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Composites Science and Technology 61 (2001) 2007–2016

COMPOSITES SCIENCE AND TECHNOLOGY

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Micromechanical parameters from macromechanical measurements on glass reinforced polyamide 6,6

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Received 10 July 2000; received in revised form 3 May 2001; accepted 8 May 2001

Abstract

Many elegant techniques have been developed for the quantification of composite micromechanical parameters in recent years. Unfortunately most of these techniques have found little enthusiastic support in the industrial product development environment. We have developed an improved method for obtaining the micromechanical parameters, interfacial shear strength, fibre orientation factor, and fibre stress at composite failure using input data from macromechanical tests. In this paper we explore this method through its application to injection moulded glass-fibre-reinforced thermoplastic composites. We have measured the mechanical properties and residual fibre length distributions of glass fibre reinforced polyamide 6,6 containing different levels of glass fibre. These data were used as input for the model. The trends observed for the resultant micromechanical parameters obtained by this method were in good agreement with values obtained by other methods. Given the wealth of microstructural information obtained from this macroscopic analysis and the low level of resources employed to obtain the data we believe that this method deserves further investigation as a screening tool in composite system development programmes.

Keywords: A. Glass fibre; B. Interface; B. Interfacial shear strength; E. Injection moulding; Thermoplastic; Fibre strength

1. Introduction

There has been a rapid growth in the development and application of fibre-reinforced thermoplastic polymer composites in recent years. Parallel to this growth has been the increasing recognition of the need to better understand and quantify the micromechanical parameters which control the strucure-property relationships in such composites. The properties of thermoplastic composites result from a combination of the fibre and matrix properties and the ability to transfer stresses across the fibre/matrix interphase. Variables such as the fibre content, fibre aspect ratio, fibre strength, fibre orientation and the interfacial strength are of prime importance to the final balance of properties exhibited by injection moulded thermoplastic composites [1–17]. Fibre strength may be reduced significantly after fibre formation by damage caused during both the fibre and composite production processes [16,17]. Although there has not been any direct measurement of the residual strength of fibres in a moulded composite part, there is a growing body of indirect evidence that the strength of glass fibres may be significantly reduced by the time that they actually become the load bearing component of a composite [3,7,16–19]. The ability to transfer stress across the fibre-matrix interface is often reduced to a discussion of 'adhesion' which is a simple term to describe a combination of complex phenomena on which there is still significant debate as to what it means and how to measure it. Certainly, one of the generally accepted manifestations of 'adhesion' is in the mechanically measured value of interfacial shear strength (IFSS). However, many methods of determining IFSS exist and there is no overall consensus as to which method is best [20]. This situation is further complicated by the fact that sample preparation for many of these techniques is not optimised for use with thermoplastic matrices.

Despite the elegance of the many techniques which have been developed for the quantification of composite micromechanical parameters, these techniques have found little enthusiastic support in the industrial product development environment. It should be clear that, the more dissimilar the experimental sample must be from the final composite part the greater must be the

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0266-3538/01/$ - see front matter
PII: S0266-3538(01)00062-8

extrapolation and consequent uncertainty between the measured result and real composite performance. Furthermore, the higher the number of measurements that must be made for 'reliable' statistics, the longer and more labour intensive the measurement. Finally, the more complex and disputed the underlying theories supporting the analysis then, together with the foregoing, the less likely the technique is liable to gain acceptance in an industrial environment. There continues to be discussion and disagreement about many of these complex areas — which is healthy and acceptable in an academic environment — but gains little support in an industrial environment where time scales and resources are ever diminishing. It is unfortunate that many of these techniques are indeed viewed as time-consuming, complex, inefficient, labour-intensive, and in many cases unproven or inapplicable in 'real' systems. Consequently their application in most industrial product development programmes is rare. This leads to a classical situation where, because these methods have little support in an industrial environment, they rarely get the time and development to show their usefulness in that environment. This occurs despite the fact that even the most mundane industrial problems often require solutions that can only be obtained through a deep understanding of structure-performance relationships and microstructural analysis of composite materials. Many traditional product development strategies are reaching a level on the 'S-curve' of rapidly diminishing returns and there is a real need for user-friendly micromechanical tests to aid composites to move to the next level of development. In addition to access to such knowledge, composite product developers also need tools which can fit their toolbox and do not need a new and expensive facility to house them.

