positions of the grips (a) and by tracking reference marks (b) of the specimen.

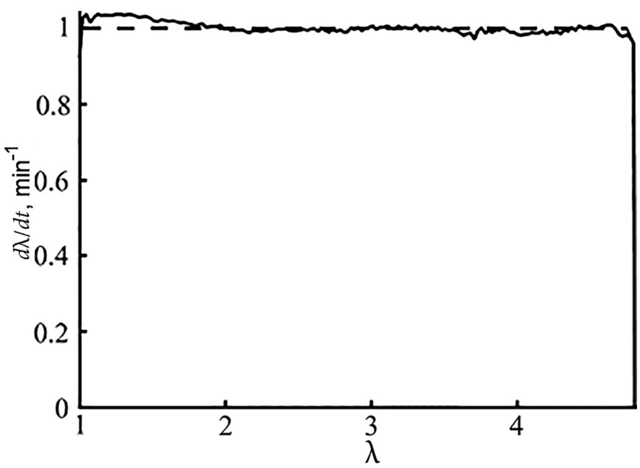

Fig. 10. Rates of change of the stretch ratios determined by tracking marks (solid line) and by analyzing the positions of the grips (dashed line) in the experiment with ring-shaped specimens.

6. Conclusions

We have investigated the specific features of the experiments with nested stress/strain cycles conducted to analyze the properties of the material under finite deformations. Each time the modes of movement of the grips used in these tests changed, a long-time delay was set. Such an approach enabled us to obtain, within the framework of only one experiment, information about the viscoelastic behavior of the material in a wide range of strain rates, as well as data on the softening effect, residual strains, and the equilibrium points of the material. Additional information was obtained in the experiments with increasing strain amplitude and longtime delays. However, the presence of inhomogeneous deformation in the gauge section of dumbbell specimens led to additional difficulties associated with finding constants for constitutive equations; difficulties also arose in comparing the mechanical properties of different materials.

Analysis of the results of the experiments has revealed a very strong difference between the strain rates in the central part of the specimen and those determined by analyzing the positions of the grips. The strain rate of the gauge length of the dumbbell specimen is not a constant value at a prescribed rate of grips displacement. The more complex is the deformation mode used to analyze the mechanical behavior of the material, the stronger is the difference. This can be attributed to a significant difference between the viscoelastic processes and the softening effect of elastomers. The behavior of the elastomers in the region near the grips differs significantly from the material behavior in the gauge section of the specimen, and this difference increases with increasing deformation. A remedy to this situation is to use specimens of different shape, e. g., ring-shaped specimens, rather than dumbbell specimens.

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