Polymer Bulletin 34, 243–248 (1995)

Polymer Bulletin

© Springer-Verlag 1995

Split rate fatigue propagation in polymer blends

C. Koster[1], **V. Altstädt**[2], **H. H. Kausch**[1], **W. J. Cantwell**[1]

[1] Laboratoire de Polymères, Ecole Polytechnique Fédérale de Lausanne, Switzerland
[2] BASF AG, Karl-Bosch-Strasse 38, D-67063 Ludwigshafen, Germany

Received: 22 August 1994/Revised version: 14 November 1994/Accepted: 15 November 1994

Summary

The long-term properties of PαSAN/PMMA blends have been examined by conducting fatigue tests on a fracture mechanics-type specimen. Initial testing on the individual constituents showed that the crack initiation threshold was higher in the PMMA polymer than in the PαSAN system. Alloying these polymers resulted in the crack initiation thresholds between those of the two base polymers. A scanning electron microscopy study of the fracture surfaces indicated that fatigue loading resulted in the formation of well defined striations, the spacing of which depended on the crack velocity and morphology. The fatigue striations in a fine-structured blend remained straight and did not appear to be modified by the presence of the secondary phase. Crack extension in a coarser structure depended upon the characteristics of the particular phase in which the crack was extending. It was observed that the striations in the less ductile PαSAN phase were more closely spaced as well as being curved. It is believed that the crack propagated more rapidly in this phase resulting in a split rate crack velocity within the polymer blend.

Introduction

Blending polymers to combine and optimize the properties of the individual constituents has attracted considerable attention in recent years. To date, however, much of this work has concerned synthesis and subsequent study of the morphology of these materials. Only a relatively small amount of published work concerns their mechanical behaviour under long-term loading conditions. The objective of this study is to examine the influence of polymer alloying on the process of fatigue crack propagation in a PαSAN/PMMA blend.

Experimental Procedure

The materials studied in this programme consisted of a blend of PαSAN (LURAN® from BASF) and PMMA (LUCRYL® from BASF). The former contained 70% by weight α-methylstyrene and 30% by weight acrylonitrile and had a molecular weight of 92000 g/mol and a glass transition temperature, T_g, of 123°C. The PMMA component was composed of 95% by weight methyl methacrylate and 5% methyl acrylate. The molecular weight of the latter was 80000 g/mol and it's T_g 111°C. At temperatures above 155°C, blends of PαSAN/PMMA are not miscible but form distinct phases of the individual components. Such spinodal separation makes it possible to create different morphologies by varying either the processing temperature or the blend ratio. In this study, blends of 40/60% and 60/40% by weight PαSAN/PMMA were manufactured from granulates by extrusion in a ZSK 30/2 extruder. The heating element in the extruder was maintained at 200°C and the temperature of the polymer upon exiting the extruder was 211°C. After extrusion, the polymer blend was ground and dried for six hours at 80°C resulting in granulates suitable for compression moulding flat plates. Plates having a thickness of 4 mm were prepared in a two step operation. In the first step, the granulates were placed in a 160x180x4 mm mould and maintained in a press at 230°C under a moderate pressure for seven minutes. Once the granules had softened sufficiently, the mould was transferred to a second press maintained at 195°C and subjected to a pressure of 15 bar for four minutes. The mould was then cooled to 40°C in approximately seventy seconds while maintaining a pressure of 15 bar. This procedure resulted in high quality plates having a clear, well defined morphology. The morphology of certain plates was subsequently varied by annealing in a press for four hours at 220°C.

In preparation for testing, compact tension specimens with dimensions 62.5x60 mm were cut from the plates. A natural pre-crack was propagated by fatigue loading at 10 Hz. Fatigue testing was conducted on an MTS 300.05 servo-hydraulic machine at a frequency of 10Hz and a fatigue ratio R=0.1. The crack opening displacement (COD) was measured using a COD gauge mounted in front of the specimen. Following testing, a scanning electron microscope was used to investigate the fracture surface morphology of the failed samples.

