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## Spectra of Creep Rates of Small Inelastic Deformations in Solids

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The events of laser interferometric measurements of creep rates associated with small deformations of solids are reviewed to illustrate the potential of this method to characterize the mechanical responses of solids under stress.

**KEY WORDS** Creep rates, laser interferometry, transitions in polymers, mechanical properties

### INTRODUCTION

The laser-interferometer method allows us to record  $\sim 10^{-9}$  m/s rates of creep in experiments involving deformation increments of a few thousandths of a percent.<sup>1,2</sup> Qualitatively new results were obtained in various fields of research related to creep phenomena. Mention should first be made of the results of studies of dependence of the creep rate of polymers  $\dot{\epsilon}$  on their deformation ( $\dot{\epsilon} = f(\epsilon)T, \sigma$ ), as well as of the discovery of regularities of changes in the activation energy  $Q_0$  and the activation volume  $\alpha$  versus the creep deformation  $\epsilon$  and the temperature  $T((Q_0, \alpha) = f(\epsilon, T))$ .<sup>3,4,8</sup>

Recently the above-mentioned precision method was widely applied in studies of the rates of creep associated with the initial inelastic deformation stage of such materials as polymers, metals and ceramic materials.<sup>5,6,7</sup> At a certain initial temperature the rate was measured at a definite moment after the loading at  $\sigma < 0, 1\sigma_c$ , where  $\sigma_c$  is the yield or fracture stress. Next the specimen was relieved and  $\dot{\epsilon}$  measured under identical conditions at a temperature 5 to 10°C higher. The procedure repeated over a chosen temperature range of study yields a set of creep rate values called a "spectrum of creep rates," which differs for each particular material.

Although each group of tested materials has its special characteristics, analysis of available data makes it possible to distinguish a phenomenon common to all these materials: the temperature dependences of the rate of small inelastic creep deformations in the solids are spectra whose particular points correspond to regions of thermal activation of definite deformation mechanisms.

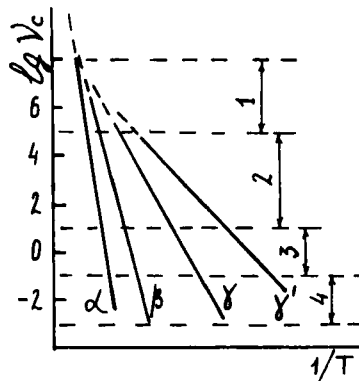


FIGURE 1 Simplified diagram of frequency-temperature dependences of glass polymers ( $\nu_c = 1/2\pi t$ ). Horizontal dashes denote frequency ranges of applicability of resonance methods (1-3 from Staverman<sup>6</sup>). Wave methods = 1; forced oscillations = 2; relaxation, conventional creep = 3; and spectra of creep rates = 4.

Let us consider some interesting data which were obtained for polymers, metals, and ceramics. In one way or another, all solids dissipate the energy coming from the outside when the temperature or the frequency is changed. Relaxation processes are most strongly exhibited in polymers, reflected in the fact that the group of polymers yielded abundant evidence on correlation of  $\alpha$ ,  $\beta$ ,  $\gamma$  and other relaxation transitions with the temperature dependences of the dynamic modulus, strength, deformation capability and other characteristics of materials.<sup>8</sup> In forecasting polymer properties, it is thus important to determine the transition temperatures, which explains the large variety of methods of transition temperature measurement. However, the abundance of methods sometimes produced ambiguous data sets. While the  $\alpha$ -transition temperatures, measured by different methods, agree satisfactorily with the corresponding branch of the temperature-frequency dependence of transitions (Figure 1), because of a scatter of points it is sometimes only with great imagination that the data for the  $\beta$ ,  $\gamma$ , and  $\gamma'$  transitions can be grouped in one branch, i.e. be referred to the same type of movement, even with an identical affecting frequency.<sup>8</sup> Consequently, in a majority of cases these transitions cannot be interpreted unambiguously, which can result in substantial errors in frequency-extrapolation, comparison and forecast of transition temperatures.

