JOURNAL OF MATERIALS SCIENCE 10 (1975) 393–398

# Influence of fillers on the stiffness and strength of a polyester resin

R. M. OGORKIEWICZ, P. E. R. MUCCI*
*Mechanical Engineering Department, Imperial College of Science and Technology, London, UK*

Stiffness of a dough moulding compound containing a typical amount of calcium carbonate and glass fibres was found to be little higher than that of the polyester resin filled with the same amount, by weight, only of calcium carbonate but considerably lower than that of the resin containing the same quantity of glass fibres. Ratios of the moduli of the filled to the unfilled resin were as high in shear as in bending and the creep in bending indicated that the effectiveness of the fillers in stiffening the resin was maintained or even increased with time under load. Inclusion of calcium carbonate alone in the resin significantly reduced the stresses and even more the strains at which it failed, in contrast to the fibres, but in combination with the fibres resulted in the highest work to failure.

## 1. Introduction

The mechanical properties of polyester dough moulding compounds account for many of their technological applications but, in spite of this, they have received less attention than those of other plastics materials. As a result, there is a need for further study of such problems as that of the influence of fillers on their stiffness and strength.

Earlier work by one of the authors indicated that much of the improvement in the stiffness of the compound over that of the base resin was due to the filler [1]. On the other hand, the filler did not increase the strength of the resin, and the improved strength of the compound was attributable to its other constituent, namely glass fibres.

To help clarify the relative influence of filler and glass fibres on the stiffness and strength of dough moulding compounds the authors investigated the mechanical properties of a typical compound together with those of its base resin, by itself and in combination with the same amounts of filler or glass fibres alone.

## 2. Materials and specimen preparation

The materials used by the authors in their investigation were based on a polyester resin of the orthopthalic type. This was moulded by itself or combined either with a calcium carbonate filler or with glass fibres, or with both at once. When both fibres and filler were added to the resin their nominal proportions, by weight, were fibres:filler:resin = 1:2:2; which corresponds to the composition of a typical dough moulding compound. When fibres or filler were added by themselves their proportion was increased to equal that of them put together into the dough moulding compound, so that the proportion of the resin to that of the other constituent remained constant at 2:3, by weight.

The actual composition of the materials used was determined by ashing samples and is given in Table I. The fibres were of E-glass and had a mean diameter of 13 μm and an average length of approximately 6 mm. The calcium carbonate consisted of two grades in the ratio of 2:1, by weight, having relative densities of 2.547 and 2.595 ± 0.003 respectively, and a particle size ranging from 2 to 10 μm.

All four materials were compression moulded into 200 × 200 mm plaques having a nominal thickness of 3 mm and in all cases, except that of the resin, this was under a pressure of 7 MN $m^{-2}$ and at a temperature of 140°C. It was not found possible to hot cure the unfilled resin which was therefore moulded cold but heated subsequently for 3 h at 80°C.

*Present address: STC Ltd, London, UK.

## 3. Experimental approach

Because some of the materials to be investigated were known to be very brittle and difficult to machine and also because it was suspected that they might not be homogeneous, the authors decided to base their investigation on three-point bending tests rather than uniaxial tensile tests used for the study of other reinforced plastics [2, 3]. This made it possible to use larger, 10 mm wide, 3 mm thick specimens, which were 200 mm long so that they could be supported over a span of 150 mm. The decision to rely on three-point bending tests was further influenced by the close correlation obtained between them and tensile tests by the authors as well as others [4, 5].

The three-point bending tests were performed in a machine specially designed for the testing of plastics in bending which incorporates a system for automatically recording deflections measured by means of superlinear capacitive transducers, or on rigs incorporating identical specimen supports and a similar system of specimen loading, as well as identical instrumentation [4, 6].

All the tests were done in a controlled environment at a temperature of 20 $\pm \frac{1}{2}$° C and a relative humidity of 60 $\pm$ 5%. The test materials had been stored for several weeks in the same environment before being tested.

## 4. Deformational behaviour

### 4.1. 100 sec creep moduli in bending

To establish the stiffness of the materials under loads of short duration, specimens of them were loaded using an interrupted step-loading technique analogous to that widely used to obtain 100 sec isochronous tensile stress-strain curves [7]. This produced 100 sec isochronous load-deflection plots, which were in fact linear and from which 100 sec moduli could be calculated using the standard solution to the central deflection of a beam in three-point bending.

