

# Compressive recovery behaviour of a polycarbonate

G. Titomanlio and G. Rizzo

*Istituto di Ingegneria Chimica, Università di Palermo, Palermo, Italy*

*(Received 3 March 1978; revised 3 June 1978)*

Polycarbonate samples were subjected to large compression strains (beyond yielding) and were unloaded after some degree of stress relaxation. The subsequent deformation recovery was measured for several values of strain, loading rate and duration of stress relaxation. All the data could be reported as a single curve by normalizing the recovered strain with the stress at the end of the relaxation period.

## INTRODUCTION

All solid materials, at sufficiently small stress levels, show a complete recovery from deformation after unloading of the sample. At larger stresses only a partial deformation recovery is observed.

Many polymeric materials, in the appropriate temperature range<sup>1,2</sup>, are capable of undergoing large deformations which, at constant temperature, appear to be mostly permanent. However, under these conditions a small recovery is also observed, part of which is instantaneous and the other part is time dependent and reaches an apparent steady value after a relatively short time interval. One should really say<sup>3</sup> that the material attempts to revert back to its undeformed state but, after a relatively short time interval and because of the extremely long relaxation times involved (unless the temperature is very close to the glass transition temperature) the sample dimensions do not show any further significant change and, as a consequence, a large part of the deformation undergone appears stable.

The study of recovery after large deformations is clearly of importance for all cold-forming techniques and especially for those where bending processes are involved. Data for large deformation recovery as a function of time are presented below and the effect of loading history and deformation level is analysed.

## EXPERIMENTAL

The deformation recovery with sample unloading was measured after compression strain ramps were made on cylindrical samples obtained by machining the material supplied in the form of a rod. The material used was Lexan, a bisphenol A polycarbonate (4,4-dioxydiphenyl-2,2-propane carbonate) manufactured by General Electric. Its average molecular weight, as determined by intrinsic viscosity measurements, was 25 000.

In order to show up the presence of frozen-in stresses, a piece of rod was heated up to the glass transition temperature. Because of the absence of any shrinkage it was concluded that the material was free of any internal stress.

All tests were performed at room temperature (about 20°C) by means of an Instron testing machine Model 1115

on samples whose initial height to diameter ratio was 1 and whose initial height was 9 mm. Although some grease was used in order to reduce the friction between the dies and the samples, these deformed into a barrel shape, albeit not pronouncedly. The barrelling was neglected when evaluating changes of the sample cross-sectional area, which was used for calculating the stress.

At the end of the strain ramps, some stress relaxation was allowed and then the samples were unloaded by moving the machine crosshead at constant velocity. The machine crosshead was stopped when the recorder indicated zero stress. Holding the crosshead at constant position produced a stress increase; releasing it using the same procedure allowed evaluation of subsequent recovery.

Some exploratory tests were made in order to detect the effect of both the crosshead speed during the unloading ramps and the amount of stress increase, 'stress steps', during the periods when the crosshead was held at constant position. In particular, holding constant all the other experimental variables, tests were performed with the following values of the unloading deformation rate: 3.3, 6.6 and 33 h<sup>-1</sup>. For each of these unloading rates two tests were performed one at each of the following 'stress steps': 0.1 and 0.2 kg/mm<sup>2</sup>. Lower data reproducibility was observed for tests performed with the larger unloading rate, in particular it was 3 and 5% at the unloading rates 3.3 and 33 h<sup>-1</sup>, respectively. Within these reproducibilities and within the ranges studied the recovery data did not show any sensitive effect of either unloading rate or length of the 'stress steps'. All the data reported in the following were obtained with stress steps of 0.1 kg/mm<sup>2</sup> and an unloading rate of 6.6 h<sup>-1</sup>.

## RESULTS

Recovery data have been collected for two values of the loading rate and for several values of both the strain at the end of the strain ramps and the length of the stress relaxation period,  $t^*$ . For all tests the deformation at the end of the strain ramps was larger than the yield deformation of the material.