In this study the example of polycarbonate is considered to illustrate that one of the reasons for ambiguous results of transition temperature measurements made by different methods may be a heterogeneous structure of wide relaxation spectral regions, whose components react differently to the same effect.

## EXPERIMENTAL

For the polymers and metals, the specimens used were cylinders 6 mm high and 2-3 mm in diameter. The ceramic specimens were parallelepipeds measuring about  $3 \times 3 \times 8$  mm. The measurement of the rate of creep always started 10 . . . 100

s after the start of the loading. The rate of creep measurement proper lasted approximately the same time.

## RESULTS AND DISCUSSION

Polycarbonate (PC) was chosen as a material whose internal friction spectrum (IFS) exhibits a pronounced secondary  $\beta$ -transition with a wide dispersion region and a maximum near  $-100^\circ\text{C}$  (Figure 2).<sup>9</sup> The low-temperature relaxation region, which occurs in the spectrum of creep rates (SR) of the PC (see Figure 3), belongs to the same temperature interval which is characteristic of the IFS, but has a more complex structure. Curves I and II in Figure 3 show data for the same specimen at different times after loading. The SR display four maxima, rather than one, with the peaks on curves I and II differing in height considerably (for example, a longer time corresponds to a relatively larger contribution of the second peak). With longer times there is a noticeable shift of the first peak to the left on the temperature scale, while the peaks 2-4 kept their place within the limits of experimental error.

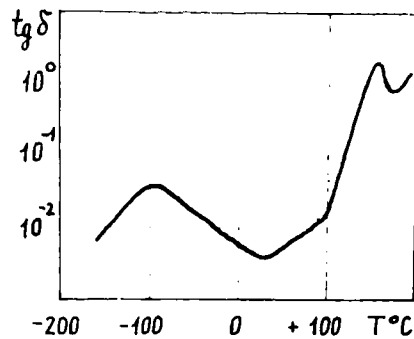


FIGURE 2 Tangent of mechanical loss angle of PC ( $\text{tg}\delta$ ) versus temperature.<sup>9</sup>

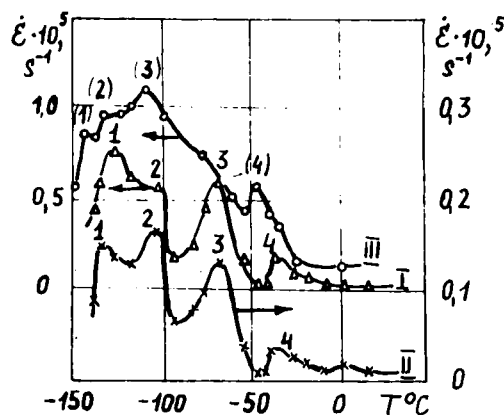


FIGURE 3 Polycarbonate. Spectrum of creep rates (15 s = (1), (3) and 150 s = (2)) after 31 MPa loading; (3) = specimen exposed to  $-196^\circ\text{C}$ – $+20^\circ\text{C}$  thermocycling.

This finding means that the displacement of the common maximum of IFS with a change of the frequency may result from a displacement of only a few components of the spectrum, rather than displacement of the entire region as is commonly assumed.

In order to check the effect of a preliminary thermal treatment of the PC on SR, experiments were made with specimens exposed to 10 cycles of temperature reversal from  $-196^{\circ}\text{C}$  to  $+20^{\circ}\text{C}$ . In Figure 3 four maxima are exhibited both by curve III (the specimen after the thermocycling) and curve I (the initial material), with the third one the most pronounced. Here lies a potential for error in the interpretation of the response of the polymer to the thermal treatment. If the ( $-150-0^{\circ}\text{C}$ ) relaxation region is determined by the maximum peak, then the thermal treatment appears to displace the spectrum towards higher temperatures (compare curves I and III). If, however, the complex structure of the relaxation region is taken into account and the maxima are correlated, the conclusions must be different: peak (3) shifted by  $50^{\circ}$  about peak 3 to lower temperatures, and peak (2) by  $40^{\circ}$  about peak 2, while peaks (1) and (4) exhibited the smallest displacement.