Results and Discussion

The results of the fatigue crack propagation study are presented in Fig. 1 and summarized in table 1. The crack propagation threshold was lowest in the relatively brittle PαSAN polymer and highest in the PMMA system.

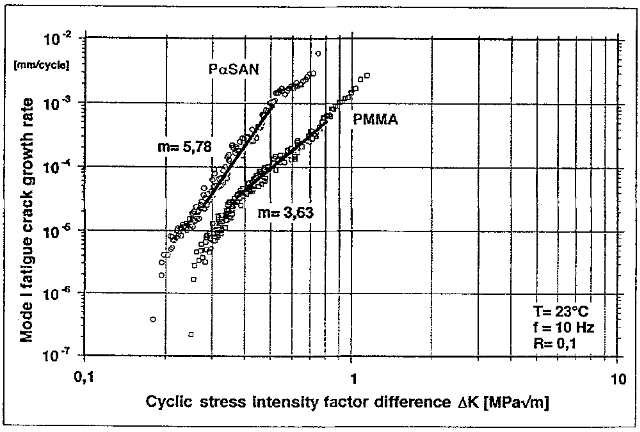

Figure 1 : Fatigue Crack growth as a function of stress intensity factor difference (the data for all blends are in between the data shown for the pure polymers)

The fatigue crack propagation thresholds in the 60/40 and 40/60 PαSAN/PMMA blends were situated between those of the pure polymers with the 40/60 PαSAN/PMMA offering the greater resistance to crack initiation. It is also interesting to note that the threshold values appear to exhibit a simple law of mixtures relationship. A Paris law of the form $\frac{da}{dN} = A\Delta K^m$ was assumed to be valid for each linear portion of the curves for each linear portion of the curves, where a is crack length, N number of load cycles and ΔK stress intensity factor difference; A and m are parameters; fitting the experimental curves to the Paris law yields the values of m presented in Table 1. For three of the four materials, the fatigue crack growth curves exhibited two slopes (Fig.1). For these cases two values of '*m*' are given in the table. From the complete data it is clear that the crack propagation rates in the blends are always comprised between than those in the pure polymers.

	PαSAN	60/40 PαSAN/PMMA	40/60 PαSAN/PMMA	PMMA
ΔK MPa√m Threshold	0.17	0.2	0.22	0.24
m	5.8 and 2.5	6.2 and 3.5	6.0 and 3.5	5.4 and 3.6

Table 1. *Fatigue threshold and Paris law constant 'm' (for $da/dN > 10^{-4}$ mm/cycle) for PαSAN, PMMA and their blends without annealing. Where two values of 'm' are given, the fatigue crack growth curve exhibited two slopes, initially the first value then the second.*

Subsequent tests on annealed 40/60 PαSAN/PMMA samples showed that thermally treating the blend did affect the morphology but did not modify it's fatigue crack growth resistance with the values of $\Delta K_{threshold}$ and '*m*' remaining unchanged.

A scanning electron microscopy study of the failed PMMA and PαSAN samples highlighted the presence of distinct fatigue striations parallel to the direction of crack pro-

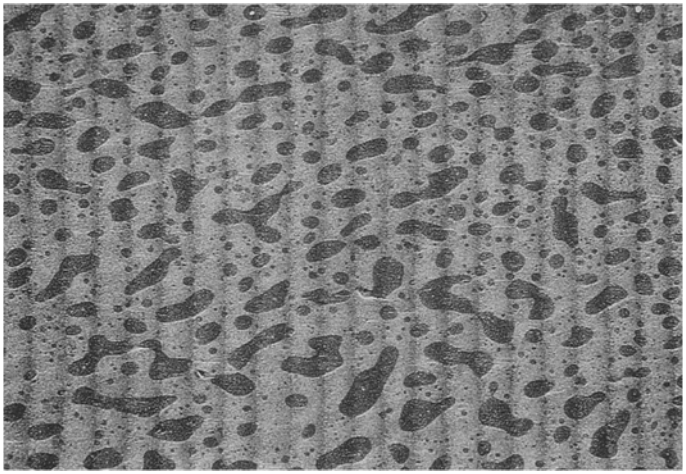

Figure. 2 : Scanning electron micrograph showing the threshold region on the fracture surface of the 60/40 PαSAN/PMMA blend. (Magnification : 1000 x)

pagation. For a crack growth rate of 10^{-6} mm/cycle the spacing of these striations was approximately 8 microns for the PMMA system and 4.5 microns for it's PαSAN counterpart.