Several of the data refer to measurements made after the samples had undergone stress relaxation for the same time,  $t^*$ . Before the relaxation, the samples were deformed by

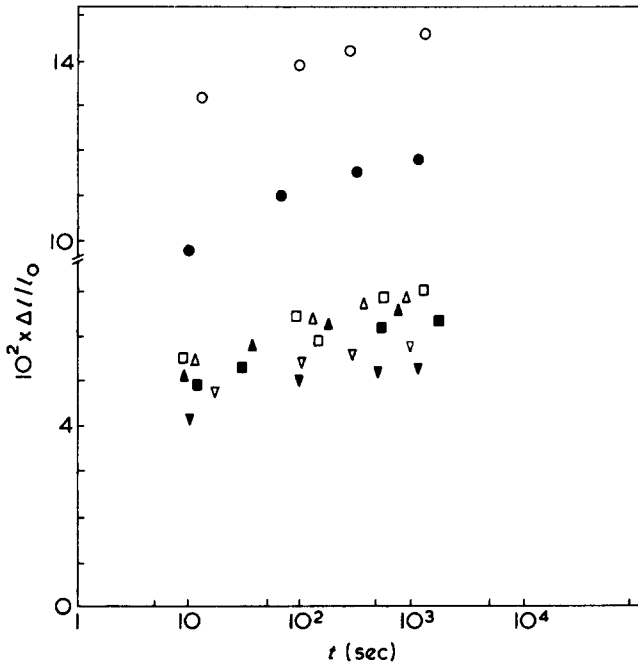


Figure 1 Recovery versus time of samples deformed up to different strains  $e_r, t^* = 15 \text{ sec}$ .

$e_r$	$\alpha_0 = 33 \text{ h}^{-1}$	$\alpha_0 = 3.3 \text{ h}^{-1}$
0.85	▲	△
0.75	▲	▽
0.59	□	■
0.32	●	○
0.30		○

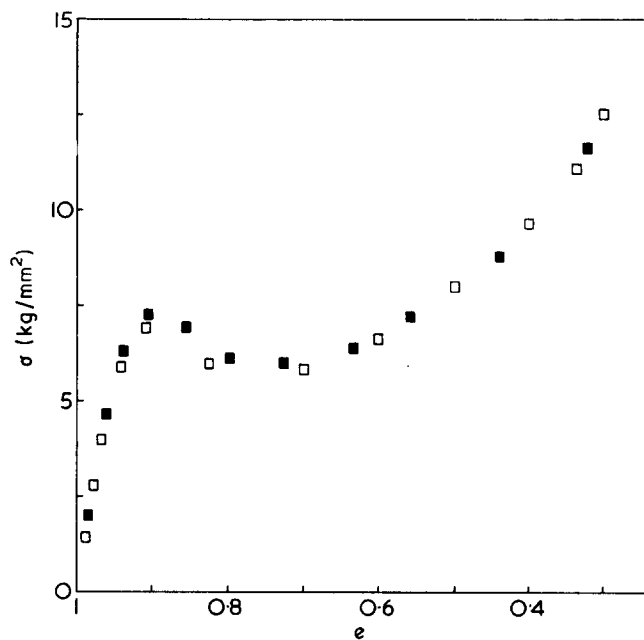


Figure 2 Stress versus strain during constant velocity compression tests.  $\alpha_0$  is the initial deformation rate. ■,  $\alpha_0 = 33 \text{ h}^{-1}$ ; □,  $3.3 \text{ h}^{-1}$

compression up to different strains  $e_r \equiv l_r/l_0$ , where  $l_0$  is the initial sample height and  $l_r$  is the sample height at the end of the strain ramp (and during the stress relaxation). The strain recovery  $\Delta l/l_0$ , where  $\Delta l$  is the increase in sample length caused by stress removal, is plotted in Figure 1 versus the time,  $t$ , as measured from the end of the stress relaxation

for two values of the initial deformation rate  $\alpha_0 \equiv v/l_0$ ,  $v$  being the velocity of the machine crosshead during the loading ramp. All the data in this Figure show that, at a fixed value of the strain,  $e_r$ , and at any time,  $t$ , the recovery is smaller for the larger  $\alpha_0$  value.

The stress measured during constant velocity tests is plotted in Figure 2 versus the strain  $e \equiv l/l_0$ , where  $l$  is the current sample length. By comparing the data of Figures 1 and 2 we observed that at each deformation rate and for each time,  $t$ , the recovery changes with the strain,  $e_r$ , in a fashion very similar to the stress,  $\sigma_r$ , reached by the material at the end of the strain ramps.