Thus relaxation regions of polymers may have complex structure, and their components may react differently to the same effect. The position of maxima is affected most by a preliminary thermal treatment, and in particular by a thermal cycling. Spectrum components may be affected in different ways by the time (or frequency) of the exposure. Hence there is a great probability that even at identical frequencies, different procedures of relaxation transition calculations selectively affect different components of the wide relaxation regions. This may result in different temperatures of the maxima and hamper correlation and forecasting of transitions.

The correlation of the maxima on SR with temperatures of relaxation transitions produced by changes in the molecular mobility was observed not only for PC, but for all the polymers investigated (PMMA, PVB, PS, epoxies etc.).<sup>10-12</sup>

It follows from the above correlation that SR temperature maxima correspond to the temperatures at which changes in macro-deformation and rupture strength take place.<sup>5,12</sup> In other words, it is possible to use non-destructive test data (spectrum of creep rates) to forecast the character of changes in the strain and strength properties of polymers at fracture.

Analysis of the findings obtained by the SR method and the established data available on temperatures of brittle-ductile transitions (Tbdt) showed a certain correlation between the data sets. Tbdt is known to depend on specific conditions of the tests, and thus changed under conditions of tension, torsion and compression (see Figure 4). It was proved for both polymers and metals that for each kind of test, Tbdt, similar to the temperature at a sharp increase of the residual deformation, is near one of the SR peaks without any intermediate values (see Figure 4).<sup>13</sup> Thus SR permits forecasts of Tbdt at different stress states, making it possible to develop a more accurate and less time-consuming method of estimation of brittle destruction tendency for steels.<sup>14</sup>

Local deformations in general and those of brittle solids in particular are a matter of great importance, because it is precisely the local shears that substantially contribute to internal friction under loading and influence the crack formation mech-

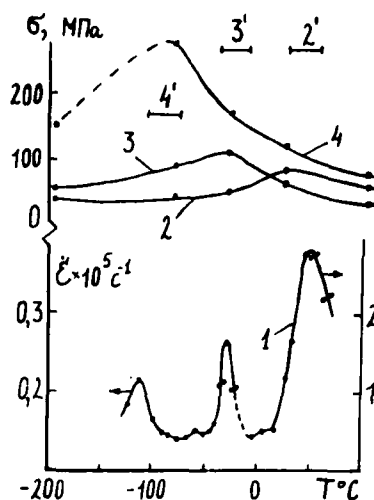


FIGURE 4 Temperature dependences of strain rates at 50 MPa (1), fracture tensile stresses (2), fracture torsional stresses (3), compression flow stresses (rupture at  $-196^{\circ}\text{C}$ – $-80^{\circ}\text{C}$ ) of polycrystal zinc (4). Regions of brittle-ductile transition: tension = 2; torsion = 3; and compression = 4.

anism and the strength. Observation and investigation of the kinetics of small deformations in brittle bodies, such as ceramics, is a complicated experimental problem. Nevertheless SR have been obtained for such brittle materials as glass ceramics, ultraprocelain, and Y-Ba-Cu-O superconducting ceramics.<sup>7,15</sup> It was ascertained that qualitative regularities of deformation kinetics of ceramic materials have much in common with similar creep characteristics of other materials.

## CONCLUSION

The determination of relaxation transitions by means of the spectrum-of-creep-rates method represents the closest approximation to actual loadings. This method is the highest-resolution low-frequency (0.1–0.001 Hz; see Figure 1) procedure of relaxation spectrometry available. This provides the basis for affirming the conclusions that were made from the data obtained by means of the SR method.

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