JOURNAL OF MATERIALS SCIENCE 9 (1974) 929–933

Temperature effect at self-oscillating necking during extension of polyethylene terephthalate (PETP)

RUTGER ROSEEN
AB Atomenergi, Fack, 611 01 Nyköping 1, Sweden

Extension of PETP films at constant rate of deformation may, under certain conditions, result in stress oscillation and a periodic necking. The periodic necking results in formation of transverse bands, alternately opaque and transparent, in the sample. In this study, the temperature rise of such an oscillating neck was measured using an AGA Thermovision camera. The measured maximum surface temperature rise is very high, up to about 95° C and the temperature in the interior is probably higher. The formation of the opaque zone starts in the centre line of the foil and spreads out to a maximum width during the advance of the neck. It then suddenly ceases without reaching the surface of the sample. The opacity is probably a secondary effect dependent only on the high temperature formed by dissipated energy from the necking process and not affecting the mechanism of self-oscillating necking.

1. Introduction

Recently, Andrianova *et al* [1] detected a new phenomenon in necking of some polymers. They found that extension of amorphous PETP films under certain conditions results in periodic stress oscillations and a periodic change in the appearance of the sample. They also concluded that the following factors are critical for the appearance of the phenomenon: rate of straining, compliance of the sample, thermal conductivity, heat transfer and crystallization. However, the effect is not restricted to PETP but is also observed in crystalline polymers such as PP and PE [1] as well as in metals at temperatures approaching absolute zero [2-4]. The conditions under which the phenomenon appears have been analysed theoretically by Barenblatt [5]. The object of this work, which was done in connection with Barenblatt's studies, was to study the temperature at an oscillating neck, using a thermovision camera. The technique has been successfully used earlier to measure the temperature rise ahead of fatigue cracks [6].

2. Material and experimental details

Experiments were conducted on samples of amorphous PETP film made in the Soviet Union. From the films, which were 250 to 300 μm thick, strips, 6mm wide and with 70 mm gauge length were cut out. The length and the rate of extension were determined using the graph shown in Fig. 1 in order to make sure that self-oscillating necking would occur. The samples were strained in an Inston tensile testing machine at a rate of 50 mm min^{-1}. The room temperature during the testing was 22°C. In order to initiate necking and avoid brittle failure, the sample was bent once (crumpled) at one point immediately before testing.

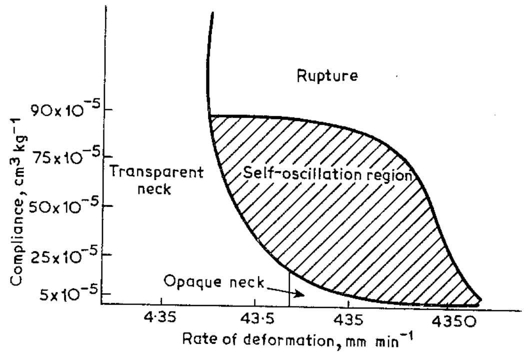

Figure 1 Regions of deformation modes corresponding to different regimes of extension of PETP specimen [1].

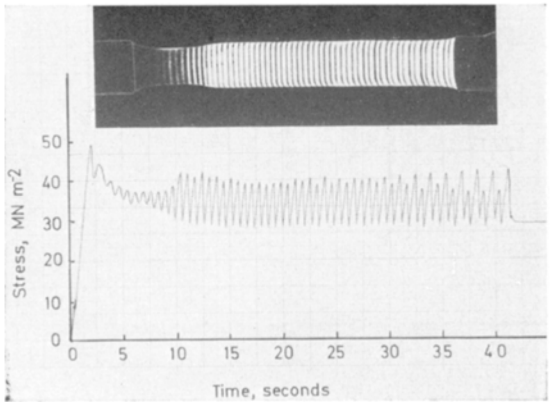

Figure 2 Stress versus time for a specimen deformed at 50 mm min^{-1} constant rate of extension. The specimen is shown against a black background.