The fracture surface in the threshold region (da/dN = 10^{-6} mm/cycle) of the 60/40 PαSAN/PMMA blend is shown in Fig. 2. Here, the two phase structure of the blend is evident. It was also clear that the level of adhesion between the two phases is good with no decohesions or interfacial failures occurring during fatigue loading. Here again, a distinctive pattern of equally spaced fatigue striations is apparent on the fracture surface. Crack propagation in this material occurred in a discontinuous manner in which the crack advanced in jumps leaving these distinctive features at regular intervals. Each striation does not result from a single fatigue cycle but occurs after a significant number of them have elapsed. For example, at a crack growth rate of 10^{-4} mm/cycle, each striation corresponded to approximately 350 load cycles. A closer examination of these striations indicated that the propagation of the crack was not modified by the presence of the secondary PMMA phase. Indeed, from the fracture surface it appeared that the crack propagated at roughly the same rate in both the primary and secondary phases. Further, the fatigue data presented in Fig. 1 suggests that the crack initiation threshold is situated between those of the two individual components. This and the morphological evidence in Fig. 2 suggest that both phases are contributing equally to the fatigue propagation response of the material. This type of morphology in which the size of the secondary phase is comparable with the striation spacing is considered to be fine in nature.

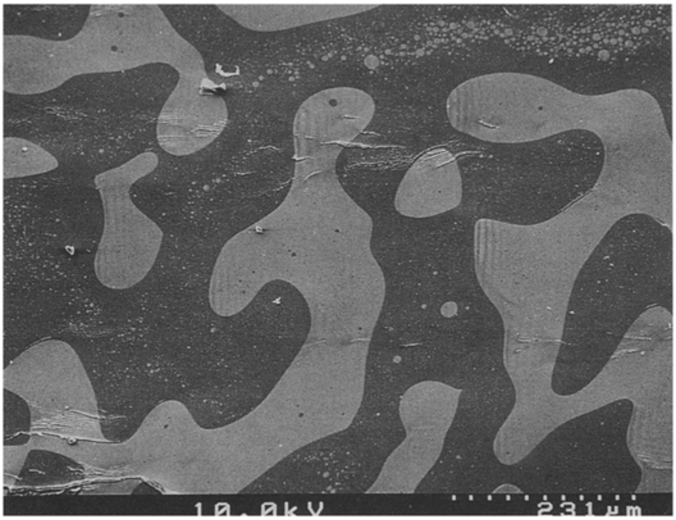

Figure 3. Micrograph of the annealed 40/60 PαSAN/PMMA blend. Crack velocity = 10^{-5} mm/cycle. (Magnification 130 x)

The fracture surface of an annealed 40/60 PαSAN/PMMA blend is shown in Fig. 3. Here, a much coarser co-continuous structure is apparent. A closer examination of the micrograph again indicates the presence of fatigue striations on the fracture surface. In this case, however, the manner in which the crack propagated was considerably different to that observed in the 60/40 blend with the separate phases playing individual roles in the failure process. Fig. 4 shows a high magnification micrograph in which the fatigue crack encounters the PαSAN phase. As the crack entered the PαSAN phase coarse striations spaced at approximately 10 micron intervals can be seen. As the crack continues to propagate in this phase the spacing of the striations drops to below 5 microns and the crack re-orients itself slightly. These striation spacings correspond roughly to those observed in the pure PαSAN and PMMA samples suggesting that as the crack enters the PαSAN phase, the crack initially continues to advance as it did in the PMMA phase. As crack growth continues, the crack growth mechanism becomes more and more influenced by the characteristic properties of the phase in which it is growing. This evidence suggests that it is likely that the crack propagates at a faster rate in the PαSAN phase than in the less brittle PMMA matrix.