Most laboratories involved in the development of thermoplastic composites will routinely measure macroscopic composite mechanical properties such as tensile strength, and determine residual fibre length (the techniques for which have been developed by many to semi or fully automated processes). A series of papers by Bader and Bowyer [21,22] in the early seventies presented a method for deriving values for τ (the IFSS) and η_0 (a fibre orientation factor) from a simple combination of the tensile stress-strain curve and the composite fibre length distribution. It is interesting to note that, despite the recent wealth of activity in the development of micromechanical test techniques (or perhaps because of it) there has been little follow-up to these papers. In this paper we show how the original analysis can be extended to obtain another important micromechanics parameter, σ_{uf} — the average fibre stress at composite failure. We present an improved version of this macromechanical method and illustrate its application to injection moulded glass-fibre-reinforced polyamide 6,6 (PA6,6) composites.

2. Experimental

Owens Corning Cratec™ 123D-14C chopped E-glass and DuPont Zytel 101 PA6,6 were used to produce moulded composites with 0, 4, 10, 20, 30, 40% w/w glass contents. The glass bundles and pre-dried PA 6,6 pellets were dry blended to the desired glass content and compounded on a single screw extruder (2.5 inch, 3.75:1, 24:1 L/D screw). The compounds were moulded into test bars on a 200-ton Cincinnati Milacron moulding machine. Set point temperatures were 288–293°C (550–560°F) for compounding and 293–299°C (560–570°F) for moulding, at a mould temperature of 93°C (200°F). All mechanical property testing was performed at 23°C and at a relative humidity of 50%, specimens were tested 'dry as moulded'. Tensile properties were measured in accordance with the procedures in ASTM D-638, using five ASTM Type I specimens at a crosshead rate of 5 mm/min (0.2 in/min) and an extensometer gauge length of 50 mm (2 in). Flexural properties were measured on five specimens in accordance with the procedures in ASTM D-790, at a crosshead rate of 2.5 mm/min (0.1 in/min) and a span width of 50 mm (2 in). Fibre length and diameters were determined by image analysis and optical microscopy on fibre samples removed from the moulded bars after high temperature ashing. Measurement of fibre orientation was carried out on cross sections of moulded tensile bars which were cut perpendicular and parallel to the flow direction (as shown in Fig. 1). The sections were polished and a series of optical micrographs was taken systematically across the thickness of the bar. The orientation of any fibre can be determined from its elliptical profile using the Eq. (1) [23].

$$\cos(\phi) = W/L = 4A/\pi L^2 \tag{1}$$

where ϕ is the angle the fibre axis makes with the flow direction, W is the minor axis of the ellipse which should also represent the fibre diameter, L is the ellipse major axis, and A is the area of the ellipse. Either of possibilities in Eq. (1) may be used, however, it has been shown [24] that the greatest experimental error comes from the measurement of W and that the area method

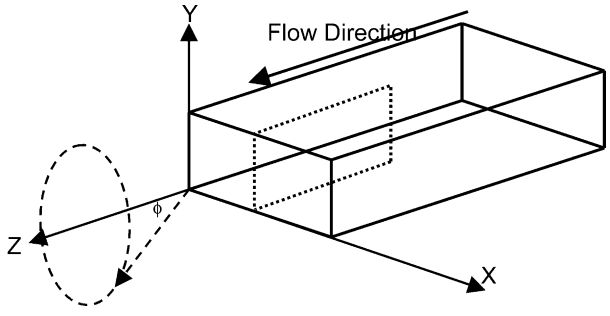

Fig. 1. Schematic view of sample preparation for optical microscopy.