The temperature measurements were conducted using the AGA Thermovision System 680. The temperature distribution was registered by isotherm functions either on a black and white or colour TV monitor, working at a picture field frequency of 16 per sec, and recorded using a Nikon F camera with automatic film advance of two frames per sec and $\frac{1}{8}$ sec exposure time.

After straining, the specimens, moulded in Araldite, were prepared and studied by means of standard metallographic techniques. In order to obtain better contrast in the crystalline parts, polarized light was used in a Leitz Orthoplan microscope.

3. Results

3. Stress and strain behaviour

At a constant clamp speed of 50 mm min^{-1}, the stress increases to an upper yield point at about 50 MN m^{-2}. After that it starts to oscillate between 30 and 40 MN m^{-2} with a frequency of about 1.5 Hz. The nature of the process can be observed in Fig. 2, which shows a typical record of the variation of stress with time, and the appearance of the sample. The fluctuating stress results in two different sections of the sample: first a period under which a transparent neck forms, which is followed by a period under which an opaque neck forms. The neck advances essentially during the period of decreasing stress as shown by Andrianova [1]. The reason for the opacity is probably that the amorphous sample crystallizes due to the high temperature generated by the necking process.

In order to examine the role of the heat transfer from the sample, an air stream was directed onto an oscillating sample. The effect of this is shown in Fig. 3. The oscillation stops abruptly and the sample fails in a brittle manner. The heat transfer from the sample was thus shown to be an important factor for the phenomenon to appear.

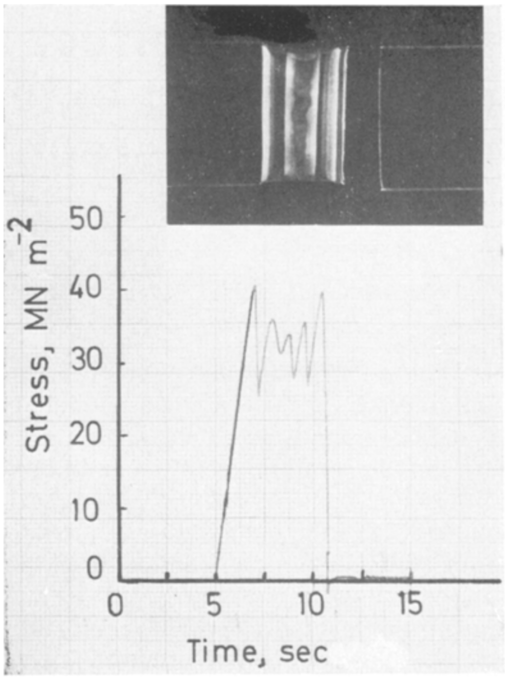

Figure 3 Stress versus time for a specimen deformed at 50 mm min^{-1} constant rate of extension. The specimen was exposed to an air stream after oscillation had begun.

3.2. Temperature measurement

The period of heat dissipation is very short compared to the exposure time of the camera, which makes it impossible to follow the whole cycle, therefore the measurement was concentrated upon the maximum surface temperature. Fig. 4 shows a typical thermograph, where one isotherm function is used. By moving the isotherm towards higher temperatures it is very easy to exactly determine the highest temperature during one cycle. An alternative method is demonstrated in Figs. 5 and 6. By using several isotherms it is possible to record the temperature distribution during the exposure time. The disadvantage of the latter method is that one cannot be sure of recording the maximum temperature during the cycle and that the temperature distribution is not correct because the heat diffuses during exposure time.