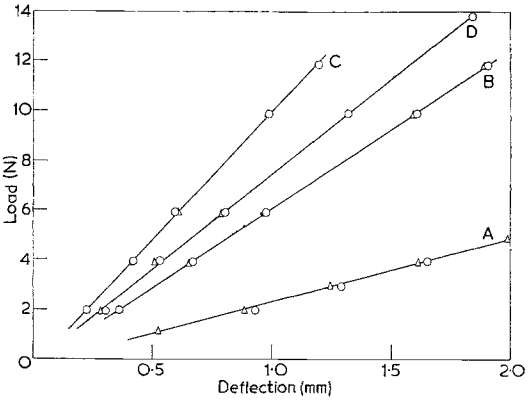


*Figure 1* 100 sec isochronous load-deflection curves for the four materials listed in Table I.

Typical results obtained with the four materials are illustrated in Fig. 1 and the average values of the moduli, based on the results obtained with three or four specimens, are given in Table I together with the maximum deviation from the mean. It is evident that the resin filled with calcium carbonate alone is almost as stiff in bending as the dough moulding compound. This is not very surprising in view of the fact that the modulus of calcium carbonate lies in the region of 60 GN m$^{-2}$ [8] which is not very different from the tensile modulus of E-glass (76 GN m$^{-2}$, [9]). The higher modulus of the resin combined only with glass may be ascribed not only to the difference between the moduli of the calcium carbonate and glass but also to the fact that fibres increase the tensile (and therefore bending) stiffness more than low aspect ratio particles [2]. As before, the relative stiffness of the different filled materials is best brought out in terms of the ratio of their moduli to that of the unfilled resin, or stiffness factor or ratio, which is given in Table I [10].

The variability of the results was due largely to inter-plaque differences, except in the case of the dough moulding compound (material D), where

TABLE I Composition and stiffness parameters in bending of the materials tested

| Material | Average composition (wt %) | | | 100 sec modulus | | Stiffness ratio |
|---|---|---|---|---|---|---|
| | Resin | Filler | Glass | Average (GN m$^{-2}$) | Maximum variation ± % of mean | |
| A | 100 | 0 | 0 | 3.28 | 6.1 | 1.00 |
| B | 38.2 | 61.8 | 0 | 8.01 | 0.4 | 2.44 |
| C | 34.4 | 0 | 65.6 | 13.7 | 10.2 | 4.18 |
| D | 37.7 | 40.4 | 21.9 | 9.26 | 3.9 | 2.82 |

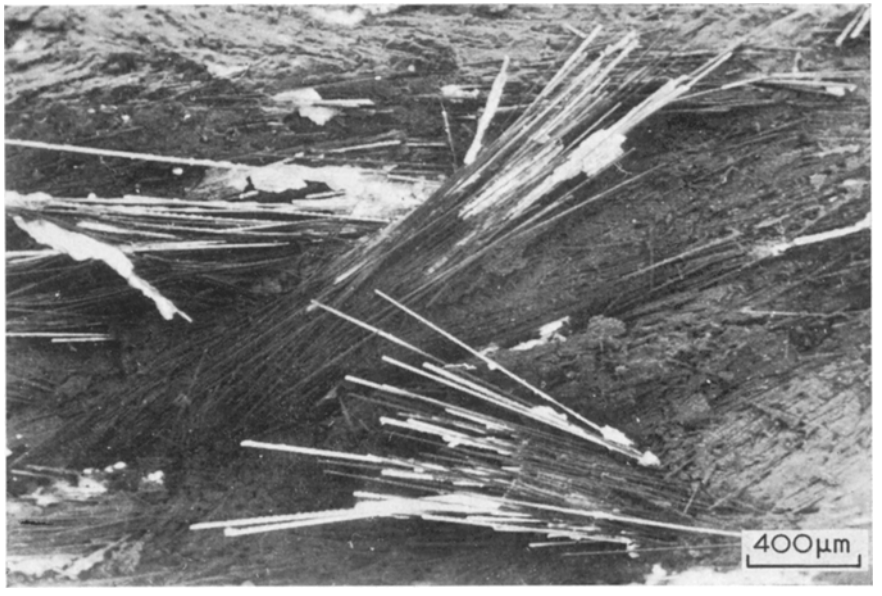


*Figure 2* Scanning electron micrograph of a fracture surface in a specimen of a dough moulding compound which shows the non-uniform distribution of glass fibres.

the variation was the same within a plaque as between plaques. In the case of the resin (material A) and the fibre-resin combination (material C), the maximum variation for specimens from the same plaque was only one third of that for the specimens from different plaques. Thus, with specimens from the same plaques the greatest variability occurred with materials C and D, both of which contained fibres and it could, therefore, be ascribed to the non-uniform dispersion of the fibres illustrated in the scanning electron micrograph of a fracture surface in a specimen of the dough moulding compound shown in Fig. 2.

