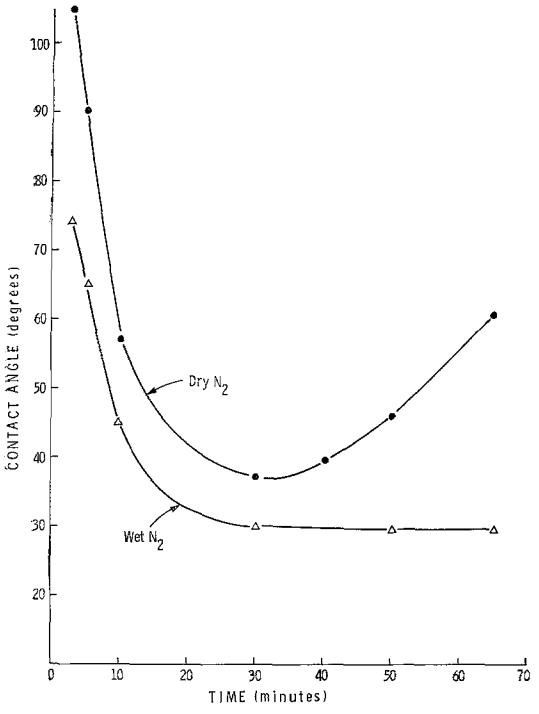

Figure 2 Wetting of borosilicate glass on evaporated thin film of chromium in wet and dry N_2 at 900° C.

in dry and wet N_2 indicate that introducing a wet atmosphere is an effective way of lowering and maintaining the wetting angle. Dry nitrogen-containing atmospheres, on the other hand, seem to raise the wetting angle with prolonged time. The mechanism contributing to this increase in wetting angle is not known for certain, but it coincides with the development of mostly nitrides and a small amount of oxides in the surface of the film [5]. Whereas a continuous layer of Cr_2O_3 is always present in the surface of the film in wet atmospheres, both oxides (oxides having been formed from 3 to 5 ppm O_2 impurity in N_2) and nitrides are present in dry atmospheres. This implies that chromium oxides contribute to better wetting than nitrides. Unfortunately, no data are available to prove or disprove the effect of chromium nitrides.

It appears that water vapour lowers the wetting angles in both glass-ceramic and glass-metal systems. The mechanism by which this occurs is not known for certain, but it appears to be largely due to lowering surface energy of glass by water vapour.

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Fibre orientation distribution in short fibre reinforced plastics

A knowledge of the fibre orientation distribution is an essential pre-requisite in seeking to predict the deformation behaviour of a short-fibre reinforced plastic. For asbestos fibres, wide-angle X-ray diffraction techniques have been applied with some success to the determination of the variation of fibre orientation distribution with position in an injection moulding [1]. This powerful technique cannot be applied in the important area of short glass-fibre reinforced thermoplastics and alternative techniques must be sought.

The texture that is often visible in mouldings when examined in transmitted light or by (macro) radiography is not necessarily related to the orientation of the well-dispersed fibres that are primarily responsible for the enhancement of the mechanical properties. Such texture or flow markings have been found to be misleading when predicting mechanical response [2].

Examination of individual fibre orientation has been widely performed using microtomed sections or surfaces prepared by the metallographic polishing technique. The latter technique is far more widely used since it is not restricted to translucent mouldings and causes less damage to the fibres and less disruption of their orientation. It is, however, a time consuming and laborious task with the results sometimes disappointing due to lack of contrast between the fibre and matrix.

These difficulties are removed by the use of the technique of contact micro-radiography (CMR) [3] which has been extensively applied to biological materials for 20 years. CMR is a modified radiographic technique in which the specimen thickness is reduced in order that the number of images cast does not confuse the total picture. A fine grain film and developer are used to enable enlarged images of the nominal 10 μm diameter fibres to be made without problems of grain size. Finally, the geometry of the system for CMR is chosen so that the fibre shadow is sufficiently well resolved to be clear on the final enlargements.