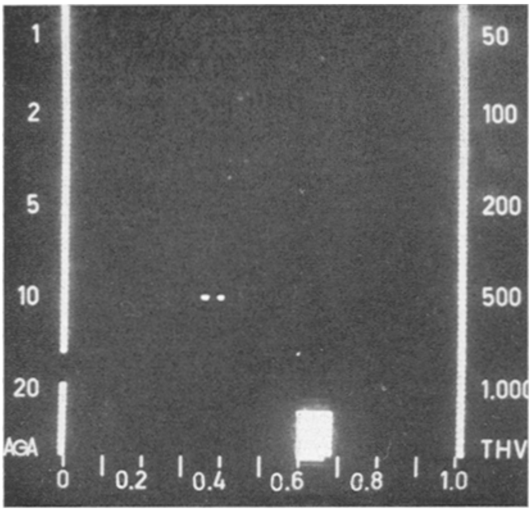

Figure 4 Example of registration of maximum temperature using one isothermal function. The temperature in the white areas is between 92 and 95°C, corrected values. The rate of extension was 50 mm min^{-1}.

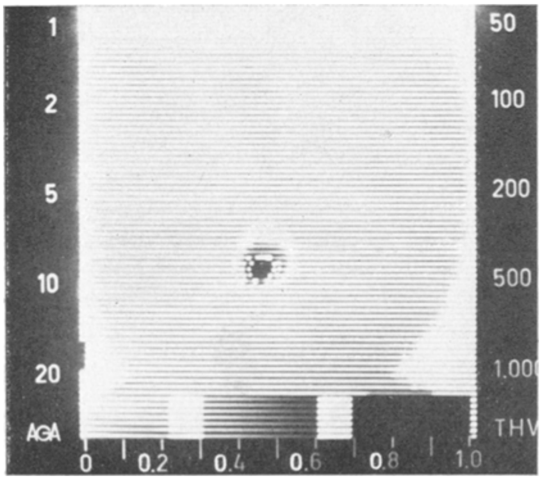

Figure 5 Example of temperature registration with two isothermal functions. The temperature is 80 to 82°C respectively, 88 to 90°C corrected values. The rate of extension was 500 mm min^{-1}.

Another important factor is that the spatial resolution of the camera is not good enough for this size of temperature zone. In order to determine the correct temperature it is necessary to use the calibration curve in Fig. 7. In this case, the width of the high temperature zone is approximated to the width of the opaque zone. Table I shows the corrected and uncorrected values of the temperature measurement. The temperature varies between 92 and 101°C.

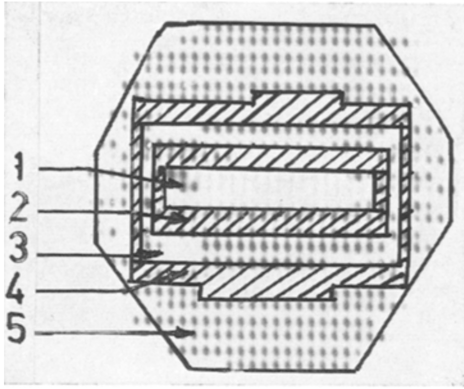

Figure 6 Example of temperature registration with ten isothermal functions. The original photograph was taken in colour. The temperatures are: (1) 86 to 90°C, (2) 82 to 86°C, (3) 78 to 82°C, (4) 74 to 78°C, (5) 70 to 74°C. These are corrected values.

There is a correlation between the length of the sample and the width of the opaque zone. As the length increases the zone width increases. This leads to an increase in the observed temperature as the sample elongates. See, for instance, sample 2 in Table I.

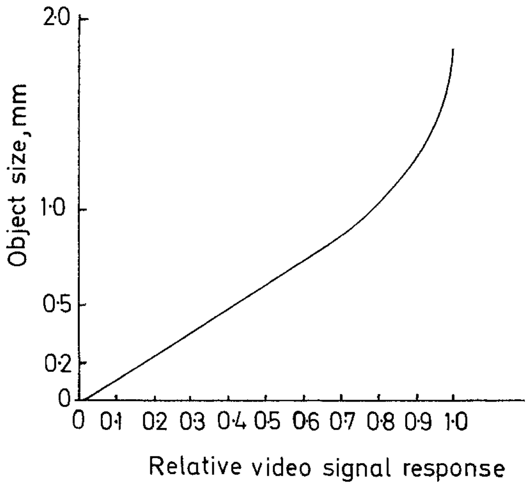

Figure 7 Calibration curve for small angle objects.

