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Deformation recovery behaviour of a solid polymer after tensile yielding

II. Effect of temperature

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SUMMARY

In this work the deformation recovery behavior of an amorphous polymer after large tensile deformation is studied at different temperatures. The effect of three parameters other than temperature has been pointed out in previous works (1-3).

The data here presented indicate that the influence of all parameters, including temperature, on recovery behavior can be related to the residual stress, σ^*, at the beginning of recovery test. This suggests to plot the recovery versus time curves, relative to different sets of parameters including temperature, by means of the same normalizing groups which were adopted in (2), thus shifting all curves towards a single master curve.

INTRODUCTION

This experimental work is the continuation of a previous one (2), where the deformation recovery after yielding of an amorphous polymer was studied at room temperature. The interest of such a study is related to the possibility of solid state processing of polymers as an alternative to the more usual melt processing (4-14).

In (2) three parameters were analyzed, which affect the recovery behavior of the material: i) the sample deformation ratio,e, just before unloading; ii) the initial deformation rate, α_i , during sample loading; iii) the time t^* the sample is kept at the deformation ratio e before unloading. A normalizing procedure was adopted which allowed to shift curves relative to different sets of

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these parameters towards a single master curve.

A "recovery master curve" of the material could be used for predicting the amount of recovered deformation for any set of parameters without performing further experimental tests. This possibility is obviously interesting for solid state processing of polymers. Furthermore, as solid state processing is generally performed at temperatures higher than room temperature, in this work the effect of this parameter was pointed out. The results confirm the effectiveness of the normalizing procedure adopted in (2) even for tests performed at different temperatures.

EXPERIMENTAL

The material used was Lexan, a bisphenol A polycarbonate (4-4' dioxydiphenil-2,2propane carbonate) manufactured by General Electric, supplied in pellet form. The samples for tensile tests were prepared in a proper press, keeping the polymer at 190°C under vacuum for 5 hours. This allowed to superate the unusual molding procedure followed in (2) in order to gradually eliminate the air which remained included in the sheets. A subsequent rapid cooling was obtained by running cold water on the press.

In order to avoid necking and thus to obtain an homogeneous deformation of samples after tensile yielding, the sheets were rolled at room temperature. A deformation ratio $e_r=l'/l_o$ of 1.24 in the drawing direction was induced, where l' is the ultimate value of sheet length in the rolling direction and l_o is its initial value. The deformation ratio in the direction perpendicular to the rolling direction on the sheet plane was between 1.03 and 1.04.

Strip samples were cut from the preoriented sheets, 10 cm long in the rolling direction and 1 cm wide. The initial distance between grips was 4 cm .

The deformation recovery tests after sample unloading were made at room temperature, at 50°C and 100°C by an Instron machine model 1115 with a transducer connected to the machine cross head, following the procedure described in (2). The transducer-bridge-recorder system allowed measuring displacements of the order of magnitude of 10^{-2} mm. Some preliminary tests showed data reproducibility within 5%.

The main parameter here investigated was the temperature T during the recovery test. For each value of temperature different sets of the following parameters were selected:

- e, the deformation ratio just before the recovery test with respect to the length of the virgin (before both rolling and drawing) sample;

- α_i , the initial deformation rate during sample loading, the ratio between the cross-head velocity and the length of the rolled sample;
- t^*, the time the sample is kept at the deformation ratio e before unloading.

The values of parameters which were tested are:

T	25 , 50 , 100 °C
e	1.4 ; 1.6 ; 1.8
α_i	3 , 30 h^{-1}
t^*	60 , 10^3 , 10^4 , $1.6x10^5$ sec.

RESULTS AND DISCUSSION

The effect of temperature on stress strain curves relative to tensile tests performed up to e=1.8 is shown in fig.1. As expected, for each value of deformation ratio, e , the higher the temperature the lower the stress.