### 4.2. 100 sec shear modulus

In addition to subjecting specimens cut from them to three-point bending, whole plaques were supported at diagonally opposite corners and loaded equally at the other two. This made it possible to calculate shear moduli in the plane of the plaques from the plots of the central deflections of the plaques versus load [1, 11, 12]. The deflections were measured by means of the same super linear capacitive transducers as used in the three-point bending tests and, as before, an interrupted step-loading procedure was followed to obtain 100 sec isochronous load-deflection curves.

The values of the moduli for the four materials considered are given in Table II. They show that the calcium carbonate and glass fibres stiffen the resin in much the same way in shear as they do in bending. In fact, the stiffness ratios were somewhat higher in shear than in bending. This is in keeping with the results obtained earlier by one of the authors [1, 13, 14] and provides further evidence to contradict the earlier belief that the shear stiffness of reinforced plastics was no greater than that of the unreinforced resins [15].

TABLE II 100 sec moduli and stiffness ratios in shear.

| Material | Modulus (GN $m^{-2}$) | Stiffness ratio |
|---|---|---|
| A | 1.27 | 1.00 |
| B | 3.38 | 2.66 |
| C | 5.70 | 4.49 |
| D | 3.80 | 2.99 |

The values of the shear modulus in Table II are the averages of the results obtained in the case of each material with two plaques. However, except for C, the results were virtually identical and in the case of C the variation was $\pm$ 2% of the mean, which is significantly less than in three-point bending. This might have been due to the much larger size of the specimens used to determine shear moduli, which reduced the possible effect of local inhomogeneities.

### 4.3. Creep in bending

The creep of all four materials was also investigated by subjecting specimens to constant loads in three-point bending for periods of up to $10^6$ sec (approximately 11 days) using the same apparatus as that from which 100 sec data were obtained. In each case specimens were subjected to three different loads, the loads being selected to give three sets of creep curves, each set starting from approximately the same central deflection and, therefore, since the specimens were nominally identical, approximately the same skin strain.

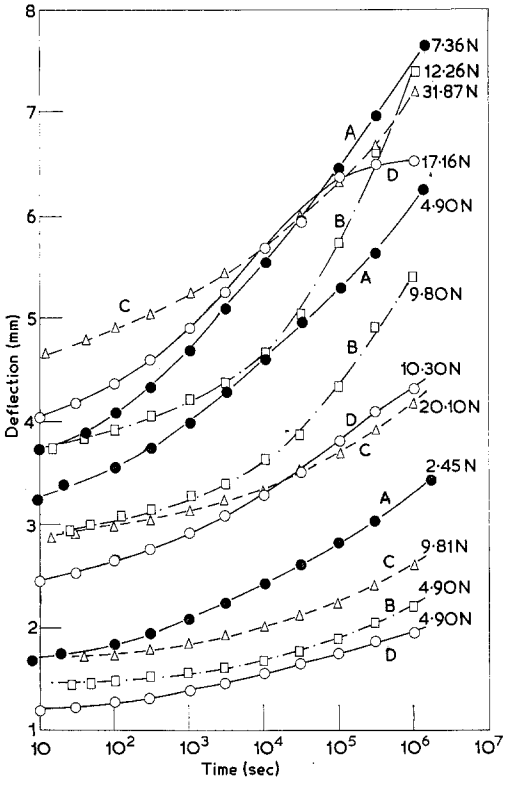


*Figure 3* Creep in three-point bending of the four materials listed in Table I.

The results are shown in Fig. 3. It is evident that all four materials creep but to somewhat different extents, so that the rate of decrease of their moduli with time increases in the order C, D, B and A. In other words, the resin with fibres alone crept least and the unfilled resin most, which was to be expected. However, decreases in the average values of the moduli between $10^2$ and $10^6$ sec ranged only from 32 to 40%.

The most significant result of the creep tests is that the stiffness factors at $10^6$ sec are higher than at $10^2$ sec, even in the case of the resin filled only with calcium carbonate, in which case it is 2.6 whereas in the case of the resin filled with fibres it rises to approximately 5.6. This means that, while the visco-elasticity of the resin causes all four materials to creep, both the filler and the fibres maintain or even increase the relative stiffness of the filled materials B, C and D, at least for durations of loading of up to $10^6$ sec. Similar effects have already been noted by one of the authors in the case of reinforced polypropylene and a glass fibre-epoxy laminate [3, 10] and are of considerable practical interest as they indicate that reinforced plastics retain their advantage of greater stiffness relative to unreinforced plastics for considerable periods of time under load.

