

Figure 5 Glass-fibre-reinforced gypsum plaster broken in flexure after the fire test showing uniform pull-out of the fibres.

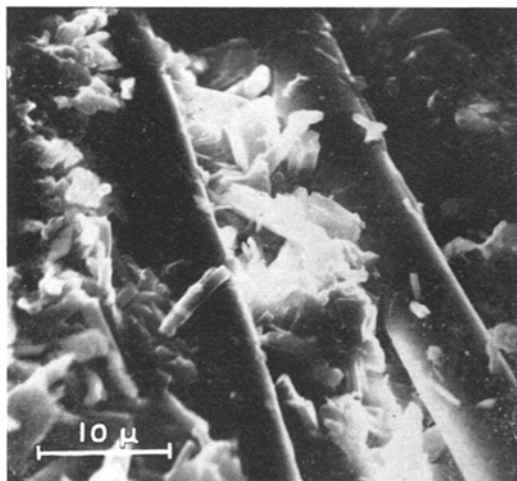


Figure 6 Bonding in glass-fibre-reinforced gypsum plaster.

case of manufacture by a spray-suction method [3] employing rovings, the strands disperse in the matrix as individual filaments, although these largely retain their original alignment.

Bonding between the fibre and set gypsum plaster does not seem to be very uniform along the length of the fibres. This is illustrated in fig. 6. The bond strength value measured previously [4] for this fibre/matrix combination is $6.76 \times 10^6 \text{ N/m}^2$ (981 lb/in²*) but it appears that much of it is frictional in origin aided perhaps by the slight volume expansion of the plaster mix during setting. This feature of discontinuous bonding also helps to explain why the density of the composite material decreases rather than increases with increase in the percentage of glass-fibre addition. With increase in the percentage of glass the voids around the fibres increase proportionately.

*1.0 lb/in.² = $7.0 \times 10^{-2} \text{ kg/cm}^2$

Acknowledgement

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References

1. C. W. OATLEY, W. C. NIXON, and R. F. W. PEASE, *Adv. Electronics and Electron Phys.* **21** (1965) 181.
2. A. J. MAJUMDAR and J. F. RYDER, *J. Glass Tech.* (1968) to be published.
3. British Patent Application 4962/67 1967, National Research and Development Corporation.
4. R. C. DE VEKEY and A. J. MAJUMDAR, (1968) in press.

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A Method for Fractographic Analyses of Nylon 66 Fibres and Some Preliminary Observations

The electron microscope has been useful in studying fracture surfaces of bulk materials. This type of study has helped determine the ultimate cause of failure and has helped the development of materials with enhanced properties [1]. However, this technique has not been extensively used to study the fracture character-

istics of small diameter yarns of synthetic polymers. The present note describes a successful technique for high resolution electron-microscopic examination of fractured 66 nylon fibres, and also presents some preliminary observations on the effect of draw ratio on this fracture surface.

The most successful method for obtaining good replicas of the fractured ends of small diameter fibres was when they were carbon coated from several different sputtering angles. This was

accomplished by using six to eight carbon sources placed at about 30° intervals around the fractured end. If fewer sources were used, the replicas often cracked during dissolution. Rotating the sample with a single source did not produce integral replicas. After the carbon was evaporated on the specimens, the yarns were dissolved in 95% formic acid. To assure absolute cleanliness of the replica, this dissolution was carried out for 24 h at 50° C. Using the optical microscope, the replica was floated on to a carbon grid. The grid was allowed to dry at room temperature and then was placed in the electron microscope for examination at high magnification. Several such fractographs are necessary to cover the broken surface of a single filament; these may be assembled as a montage such as shown in fig. 1 for 66 nylon. The surface is seen to consist of elongated dimples which are similar to those seen in ductile fractures of metal samples [3].

The tenacity for such fibres is shown in fig. 2 as a function of draw ratio. As is well known [4], the strength increases with the amount of deformation during drawing. An attempt was made to correlate dimple diameter with tenacity. The best fit was obtained as a linear relation of the



Figure 1 66 Nylon fractograph. Draw ratio $\times 5.8$ ($\times 9900$).

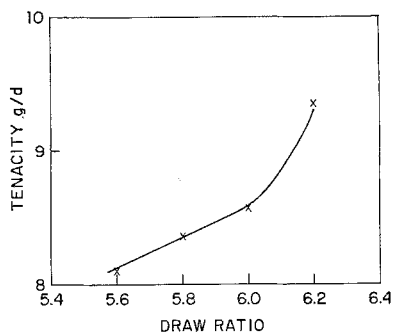


Figure 2 Tenacity versus draw ratio.