The stress relaxation behavior of the material is shown in fig.2. It can be observed that, for each value of temperature, data are affected by parameters e, α_i and t^* in the same way as reported in (1), i.e. the residual stress σ^* decreases on decreasing e and on increasing both α_i and the relaxation period t^*. As for the effect of temperature, for each set of other parameters, the higher the temperature the lower the residual stress σ^*.

As for recovery tests (2), they were performed by loading the sample with a prefixed deformation rate α_i up to a fixed value of deformation ratio e (corresponding to point A in fig.3), letting the material relax from the stress σ^* for a fixed period t^* (A-B) and finally unloading it and measuring the recovered deformation versus time. The sample was not unloaded instantaneously (B-C) but by means of the load control system of the Instron machine, which allowed the sample to reach and keep a fixed small value of the load (B-D). This needed a time, t_r , which was dependent on the cross-head velocity.

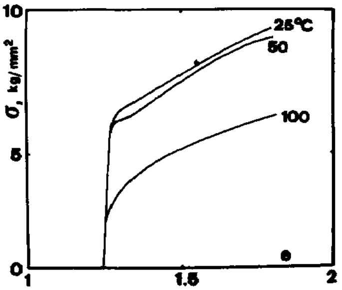

FIG.1 Tensile stress, σ , versus deformation ratio, e , at different test temperatures.

At the end of the time t_r a first recovery Δl_i was measured. After this first recovery step, recovery versus time curves were obtained by means of the load control system and the transducer-bridge-recorder assembly.

In fig.s 4-7 recovery data are plotted as $\Delta l/l$ versus time t, were $\Delta l/l$ is the recovered deformation with respect to the sample length l after tensile deformation and t is measured from point B (see fig.3) just after stress relaxation period.

For each tested temperature, the effect of the parameters e, α_i and t* was the same as observed in (2,3). In particular: i)the larger the deformation ratio the larger the recovery; ii)a larger deformation rate results in a smaller recovered deformation; iii)the larger the relaxation period the smaller the recovered deformation.

As for the effect of temperature, for each set of other para-

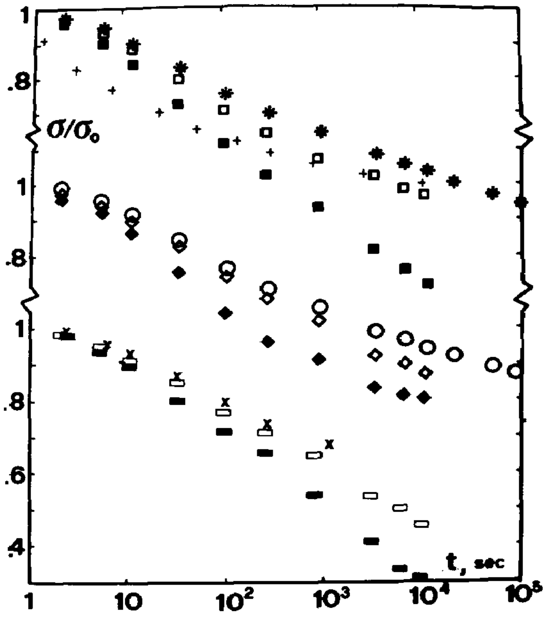

FIG.2 Dimensionless stress versus time for stress relaxation tests. Data reported by symbol + are relative to α_i=30 h^{-1}, all other tests are performed at α_i=3 h^{-1} .

T=20°C	+✱ e=1.4 ;	○ e=1.6 ;	x e=1.8
T=50°C	□ e=1.4 ;	◇ e=1.6 ;	▭ e=1.8
T=100°C	■ e=1.4 ;	◆ e=1.6 ;	▬ e=1.8

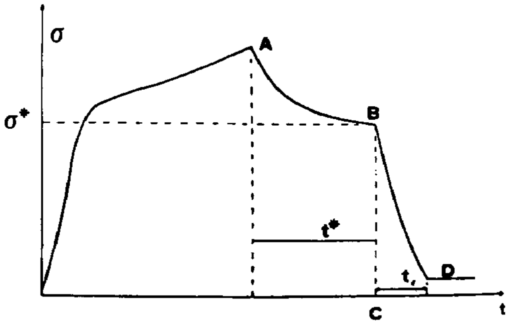

FIG.3 Typical stress versus time curve during a recovery tests.

meters the recovered deformation decreases on increasing the test temperature.

