

Test Methods for Chemical and Physical Characterisation of the Fibre-Matrix Interface in Composite Materials

A number of techniques for characterising the fibre-matrix interface have been investigated in the Composites Performance and Design programme. The techniques covered were:

- Atomic force microscopy (AFM)
- Dynamic contact angle analysis (DCA)
- Dynamic mechanical analysis (DMA)

Previous Measurement Notes have covered individual techniques. This Measurement Note summarises and compares the techniques.

All the techniques were found to be sensitive to variation in the fibre or interface. Comparison of the results shows that there is little correlation between the techniques. This is reasonable, given that each technique measures a different property and some of the techniques measure fibre properties rather than the interface properties.

As the characterised material more closely corresponded to the complete interface, the ranking of the fibres became closer to that expected from the descriptions of the fibre. This supports the suggestion that testing should only be performed on the complete interface, due to the complexity of relating measurements on individual components to the properties of the complete interface.

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Introduction

The range of techniques that can be used for the chemical and physical characterisation of the fibre-matrix interface in composite materials has been described in a critical review [1]. A selection of those techniques have been investigated further.

Atomic Force Microscopy

Atomic force microscopy (AFM) is a type of scanning probe microscopy that can be applied to either conductive or non-conductive surfaces. A probe tip, typically 10 nm in diameter, is mounted on a cantilever arm. As the probe follows the contours of the surface, the deflection of the cantilever arm is monitored. Surface roughness of a fibre can be determined from the resulting profile. Surface roughness is covered by two ISO standards [2, 3]. The work on surface roughness is described in more detail in Measurement Note CMMT(MN)024 [4].

Dynamic Contact Angle Analysis

The wetting of a fibre by a liquid can be characterised by the contact angle between the fibre and liquid. A smaller contact angle indicates better wetting. Dynamic contact angle analysis determines the contact angle indirectly by measuring the wetting force on the fibre. From a knowledge of the contact angle between the fibre and a range of liquids, the surface energy can be calculated. This work on wetting techniques is described in more detail in Measurement Note CMMT(MN)055 [5].

Following the release of the Measurement Note, the work on this technique has been extended. The contact angle of the fibres with vinyl-ester resin was determined, called "resin wetting" here. This approach should be closer to the complete interface as chemical, as well as physical, interactions influence the wetting of the fibre by the resin. No catalyst or accelerator was added to the resin, so the resin would not cure during testing.

Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) measures the viscoelastic properties of a material in terms of the storage and loss modulus. The ratio of loss and storage modulus, $\tan \delta$, was selected as the best indication of interfacial properties. A lower value of $\tan \delta$ indicates better interfacial adhesion since less energy is absorbed. Dynamic mechanical measurements are covered by ISO 6721 [6]. The fibres were incorporated in a vinyl ester composite for DMA testing. The work on DMA is described in more detail in Measurement Note CMMT(MN)060 [7].

Table 1. Description of glass fibres investigated

Material code	Type of size on glass fibre
Size 1	Water size
Size 2	Epoxy compatible size
Size 3	Polyester compatible size
Size 4	Complete size: polyester and epoxy compatible size

Comparison Of Techniques

Glass fibres in which the only variable was the type of surface coating were investigated. The descriptions of the fibres are presented in [Table 1](#). For each of these types of fibre the surface energy, resin wetting and dynamic mechanical properties were determined.

The results from each technique are presented in [Table 2](#). Alongside the values for each measurement is the ranking of that fibre, against the other fibres, in terms of the adhesion indicated by that measurement. It can be seen that the ranking of the fibres is not consistent between the techniques. This is because both the property and the material being measured varies. The surface energy of the fibre, adhesion of the liquid resin to the fibre and energy absorption of the complete interface are measured respectively.

However, there is a trend to the variation. As the material studied more closely approaches the complete interface, the ranking of the fibre is closer to that expected based on descriptions of the fibre sizing.

Table 2. Comparison of results from all techniques. (The fibres are ranked for each property, where 1 is the best adhesion and 4 is the worst adhesion implied by each measurement.)

Size	Surface energy	Resin wetting		DMA	
	γ_s (mJ m ⁻²)	Adv. θ (°)	Rec. θ (°)	Value (-)	FWHM (°C)
Size 1	50.6 [1]	53.0 ± 1.7 [2]	45.2 ± 1.0 [2]	0.867 [4]	15.8 [4]
Size 2	42.9 [3]	53.3 ± 0.9 [3]	45.8 ± 0.5 [4]	0.773 [3]	17.7 [3]
Size 3	41.6 [4]	53.5 ± 1.1 [4]	45.3 ± 0.1 [3]	0.522 [1]	20.8 [2]
Size 4	46.0 [2]	50.0 ± 2.2 [1]	44.4 ± 1.8 [1]	0.616 [2]	23.2 [1]

Concluding Remarks

A summary of all the techniques covered is presented in [Table 3](#). The techniques have been compared by a number of key considerations. Based on these considerations, the most useful technique appears to be DMA because it is a well-established, standardised technique which characterises the complete interface of many fibres

in a single test. A disadvantage is that this technique is sensitive to other properties of a composite material, so careful control and rigorous procedures would be required for measurements using this technique

Table 3. Comparison of techniques for assessment of the fibre-matrix interface.

	Surface roughness	Surface energy	Resin wetting	DMA
Sensitive to variations in fibre?	Yes	Yes	Yes	Yes
Well-established technique?	Yes	Yes	No	Yes
ISO standard for technique available?	Yes	No	No	Yes
Number of fibres/interfaces measured?	One	One	One	Many
Part of composite material measured?	Fibre	→		Interface
Affected by other factors?	Least	→		Most

References

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