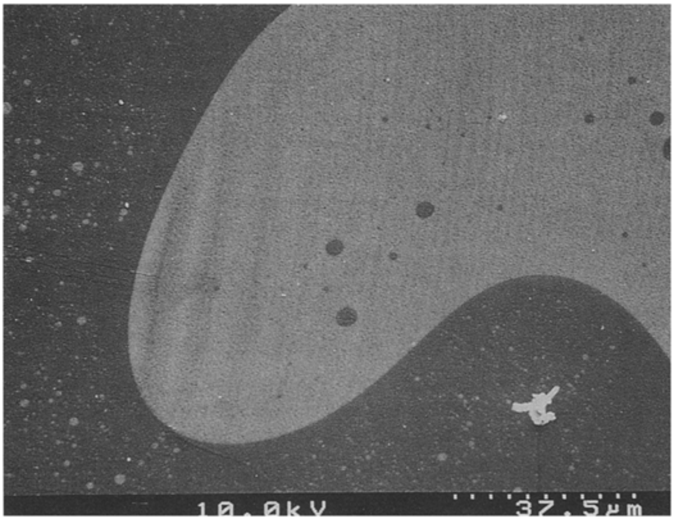

Figure 4. Higher magnification (800 x) of the specimen shown in Fig.3.

This suggestion appears to be substantiated in Fig. 5 where a crack exits a PαSAN phase. Here, the striations appear curved suggesting a gradient in crack velocity between the two phases. The fracture surface markings in the lighter PαSAN phase in Fig. 4 suggest that the crack propagated more rapidly in this domain than in the PMMA phase. It is also interesting to note that this gradient or discontinuity in crack velocities resulted in the formation of cracks at or close to the interface between the two polymer phases, Fig. 5.

Clearly, the process of crack propagation in such two-phase materials is complex, however, some conclusions can be drawn from this study. Firstly, the threshold for fatigue crack growth is strongly dependent upon the specific composition of the blend. It has been shown that the threshold value for a blend is situated between those of the individual constituents and appears to obey a rule of mixtures relationship. For fine blend morphologies, the process of crack propagation appears to be uniform across the crack front with there being no appreciable difference in crack velocity or striation spacing. However, if the structure is coarser, the individual components begin to play a more individualistic role with the crack extension tending to be determined by the properties of the specific phase in which the cracking is propagating.

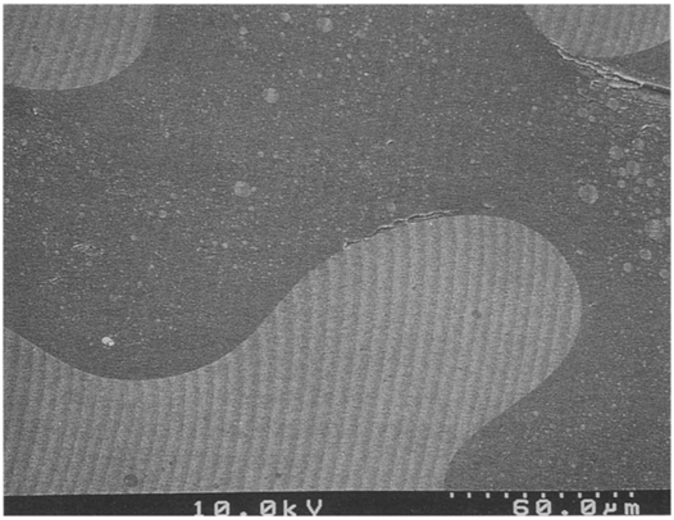

Figure 5. Micrograph of the annealed 40/60 PαSAN/PMMA blend. Crack velocity = 10^{-5} mm/cycle. (Magnification 500 x)

References

1. Herzberg, R.W. & Manson, J.A. (1980). Fatigue of Engineering polymers, Academic Press, New York, p. 185.

2. Kinloch, A.J. & Young R.J. Fracture Behaviour of Polymers, Applied Science Publishers, London 1983, p. 463.

3. Kausch H.H. , Polymer Fracture,, 2nd Edition, Springer Verlag, Berlin, New York, 1987, p. 396.