References

1. J. C. LEWIS and I. J. FLOYD, *J. Materials Sci.* **1** (1966) 154.
2. W. V. KOTLENSKY and H. E. MARTENS, "High Temperature Materials II" (AIME Met. Soc. Conf. Vol. 18) edited by G. M. Ault, W. F. Barclay, and H. P. Musinger (Interscience, New York, 1963) p. 403.
3. D. B. FISCHBACH, Jet Propulsion Laboratory Technical Report No. 32-1228 (1 March, 1968).
4. R. J. DIEFENDORF (Rensselaer Polytechnic Institute, Troy, New York, USA) private communication.

6 May 1968

D. B. FISCHBACH*
Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California 91103, USA

*On leave of absence at Department of Metallurgy, University College of Swansea, Singleton Park, Swansea, Glam, U K

Fracture Studies in Glass-Reinforced Gypsum Plaster using the Scanning Electron Microscope

Fractographs of fibre-reinforced composites are seldom published, for the simple reason that so far it has been virtually impossible to take meaningful pictures by existing techniques. With the development of the scanning electron microscope [1] many of these difficulties have been overcome and the mode of fracture of composite specimens can now be studied with high resolution. The high depth of field which this instrument provides imparts to the micrographs a marked three-dimensional quality. This is particularly important in research on fibrous materials as it enables the investigator to focus along the entire length and depth of a fibre. The ease with which fractured composite surfaces can be prepared and subsequently examined with the scanning microscope must add substantially to the attractiveness of this technique when compared to other microscope methods.

It has been shown recently [2] that a structural composite material for indoor applications in buildings can be produced by reinforcing gypsum plaster ($CaSO_4$, $2H_2O$) with glass fibres. Either α or β hemihydrate plaster ($CaSO_4$, $\frac{1}{2}$ H_2O) can be used as the starting material in the form of a thin slurry which can be sprayed simultaneously with a stream of chopped glass fibre roving on to a vacuum mould of any desired shape and size [3]. After dewatering, the material is allowed to set, and during this period the conversion of the hemihydrate to the dihydrate takes place. The resulting composite material is found to have appreciable structural strength and excellent impact strength and fibre resistance. Work is in progress to assess the suitability and economic viability of this composite material for various components in buildings such as partitions, floor-deck, staircase etc. It is foreseen that in some applications this composite material may be used as a substitute for timber.

Numerous photographs have already been taken with the scanning electron microscope (Stereoscan manufactured by the Cambridge Instrument Company) of fracture surfaces of set gypsum plaster with or without fibre-reinforcement, broken in uniaxial tension and compression, in flexure and by impact. The few micrographs presented in this communication were selected to bring out particular features. In general, the microscope was operated at 30 kV and the freshly fractured surfaces of the specimens were coated with a thin layer of carbon to make them electrically conducting.

Fig. 1 shows evidence of failure by shear in an unreinforced set gypsum plaster specimen which was broken in compression. The fault plane is clearly visible and it appears that pow-

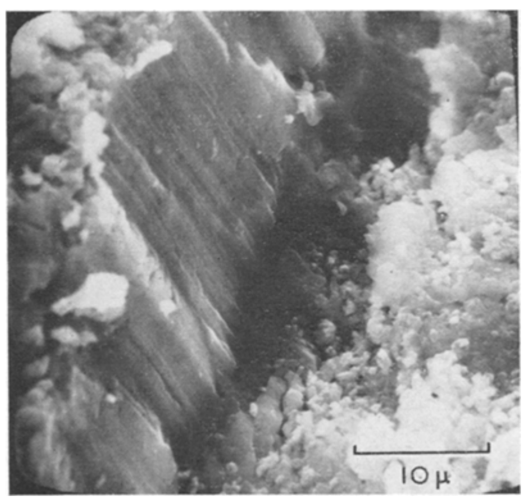

Figure 1 Shear failure in set gypsum plaster.

dered material has been transferred from one region to the other. This photograph is reminiscent of the appearance of faults in rocks. Evidence of cracks on the shear face is also seen, some of them at right-angles to the direction of faulting. Fig. 2, which is a photograph of the shear face at a higher magnification, shows indisputably that the material is badly cracked on a sub-micron scale. Fig. 3 shows a mode of fracture when the unreinforced specimen is broken in tension. A polycrystalline aggregate of gypsum appears to have been cleaved and pulled away from the fracture zone. The highly crystalline nature of the set gypsum plaster is clearly discernible in this photograph. From photographs similar to those shown in figs. 1 and 3, it is possible to form some idea about the direction of applied stresses.

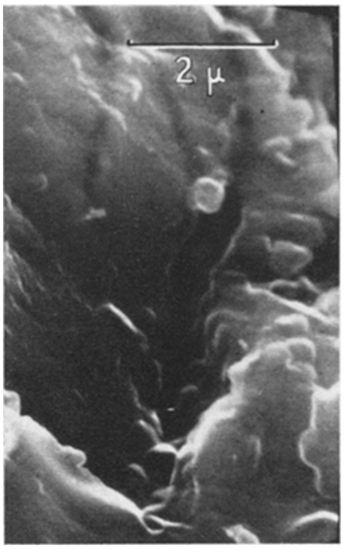

Figure 2 Shear face at high magnification.