Comparing the comments on fig.2 with these comments on fig.s 4-7, it can be concluded that temperature affects the amount of recovered deformation by means of σ^*, the residual stress at the end of the relaxation period t^*. This suggests a plot of the recovery data as $\Delta l/(l\cdot\sigma^*)$ versus t (see fig.8).

On the other hand, the curves in fig.8 are grouped into sub-bands characterized by different shapes, according to the value of t^*. The use of the parameter t^* as a normalizing factor of abscissa, see fig.9, again improves the superposition of recovery curves, which now lie into a narrow S-shaped band.

CONCLUDING REMARKS

The deformation recovery behavior of viscoelastic materials is certainly difficult to be described by models, because of the high number of parameters which affect it.

In the study, which this experimental work is part of, four parameters have been pointed out which produce significant effects on the amount of deformation recovered by an amorphous polymeric material.

These effects seem to be associated with the residual stress present in the material when it is unloaded, e.i. at the beginning of the recovery test. This idea is supported by the results of the normalizing procedure here adopted. In fact, using the residual stress as normalizing factor of the recovered deformation and plotting the recovery data on a consistent time scale, curves which were initially very different both in shape and in ordinate range draw up in a relatively narrow S-shaped band.

Although this result is not good enough to be used as a "deformation master curve" of the material, it still indicate that the

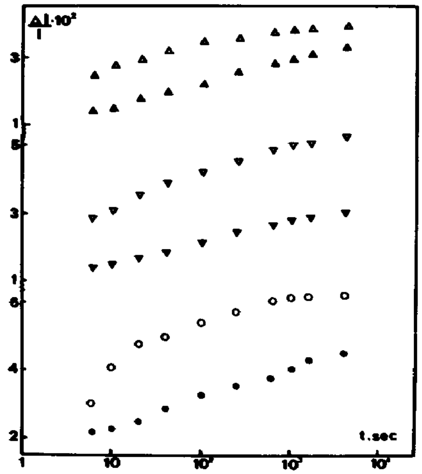

FIG.4 Recovered deformation versus time after tensile deformation at α_i=3 h^{-1} followed by a relaxation period t*=60 sec.

	T=50°C	T=100°C
e=1.4	△	▲
e=1.6	▽	▼
e=1.8	○	●

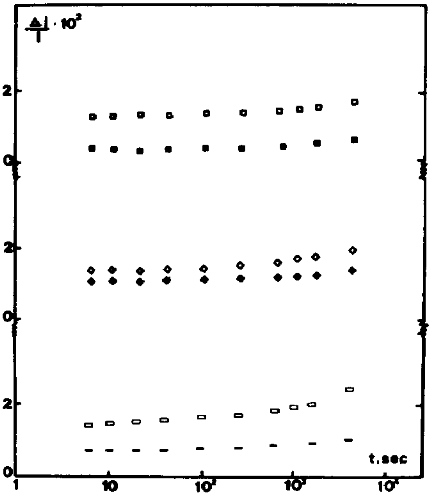

FIG.5 Recovered deformation versus time after tensile deformation at α_i=3 h^{-1} followed by a relaxation period t*=10^4 sec.

	T=50°C	T=100°C
e=1.4	□	■
e=1.6	◇	◆
e=1.8	▭	▬

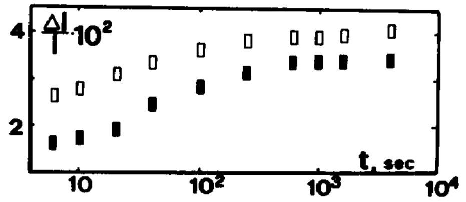

FIG.6 Recovered deformation versus time for tests performed at α_i=30 h^{-1}, e=1.4 , t*=60 sec.