The resolution obtainable is controlled by the film grain size and the width of the penumbra [3]. The Kodak High Resolution Plate employed has a grain size of 0.25 μm which is near the resolution limit of the optical microscope, permitting magnification of × 500 without difficulty. In the worst case of a fibre lying on the free surface of a 100 μm thick specimen in contact with the emulsion, the penumbra width of the fibre image may be made less than the film grain size by setting the focus-to-film distance at 50 cm. If, instead of a normal focus X-ray tube, a fine focus tube is used, this may be reduced to 25 cm.

The contrast between the image of the fibre and the background matrix depends upon the

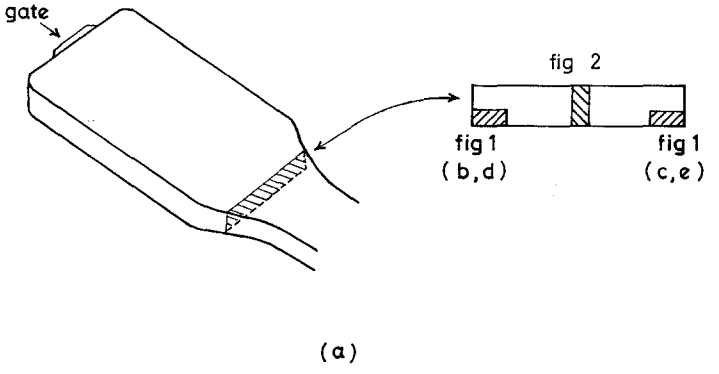
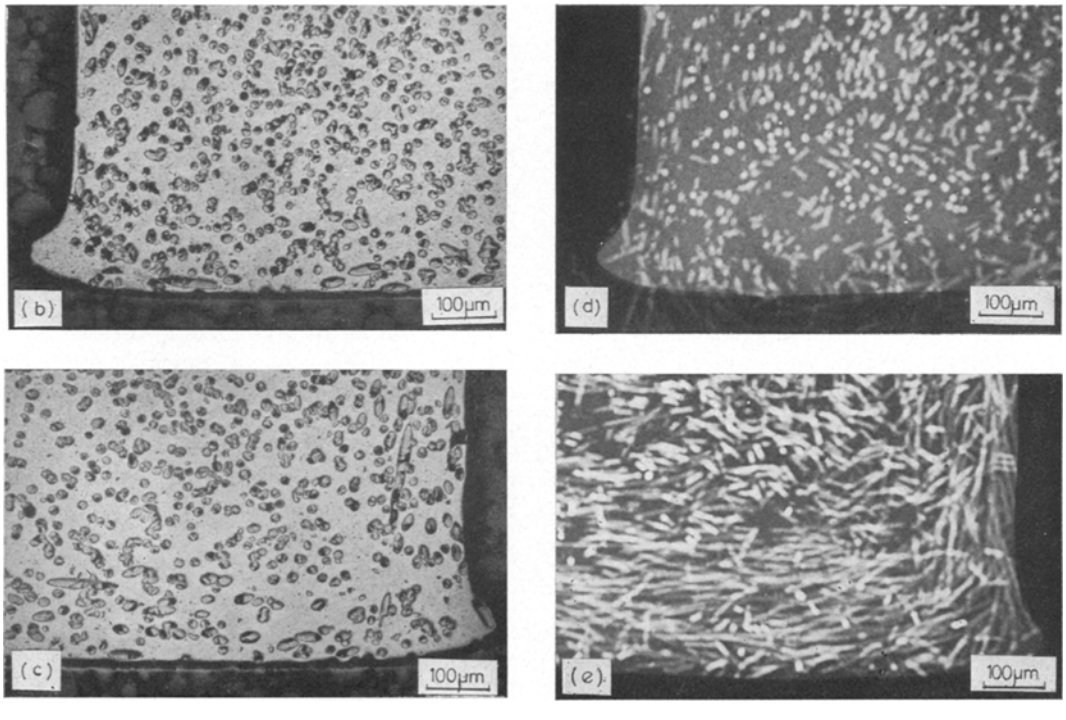


Figure 1 Fibre orientation distribution in an injection moulded ASTM tensile bar of glass fibre reinforced nylon 66. (a) Location of section cut from bar and areas of section presented in accompanying figures. (b) and (c) Photographs of two parts of the surface as prepared by metallographic polishing. (d) and (e) Photograph produced from the micro-radiograph of the section, corresponding to the parts illustrated in (b) and (c).