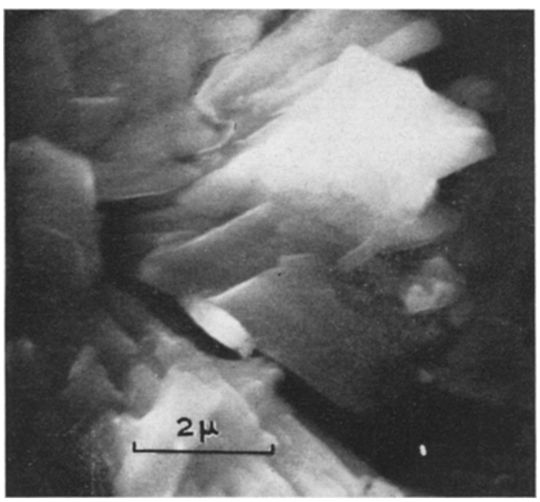

Figure 3 Tensile failure in set gypsum plaster.

Although it is possible to assign fracture modes typical of shear or tensile failure in these photographs after a close examination of large areas on the specimen, in general the evidence is not so clear cut. This is apparent in the appearance of the matrix phase shown in fig. 4 which is a micrograph of the fracture surface of a glass-fibre-reinforced specimen broken in flexure. In this photograph it can also be seen that the fibre had broken *in situ* and a portion of it had been pulled out of the matrix. Breakage of the fibre, however, is a rare occurrence. Most commonly, fibres are pulled out completely. This is clearly shown in fig. 5, which is a micrograph of a glass-fibre-reinforced gypsum plaster specimen which was subjected to a preliminary fire test and subsequently broken in flexure. The performance of this composite material in the fire test is very satisfactory, much superior to the unreinforced material. The parallel holes seen in the photograph were occupied by the fibres before the flexure test. The effect of the fire test seems to have weakened the fibre-matrix bond. Normally, the matrix phase in close contact with the fibre shows a very smooth surface but in the fire-tested specimen, this surface is badly cracked (fig. 5) and shows porosity caused by dehydration of the gypsum. This photograph also shows that even in the

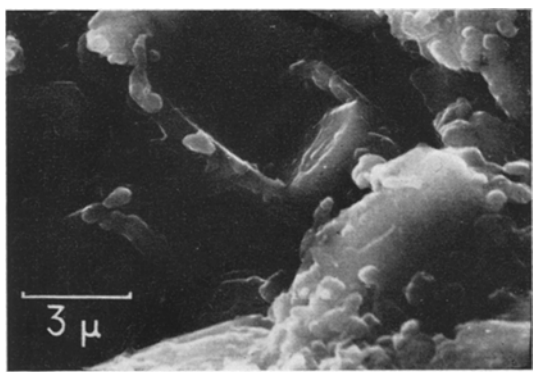

Figure 4 Glass-fibre-reinforced gypsum plaster broken in flexure showing breakage and subsequent pull-out of the fibre.

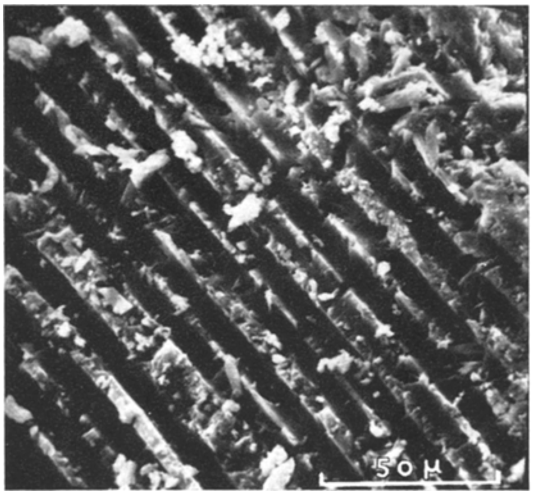

Figure 5 Glass-fibre-reinforced gypsum plaster broken in flexure after the fire test showing uniform pull-out of the fibres.

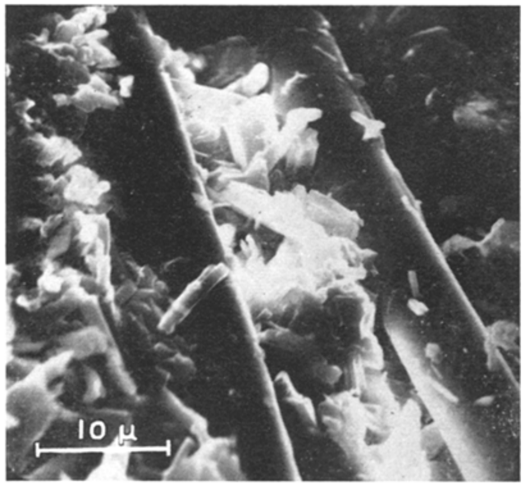

Figure 6 Bonding in glass-fibre-reinforced gypsum plaster.

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 6.76×10^6 N/m^2 (981 lb/in^2*) 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} = 7.0×10^{-2} kg/cm^2

Acknowledgement

This paper deals with work forming part of the programme of the Building Research Station and is published by permission of the Director.

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.

25 April 1968

A. J. MAJUMDAR
J. F. RYDER
D. L. RAYMENT
Building Research Station
Watford, Herts, UK

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 characteristics 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