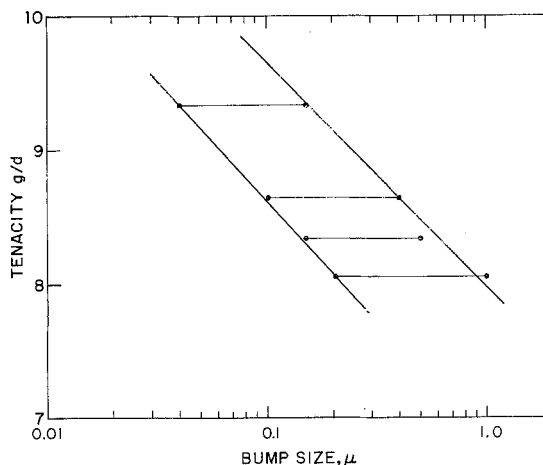


Figure 3 Tenacity versus bump size.

tenacity as a function of the log of the dimple diameter, shown in fig. 3. This relationship has precedence, as it is similar to the grain size correlations which Petch *et al* [5, 6] have observed in metal alloys. In the 66 nylon, however, the origin of the dimples and the reason for their size-dependence on draw ratio is not readily understood at this time.

Another structure is observed at highest magnification, consisting of very fine striations with a 7 μm spacing as shown in fig. 4. This striation spacing was not significantly affected by draw ratio, yet does not seem to have been an artifact. The origin of this structure is also not clear at this time. It is, however, noteworthy that these striations resemble the balls observed by Yeh and Geil [7] in polyethylene terephthalate. It would be tempting to relate it to chain folding and lamellar growth as the balls appear to be of the correct thickness [8].



Figure 4 66 Nylon fractograph. Draw ratio $\times 5.8(\times 18030)$.

References

1. "Fracture of Solids", edited by D. C. Drucker and J. J. Gilman (Interscience, New York, 1962).
2. E. A. TIPPETTS and J. ZIMMERMAN, *J. Appl. Polymer Sci.* **8** (1964) 2465.
3. W. L. PHILLIPS JR, *Trans. Amer. Soc. Metals* **56** (1963) 778.
4. N. J. PETCH, *J. and Steel Inst.* **173** (1953) 25.
5. A. CRACKNELL and N. J. PETCH, *Acta Met.* **3** (1955) 186.
6. J. HESLOP and N. J. PETCH, *Phil. Mag.* **1** (1956) 986.
7. G. S. YEH and P. H. GEIL, *J. Macromol. Sci. (Phy)* **B(2)** (1967) 235.
8. P. F. DISMORE and W. O. STATTON, *J. Polymer Sci.* **13** (1966) 133.

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The Quasibinary System $\text{SnTe}/\text{As}_2\text{Te}_3$

A search of the technical literature failed to reveal any reports of work on the Sn/As/Te ternary system. A preliminary X-ray and metallographic survey of the ternary system, conducted in our laboratories, indicated the probable existence of several isopleths which could be quasibinary in nature. These are the following pairs: SnTe/As, SnTe/SnAs, SnTe/Sn₃As₂, and SnTe/As₂Te₃. This report presents the results of an investigation of the latter system by thermal analysis, X-ray methods, and metallographic examination.

The As₂Te₃ structure was reported to be monoclinic by Singer and Spencer [1]. According to Carron [2] the lattice parameters are: $a = 14.339 \pm 0.001$, $b = 4.006 \pm 0.005$, $c = 9.873 \pm 0.005$ Å and $\beta = 95.0^\circ$. Eifert and Peretti [3] measured the melting point of As₂Te₃ at $381 \pm 0.5^\circ$ C. The crystal-structure of SnTe is the NaCl type with $a = 6.327$ Å [4]. The melting point is 806° C.

The tellurium used in this investigation was obtained from the American Smelting and

Refining Company, South Plainfield, New Jersey, and had the following spectrographic analysis: Mg, 0.0001%; Si, 0.0001%; Fe, 0.0002%; Cu, 0.0001%; Hg, 0.0002%; and 99.999+ % Te (by difference). Mallinckrodt's (St Louis, Missouri) "analytical reagent" Sn was used; it had a lot analysis of 0.005% Zn, 0.005% Pb, 0.005% Fe, 0.001% Cu, and 0.0001% As. Arsenic was supplied in lump form by the United Mineral and Chemical Corporation of New York, New York. It had a purity of 99.99+ % and was resublimed and stored under vacuum until ready for use.

The compositions As₂Te₃ and SnTe were prepared from the elements by weighing the proper amount of each element on an analytical balance and transferring the material to a quartz tube. After evacuating and sealing off the tube, it was placed in a muffle furnace controlled at a temperature which was 50° C above the expected melting temperature and held for 30 min, during which time it was vigorously shaken. The alloy was then allowed to air-cool before use. Combinations of As₂Te₃ and SnTe were prepared in a similar manner, using the