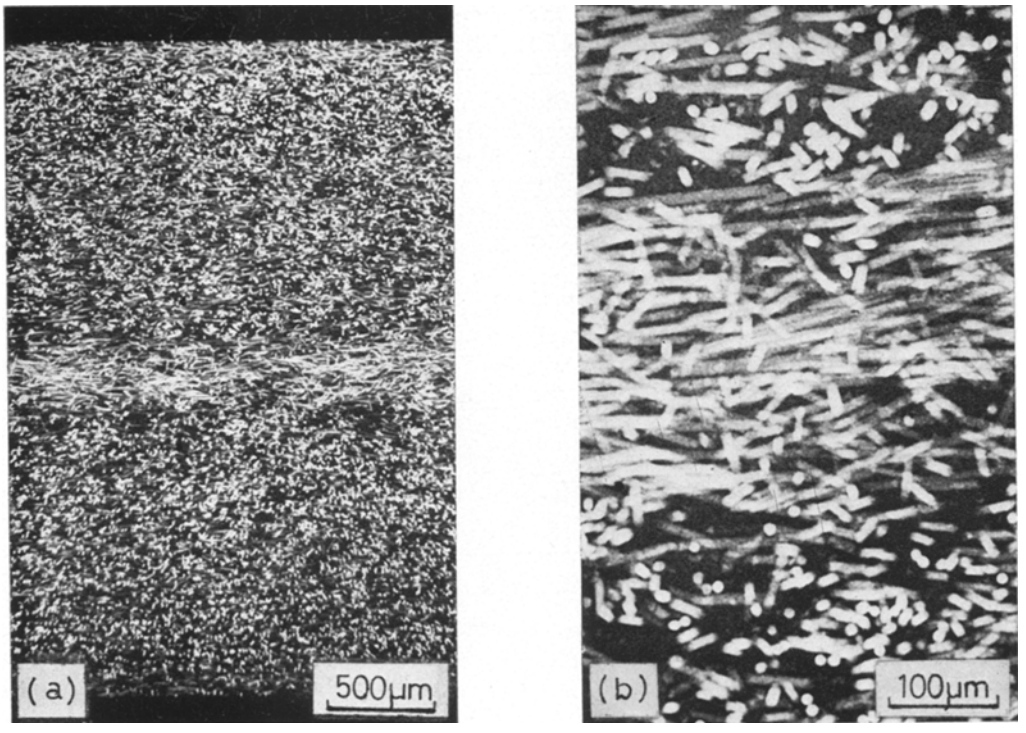


Figure 2 Variation of fibre orientation through the thickness direction for the moulding shown in Fig. 1, determined using the CMR technique. (a) View from top to bottom surface (the approximate site of this photograph on the micro-radiograph of the section is shown in Fig. 1a). (b) Part of the central region of Fig. 2a at higher magnification.

thickness of the fibre in the direction of the X-ray beam and the difference between the X-ray absorption coefficients of the fibre and matrix. Since this difference increases with X-ray wavelength, the softer radiations are more desirable. In practice, however, unfiltered copper radiation is found to give satisfactory results and the shortest exposure times.

The optimum specimen thickness for CMR varies with fibre content and the orientation of the fibres in the section. Usually it lies in the range of 50 to 150 μm. The surface condition is relatively unimportant; the only requirements are that the fibre orientation is not disturbed by the cutting process and that the cutting debris is removed. Such specimens are easily prepared using a low-speed diamond cutting saw.

As a comparison of the CMR technique with the conventional sectioning and polishing method, an injection moulded ASTM tensile bar of glass reinforced nylon 66 has been mounted and polished on the plane shown in Fig. 1a. Two micrographs of the surface are shown in Fig. 1b and c. A 100 μm slice was then removed parallel to and including the polished surface and examined by the CMR technique. Fig. 1d and e are then the identical fields of view to those of Fig. 1b and c, except that the fibres seen are not only those that penetrate the polished surface but also all those underneath the surface to a depth of 100 μm. The clearer representation of fibre orientation distribution by the CMR technique is apparent. An unexpected lack of symmetry in the flow patterns in the two corners of the mould is shown much more clearly by the CMR technique.

