The effect of Poisson contraction, while not having as direct an influence on multiple fracture as the Kelly–Zweben paper would indicate, will certainly result in a decreased fibre/matrix normal force when the matrix cracks. Only in the case of composites prepared with an epoxy matrix when $\nu_m > \nu_f$ was the Poisson contraction alone found to be sufficient to maintain fibre/matrix contact. However, the final result indicates that the Poisson contraction will lead to a decreased pressure at the interface on matrix cracking and hence a reduced frictional stress transfer at the interface. This reduction in normal stress at the interface will be the subject of a future paper from our laboratory.

Acknowledgement

I wish to express my thanks to Dr Elizabeth Yoffe for her guidance in the analysis of the matrix deformation and the encouragement of Professor D. Tabor under whose supervision this project is being carried out. I also wish to thank the Building Research Establishment for financial support and assistance.

References

1. A. KELLY and C. ZWEBEN, *J. Mater. Sci.* **11** (1976) 582.
2. R. HILL, *J. Mech. Phys. Solids* **12** (1964) 199.
3. A. E. H. LOVE, "A Treatise on the Mathematical Theory of Elasticity" p. 144.
4. D. J. PINCHIN and D. TABOR, Mechanical Properties of the Steel/Cement Interface: Some Experimental Results, in "Fibre Reinforced Cement and Concrete", Vol. 2 (Construction Press, Hornby, Lancs, 1976) in press.

Received 13 April
and accepted 28 April 1976

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A new method of film casting for electron microscopy

Many studies have been reported of microphase spearation in block co-polymers [1] which can lead to ordered arrays of regular domains [2–4]. One widely used method of studying these arrays in thermoplastic elastomers such as SBS and SIS block co-polymers is electron microscopy. This requires the preparation of very thin films of the material either by cutting using an ultramicrotome or by solvent casting. The present article deals with the application of the latter technique to rubbery styrene–butadiene block co-polymers, and describes an improved solvent casting method.

A variety of casting techniques have been described in the literature [5–7]. With block co-polymers, the Kato [5] method gave a very poor success rate. Hall [6] has suggested a modification of Kato's technique involving casting the film on a clean water surface. However, Lewis and Price [7] have reported that Hall's method is unsatisfactory for ABA poly (styrene-*b*-butadiene) co-polymers as the films tended to break when dried. They suggested a method [7] involving casting on a freshly distilled and cleaned mercury surface. The grids are held by tweezers and dragged over the surface to collect the film. This method has also been found to be rather unsuccessful with highly rubbery materials which appear to collect at the edge of the grid. However, when this technique worked the film was very satisfactory suggesting that the surface obtained is good for ABA poly (styrene-*b*-butadiene) co-polymers and that the problem simply lies in the transfer of the film onto the grids.

Fig. 1 shows a cross-sectional view of the apparatus developed for solvent casting on to electron microscope grids. Freshly cleaned and distilled mercury (A) is placed in a circular steel vessel (B) fitted with a push-fit Teflon ring (C). The ring just touches the mercury surface to prevent solution running into the trough round the edge of the vessel. A circular disc composed of two layers (D and E is then placed in the mercury pool and allowed to float. The top half of the disc is formed

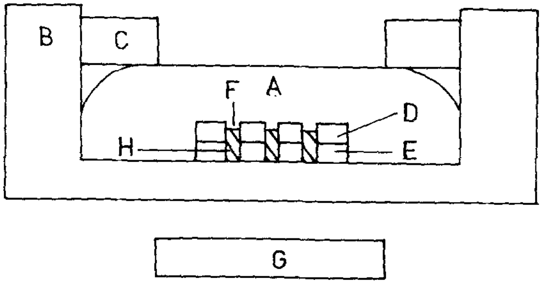

Figure 1 Cross-sectional view of casting apparatus.

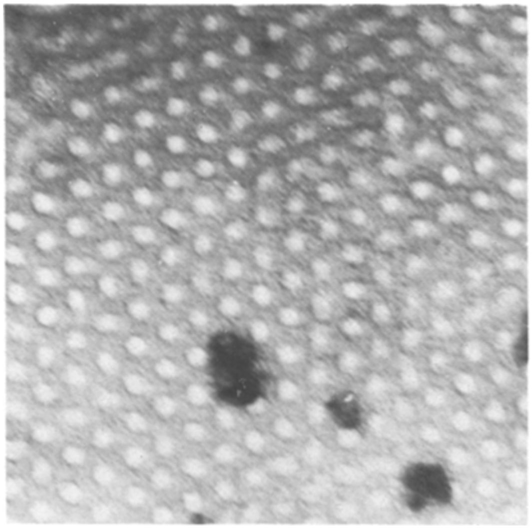

Figure 2 An electron micrograph of an ABA poly(styrene-*b*-butadience) co-polymer blended with polystyrene (10% by weight), × 104 000.

of copper which has been allowed to amalgamate with mercury, whilst the bottom layer is steel. Electron microscope grids are placed in the recesses (F) on the top of the disc into which they fit snugly. An electromagnet (G) is then switched on and the disc is pushed carefully to the bottom of the mercury pool where is is held by the magnetic field. A dilute solution of the polymer is poured onto the mercury surface and allowed to evaporate to give a film about 40 nm thick. When the film is completely dry the electromagnet is switched off allowing the disc to rise to the surface. The disc floats practically level with the surface and no troughs, caused by surface tension, are formed around the disc because the disc surface is a copper–mercury amalgam. Therefore, the the rubber film is not torn by the rising disc. The disc is carefully retrieved from the mercury pool and the grids removed by means of ejector pins (H).

A very high success rate has been found for this technique: nearly every grid examined to date has had suitably thin and uniform samples on it.

Contrast between the phases in this material was achieved by staining in osmium tetroxide vapour. The grids were suspended above an aproximately 1% aqueous solution of osmium tetroxide in a closed vessel for 30 min, and were then examined with a Siemens Elmiskop 1A electron microscope using an accelerating voltage of 80 kV.

Fig. 2 illustrates the quality of electron micrographs obtainable by this technique. The system chosen is a blend of an ABA poly(styrene-b-butadiene) co-polymer with polystyrene. This is a very difficult system as all the added homopolymer is not accommodated in the pre-existing polystyrene domians, some forming small interdomain polystyrene regions resulting in a material exhibiting much poorer phase contrast than the unblended copolymer.

Acknowledgements

A. Beamish wishes to thank the Science Research Council for a Research Studentship. We also wish to thank Mr R. Hartley of this Department for constructing the apparatus.

References

1. D. C. ALLPORT and W. H. JANES, Eds., "Block Copolymers" (Applied Science, London, 1973).
2. M. MATSUO, *Japan Plastics* 19 (1968) 6.
3. P. R. LEWIS and C. PRICE, *Nature* **223** (1969) 494.
4. G. KÄMPF, H. KRÖMER and M. J. HOFFMANN, *Macromol. Sci.* **B6** (1972) 167.
5. K. KATO, *J. Polymer. Sci.* **B4** (1966) 35.
6. C. E. HALL, "Introduction to Electron Microscopy" (McGraw-Hill, New York, 1966) p. 278.
7. P. R. LEWIS and C. PRICE, *Polymer* **13** (1972) 20.

Received 7 April
and accepted 4 May 1976

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