3.3. Structural observations

Fig. 8 shows the detail of Fig. 2 at higher magnification. Two observations can be made: first the front of the opaque zone is not a straight line and secondly the opaque zone never reaches the edge of the sample. The large heat transfer from the edge of the sample is the probable explanation for this. The same feature

TABLE I Corrected and uncorrected maximum temperature measurements in isotherm unit (iu) and °C

	Reference temperature		Observed temperature	Width of opaque zone (mm)	Corrected temperature	
	(°C)	(iu)	(iu)		(iu)	°C
1	38	19.5	22.5	0.30	87	97
2A	39	20	24	0.35	80	92
2B	39	20	39	0.50	95	101
3A	40	20.5	32.5	0.50	80	92
3B	40	20.5	33.5	0.50	82	94
4	49	27	29	0.40	85	96
5	50	28	31	0.45	82	94

is evident from Fig. 9, which shows a section along the sample.

The transformed (crystallized) zone starts in the centre line of the foil and spreads out to a maximum width during the advance of the neck after which it suddenly ceases. In no instance during this study was it possible to find a transformed zone, extending to the surface of the sample. From these illustrations (Fig. 9) it is obvious that there are only two different zones in the sample. Another observation which can be made from Fig. 9 is that the sample is thicker along the opaque zone, probably because the high dissipated heat makes the propagation of the neck easier.

The most probable explanation for the opaque zone is that crystallization is a secondary effect, which only depends on the temperature generated in the neck. This is also consistent with the fact that a jumping neck also occurs in some crystalline metals.

In order to evaluate the effect of temperature on opacity, a few samples both in the as-received and already necked conditions were heat-treated. The result was that they started to grow opaque at about 117°C, but the degree of opacity was lower than that observed under testing until heat-treatment at a temperature as high as about 175°C for 2 min was used. These conditions were the same for both necked and non-necked samples. Of course the opacity increases with both time and temperature, hence the conclusion is that the temperature in the central parts of the neck must be higher than 175°C due to the very short time at each deformation cycle.

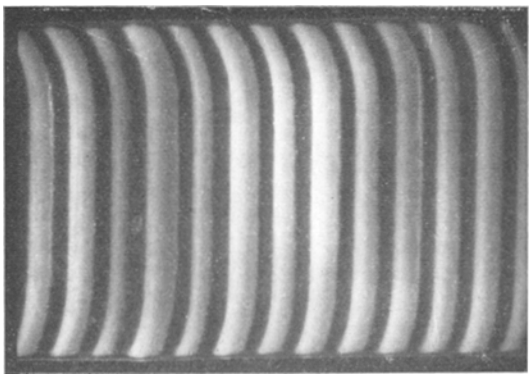

Figure 8 The appearance of the specimen after oscillating necking at 50 mm min^{-1} constant rate of extension. The edge of the sample is quite even, whilst the opaque area never reaches the edge. Polarized light (× 12).

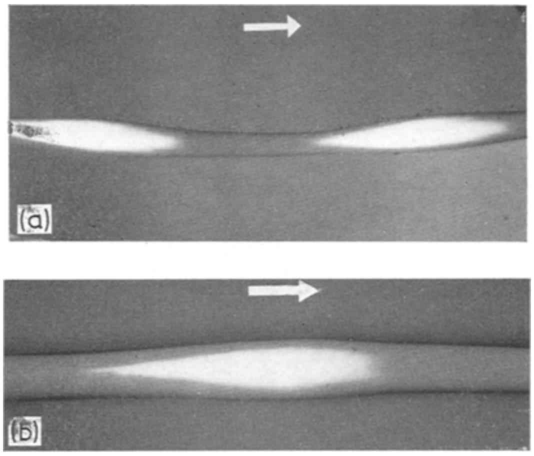

Figure 9 (a) A section through the specimen shown in Fig. 8. The arrow marks the direction in which the neck advances. The specimen is thicker along the opaque areas. Polarized light (× 60). (b) Same specimen as in (a). Only the centre part of the sample is opaque. Polarized light (× 128).