The data reported in Figure 3 describe the effect of duration of stress relaxation: samples which had relaxed for different times,  $t^*$ , were allowed to recover; before the relaxation all of them were deformed by the same amount and again both values of the initial deformation rate,  $\alpha_0$ , were considered. At any time,  $t$ , the recovery is smaller for larger  $t^*$  values. Furthermore, as already shown by the data of Figure 1, a larger recovery was observed for the sample loaded with the larger deformation rate.

A few tests were performed in order to analyse better the influence of the sample loading history on the recovery. Instead of a single loading strain ramp, more complex loading procedures were adopted. The recovery behaviour observed during two of these tests is compared in Figure 4 with the behaviour observed during a 'normal' test (i.e. performed on a sample loaded with a single ramp). The three samples were deformed to the same strain,  $e_r$ , and were allowed to relax for equal time intervals,  $t^*$ . The loading procedures were as follows. Sample b in Figure 4, was loaded with two consecutive strain ramps: the first being performed with a velocity ten times smaller than the second whose velocity was equal to that of the 'normal' test, a. The sample of test c was loaded with two strain ramps but these

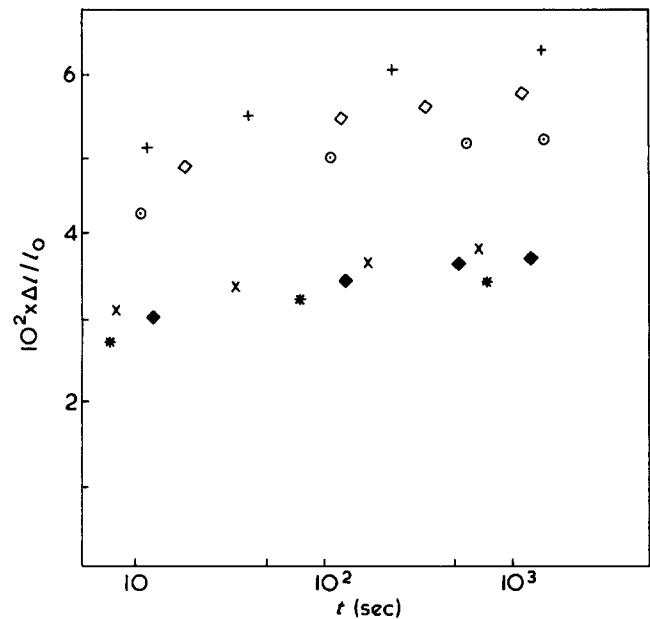


Figure 3 Recovery versus time after different relaxation intervals  $t^*, e_r = 7.5$ .

$t^*$ (sec)	$\alpha_0 = 3.3 \text{ h}^{-1}$	$\alpha_0 = 33 \text{ h}^{-1}$
2	+	
25	◇	○
900	x	◆
9600	*	

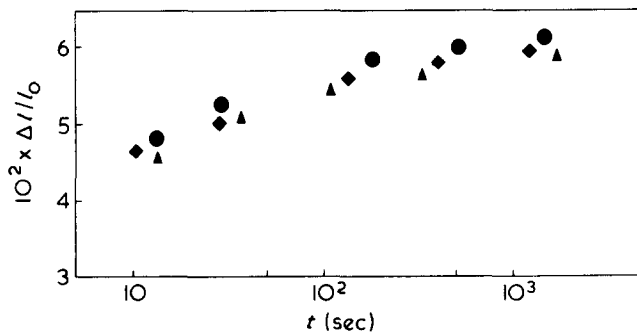


Figure 4 Recovery versus time for samples loaded with different procedures.  $\alpha_0$  is the ratio between the machine crosshead in the last strain interval and the initial unoriented sample height  $l_0$ .  $t^* = 15$  sec;  $e_r = 0.59$ ;  $\alpha_0 = 33 \text{ h}^{-1}$ . ●, a; ◆, b; ▲, c