## 5. Strength characteristics

To establish the relative strength of the materials under investigation, the authors tested specimens of them to failure in the bending test machine referred to earlier but with its dead weight loading replaced by a container filled with water at a known, constant rate. In consequence the specimens were loaded at a constant rate of 0.235 N $sec^{-1}$ and the load together with the resulting specimen deflections was recorded by the test machines data logging system at 1.96 N intervals.

The time scale of the tests ranged from 80 to 250 sec and the initial slopes of the recorded load-deflection curves correlated closely with the 100 sec isochronous load-deflection curves obtained earlier. Additional specimens were tested to failure in an Instron TM-SM-L universal testing machine at a cross-head speed of 0.42 mm $sec^{-1}$, using the same specimen supports as before, and the results obtained were found to be comparable with those in the bending machine.

Five or six specimens were tested of each of the four materials and the average load-deflection curves are shown in Fig. 4, while the average loads and deflections at failure are listed in Table III, all obtained at, or corrected to, the same specimen cross-sectional dimensions.

It is evident from Fig. 4 that while the addition of the filler to the resin increases its stiffness, it reduces the load, and therefore stress, at failure and even more the deflection at which the specimens fail. In consequence, the work done to failure of the resin filled only with calcium

TABLE III Strength parameters of the materials tested in bending

| Material | Load at failure | | Average deflection at failure (mm) | Area under curve (mm N) | Failure load ratio | Work done ratio |
|---|---|---|---|---|---|---|
| | Average (N) | Maximum variation ± % of mean | | | | |
| A | 23.17 | 21.2 | 19.6 | 241 | 1.00 | 1.00 |
| B | 16.82 | 22.5 | 5.1 | 43 | 0.73 | 0.18 |
| C | 51.85 | 9.5 | 10.1 | 287 | 2.24 | 1.19 |
| D | 41.83 | 8.6 | 13.6 | 330 | 1.81 | 1.37 |

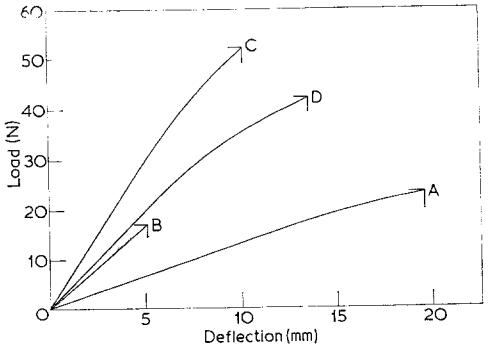


*Figure 4* Load-deflection curves to failure obtained under a constant rate of loading with the four materials listed in Table I.

carbonate (material B), which is equal to the area under the load-deflection curve, is only 18% of that of the area under the resin (A) curve, indicating a very considerable reduction in toughness.

The addition of glass fibres to the resin more than doubled the average load at failure but simultaneously almost halved the average deflection to failure and, as a result, the work to failure of material C increased by only a moderate amount compared with that of the resin. Specimens of the dough moulding compound (material D) failed at lower loads than those of resin with fibres but at a higher deflection than the latter so that their work to failure was higher not only than that for A but also for C.

The actual values of the ratios of the load at failure and the work done to failure are given in the last two columns of Table III. It should be noted that they are based on average values and, in view of the variability of the results, the values should not be regarded as absolute but rather as indicative of the relative strength and toughness of the materials considered.

## 6. Conclusions

The comparative study of a polyester resin filled with calcium carbonate or glass fibres, or a combination of both, has shown that much of the increase in the stiffness of a typical dough moulding compound over that of the resin is due to the calcium carbonate filler.

The increase in the moduli of the filled over the unfilled resin was found to be at least as high in shear as in bending and the relative improvement was maintained under loads applied for up to $10^6$ sec.

In contrast to its beneficial effect on the stiffness of the resin, calcium carbonate reduced its strength and even more its deflection to failure, so that the work done to failure of the calcium carbonate-filled resin was considerably less than that of the unfilled resin. On the other hand, the combination of calcium carbonate with glass fibres as well as resin in proportions typical of dough moulding compounds proved to require the greatest amount of work to failure, although it failed at somewhat lower loads than the resin reinforced only by fibres.

## Acknowledgements

The authors wish to thank Mr D. Sturman of the Chemicals Division, British Industrial Plastics Limited, for his help in preparing mouldings and the Analytical Services Laboratory, Imperial College, for the help in analysing the moulding materials. They also wish to thank the Science Research Council for the financial support which made this study possible.

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Received 8 July and accepted 28 August 1974.