Another interesting fact in this connection is the extent to which heat-treatment influences the mechanical behaviour of the sample. A comparison in stress-strain behaviour for a heat-treated sample (1 min, 130°C) and a non-heat-treated sample is plotted in Fig. 10. The heat-treated

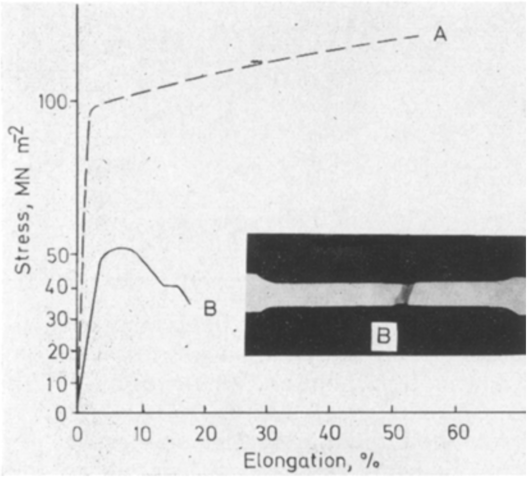

Figure 10 Comparison in tensile behaviour at 1 mm min^{-1} rate of extension between (a) original specimen and (b) heat-treated at 130°C for 2 min. The photograph shows the heat-treated sample.

sample has a lower yield stress and shows marked necking. However, in this case the sample is partly crystallized; fully crystallized PETP is extremely brittle.

4. Discussion

The values of stress-time behaviour are in excellent agreement with those reported by Andranova [1]. However, the maximum temperature observed here is only slightly higher, because the approximation of the width of the maximum surface temperature zone is probably too large, and this greatly influences temperature measurements. In this study, the width of the whole opaque zone was chosen, but the maximum temperature zone is probably smaller, as can be judged from Fig. 9. The method used here is not very satisfactory owing to insufficient spatial resolution of the camera. The results should, therefore, be regarded as lower limit values.

Contradictory to Andrianova [1] it was not possible to observe three distinct zones in the neck, but rather a continuous change from transparent to opaque neck. The opacity starts from the central parts of the neck and spreads to a maximum width, indicating that crystallization and opacity is only a result of the high temperature and not of mechanical factors. This corresponds to the results with metals [2-4], where no crystallization occurs.

5. Conclusions

1. During oscillating necking, the surface temperature of a neck rises to a maximum of about 95°C.
2. The corrected surface temperature of about 95°C is exceeded in the interior, probably by many tens of degrees.
3. The temperature distribution and opacity along the sample does not show any distinct zones, but rather a continuous increase as the neck advances until the necking suddenly ceases for a while.
4. The opacity is probably a secondary effect only depending on the high temperature formed by dissipated energy in the necking process, and not affecting the mechanism of self-oscillating necking.

Acknowledgements

This work was carried out at Aktiebolaget Atomenergi, Studsvik, Sweden, to which the author is indebted for permission to publish this paper. Thanks are due to Professor G. I. Barenblatt and Dr G. Östberg for their interest in and support of the investigation.

References

1. G. P. ANDRIANOVA, A. S. KECHEKYAN and V. A. KARGIN, *J. Polymer Sci. A2* **9** (1971) 1919.
2. G. Y. CHIN, W. F. HOSFORD and W. A. BACKFOTEN, *Trans. Met. Soc. AIME* **230** (1964) 1043.
3. V. RAMACHANDRAN, D. H. BALDWIN and R. E. REED-HILL, *Met. Trans.* **1** (1970) 3011.
4. L. P. KUBIN and B. JOUFFREY, *Phil. Mag.* **23** (1973) 437.
5. G. I. BARENBLATT, *Mechanika Tverdoga Tela* no. 5 (1970) 121.
6. R. ATTERMO and G. ÖSTBERG, *J. Frac. Mech.* **7** (1971) 122.

Received 5 October and accepted 30 October 1973.