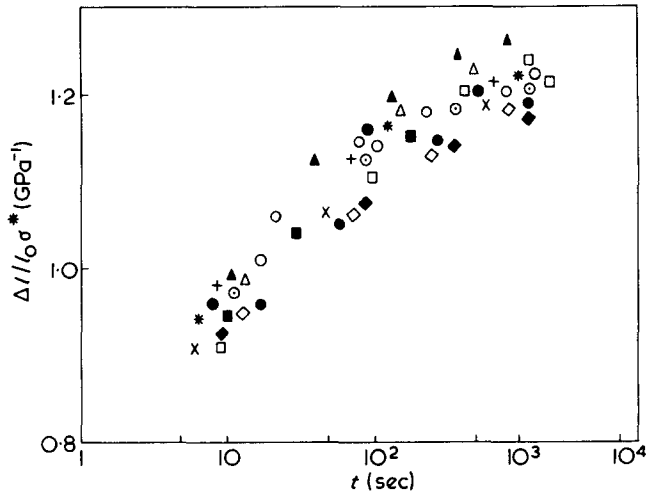


Figure 5 Recovery master curve. Key to symbols as in Figures 1 and 3.

were not consecutive, the sample being unloaded and stress free for 0.5 h after the first ramp; the second ramp was long enough to overcome the new yielding. In this case the second ramp was performed with a velocity equal to that of the test a. For tests b and c the strain reached during the first loading ramp was  $e = 0.65$ . Although it is close to  $e_r$ , the recovery behaviour of the three samples can be considered identical, within the experimental error. One can then infer that at fixed  $t^*$  and  $e_r$  levels the recovery depends essentially upon the deformation rate in the last strain interval. It should be pointed out that in Figure 4 the recovered strain is measured for all samples with respect to the initial unoriented sample length,  $l_0$ . Had the recovery of sample c been measured with respect to the sample length after the stress free period, the observation described above would have not been verified.

## CONCLUSIONS

In Figures 1 and 2 a proportionality was observed between the recovery and the stress,  $\sigma_r$  reached by the material at the end of a constant velocity compression ramp up to the deformation,  $e_r$ . This observation was limited to data taken after stress relaxation of the same length,  $t^*$ . On the other hand the data of Figure 3 show that for a given  $e_r$  the recovery decreases when either  $t^*$  or the loading rate increases.

Stress relaxation tests performed in compression on the same material<sup>4</sup> have shown that after yielding the rate of stress relaxation depends on the strain as measured with respect to the virgin unoriented material configuration. In particular there is a slower relaxation at larger reductions of the sample length. Furthermore the larger the loading rate of the sample the larger the relaxation rates which have been observed in tests performed on this and other materials<sup>4,5</sup>.

All these observations indicate that qualitatively the recovery changes together with the stress,  $\sigma^*$ , at the end of the stress relaxation (and just prior to the recovery itself). In fact both  $\sigma^*$  and  $\Delta l/l_0$  increase with  $\sigma_r$  and decrease when either  $t^*$  or the deformation rate increases. These considerations have suggested plotting the ratio  $\Delta l/l_0 \sigma^*$  versus the time,  $t$ , for all data reported in Figures 1 and 3. The resulting plot is shown in Figure 5 where the main features of the observed recovery behaviour seem to be accounted for by the chosen group.

In conclusion, if the master procedure, used for Figure 5, is shown to be valid for different polymers, the strain recovery of a material for any deformation level in the plastic region (i.e. after yielding), loading history and  $t^*$  value could be evaluated simply from a knowledge of its stress relaxation behaviour.

Furthermore the observation that in any case there is a smaller recovery when the material is loaded more rapidly in the last strain interval is of obvious interest in all cold forming operations.

## REFERENCES

- 1 Ender, J. *Macrom. Sci. (B)* 1970, 4, 635
- 2 Roe, J. M. and Baer, E. *Int. J. Polym. Mater.* 1972, 1, 133
- 3 Rusch, K. C. and Forrester, Jr, J. R. *Soc. Plast. Eng. (Tech. Pap)* 1971, 17, 59
- 4 Titomanlio, G. and Rizzo, G. *J. Appl. Polym. Sci.* submitted for publication
- 5 Titomanlio, G. and Rizzo, G. *J. Appl. Polym. Sci.* 1977, 21, 2933