The CMR technique is extremely useful for examining the through-thickness fibre orientation distribution in mouldings, as illustrated in Fig. 2. This represents a section near the centre of the slice identified in Fig. 1a. The relatively high degree of fibre alignment along the axis of the bar, and the transverse orientation in the central zone are readily apparent.

A micro-radiograph of a slice from a commercial moulding in pigmented short glass fibre

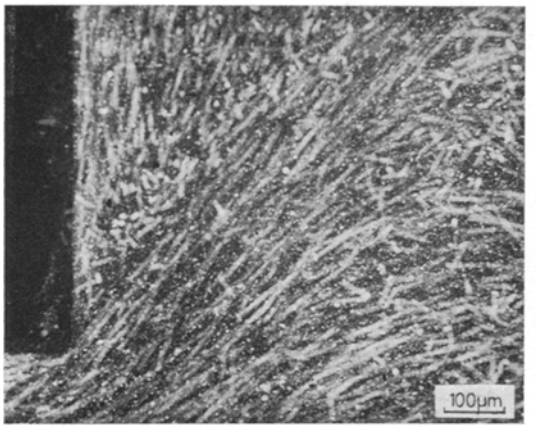


Figure 3 Part of a micro-radiograph of a slice cut from a commercial injection moulding produced in pigmented, short glass fibre reinforced thermoplastic polyester.

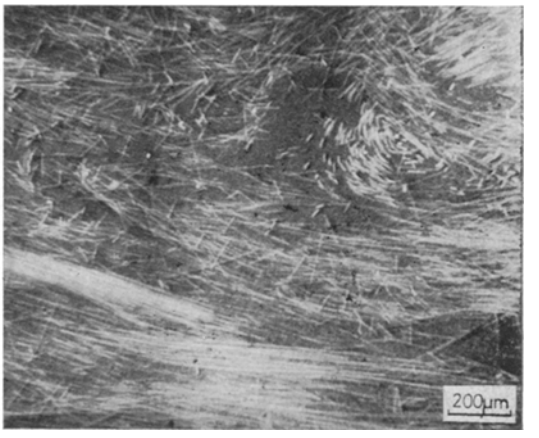


Figure 4 Part of a micro-radiograph of a slice cut from a commercial moulding in a dough moulding compound.

reinforced thermoplastic polyester is presented in Fig. 3. The flow around a corner is shown with the turbulent eddy flow region on the downstream side of the corner. It is apparent that the observation of the fibre distribution is not hindered by the presence of the pigment. Furthermore, the radiograph enables the dispersion of the pigment particles to be examined as previously reported for un-reinforced polymers [4]. In another region of the same radiograph, the meeting of two flow fronts was clearly visible.

Fig. 4 shows an example of a dough moulding compound. The high percentage of filler material, and the presence of high atomic number additives has not interfered with the observation of the fibre distribution, which is seen to be far from uniform in dispersion or orientation. The technique has also been applied successfully to sheet moulding compounds.

In addition to the clearer representation of orientation, the CMR technique is less time-consuming since the specimen preparation by diamond saw is straightforward and semi-automatic. Although a significant volume of the sample is imaged, all fibres lying through the thickness of the slice are projected in focus, onto the plane of the photographic film. This ensures that in subsequent examination of the film by high magnification optical microscopy, there are no depth of field problems as are encountered in the examination of microtomed sections.

The use of a slice (of known thickness) whose thickness is significantly less than the mean fibre length suggests the possibility of estimating, from the radiograph, the orientation of the fibres to the plane of the slice (in addition to the obvious determination of the orientation in the projected plane). The three-dimensional orientation distribution through the thickness of a moulding could then be assessed from a radiograph of one slice cut perpendicular to the moulded surface. However, unless the fibre distribution is symmetrical about the plane of the slice, the angle of the fibre to the plane of the slice cannot be determined unambiguously from one such radiograph.

The use of stereo-CMR [3] may overcome this problem. Alternatively, the examination of several slices cut at a variety of angles from the moulding could be undertaken. In principle, both techniques offer the possibility of obtaining quantitative information on the three-dimensional fibre orientation distribution through the moulding. This specification would then be equivalent to the X-ray pole figure study on an asbestos reinforced polymer mentioned above.

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