▯ T=50°C ; ▮ T=100°C

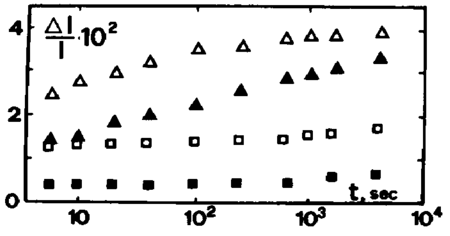

FIG.7 Recovered deformation versus time for tests performed at α_i=3 h^{-1} and e=1.4

T=50°C	△ t*=60	□ t*=10^{4}sec
T=100°C	▲ t*=60	■ t*=10^{4}sec.

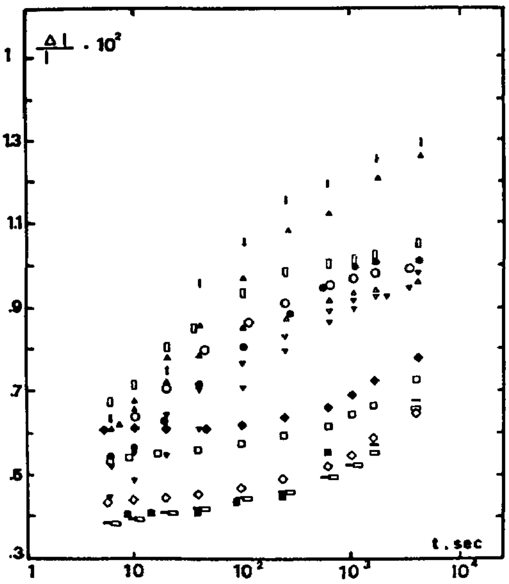

FIG.8 Normalized recovered deformation versus time. Key for symbols as in fig.s 4-7.

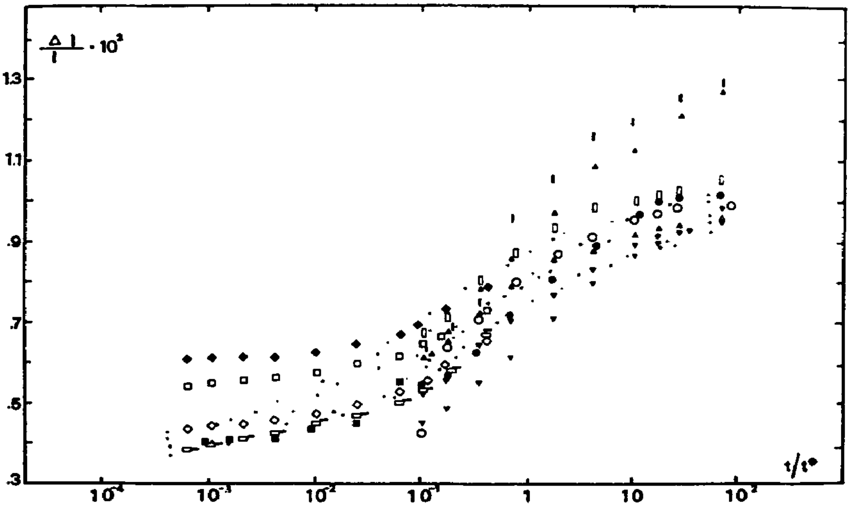

FIG.9 Normalized recovered deformation versus dimensionless time. Dotted curves are relative to tests at room temperature reported in (2). For other symbols see fig.s 4-7.

adopted normalizing procedure takes into account most of the phenomena involved in deformation recovery behavior of the material and thus offers useful suggestions in view of an accurate modeling of the recovery phenomenon.

Aknowledgement

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