

Characterisation of Surface Roughness of Fibres by Atomic Force Microscopy

In a composite, the development of a stable bond between the resin matrix and the reinforcing fibre depends on the efficiency of adhesion at the interface. Mechanical interaction associated with the surface roughness is one of the mechanisms involved, although the effect has not been quantified at this stage.

Some of the available techniques that give information of the physical state of the fibre surface are:

- X-ray Diffraction (XRD)
- Brunauer-Emett-Teller Surface Area Measurement
- Microscopy
 - Transmission Electron Microscopy (TEM)
 - Scanning Electron Microscopy (SEM)
 - Scanning Probe Microscopy (SPM)

The purpose of this study is to describe a method for analysing the surface texture of reinforcement fibres using Atomic Force Microscopy (AFM), a type of SPM. The technique can be used to analyse three-dimensional surface features with very high spatial resolution in a submicron size range. The images and data can be used for textural quality assessment. To illustrate the capabilities and limitations of the technique, a range of representative fibres used as reinforcements in composite materials have been studied.

This note was prepared as a result of investigations undertaken under the DTI Composite Performance and Design project on Interface Characterisation and Performance.

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May 1998

Atomic Force Microscopy

Scanning Probe Microscopy is a collective name given to a technique which consists of a probe with a measuring device to analyse the small vertical movements as it travels over a sample. As the probe scans across the sample, computer controlled feedback to a piezo-electric transducer maintains a constant interaction between the stylus and sample surface. A wide range of interactions may be used. Of these, the Scanning Tunnelling Microscope (STM) uses constant electron tunnelling current and the Atomic Force Microscope (AFM) uses constant force for the measurement.

The STM was first proposed as a method to measure forces on particles as small as single atoms. The AFM was developed as an extension, combining the principle of the STM and a stylus profilometer. It was shown to have a lateral resolution of 3 nm and a vertical resolution of less than 0.1 nm [1]. The measurement ranges achievable differ depending on resolution of the instrument and radius of the tip used and can be determined from the Stedman amplitude-wavelength diagram [2]. On this basis both STM and AFM are applicable to the study of fibre surface characteristics. However, STM can only be applied to conductive or semi-conducting materials. An AFM typically consists of a silicon or silicon nitride conical probe with a tip 10 nm in radius, mounted on a low spring constant cantilever arm. The tip follows the contours of the surface being studied and a computer monitors deflection of the cantilever to maintain a constant repulsion by feedback.

Sample preparation is very straight forward simply consisting of fastening the fibres onto a metallic disc with double-sided adhesive tape. The disc is then mounted magnetically under the probe. The sample is scanned at the chosen pixel density (256 x 256) to give a 2D array of sample heights, which can be captured and displayed as a shaded height map or as a 3-dimensional image of the real surface. The data are in digital form and can be analysed as required at different cross sections. In this work each scan yielded two images, one taken from left to

right and the other from right to left. A third order polynomial was then subtracted from the original data to remove the form and leave the higher spatial frequencies of the surface roughness for analysis. In the present study, eight line profile measurements were made on each scan and the height profiles were used to determine the roughness parameters as defined below.

The x, y and z displacements were calibrated using a calibration sample with periodic features, a silicon etched sawtooth where the gradient of each facet is known.

Standards for Determining Surface Roughness:

Although normally applied to Workshop machined surfaces, the same standards, terms and parameters are applicable to AFM.

BS ISO 4287: 1997 [3] specifies terms, definitions and parameters for the determination of surface texture (roughness, waviness and primary profile) by profiling methods. It is a General Geometrical Product Specification (GPS) standard and a major rewrite and reorganisation of BS 6741:Part 1: 1987/ ISO 4287/1-1984. Whereas the older standards defined only roughness profile and its parameters, the new standard, along with ISO 11562 and ISO 3274 additionally covers the waviness profile, the primary profile and their parameters. ISO 4287/1-1984 referred to ISO 468 [4], which is of relevance to the present study. This standard was taken into account in BS 1134 [5] which supersedes BS 6741 Parts 1 and 2 which were accordingly withdrawn. This latter standard includes a description of the instrumentation, viz. Stylus-type measuring instruments which, in their general form, include AFM.

Terms and Definitions for a Profile:

Co-ordinate System: That co-ordinate system in which surface roughness parameters are defined. It is usual to use a rectangular co-ordinate system in which the axes form a right handed Cartesian set, the x-axis being the direction of tracing collinear with the direction of probe movement, the y-axis nominally lying on the real surface and the z-axis being in the outward direction from the material to the surrounding medium.

Real surface: Surface separating the object from the surrounding medium.

Surface profile: Profile that results from the intersection of the real surface by a specified plane. In practice it is usual to choose a plane with a normal that is parallel to the fibre axis.

Sampling length, l : The length of the reference line used for identifying the irregularities characterising the surface roughness.

Profile peaks: Outwardly directed portions of the assessed profile connecting two adjacent intersections of the profile with the x-axis.

Profile valley: Inwardly directed portions of the assessed profile connecting two adjacent intersections of the profile with the x-axis.

Peak-to-Valley Distance: This measurement gives the maximum peak-to-valley distance within the selected profile. It is given by the highest data point minus the lowest point:

$$R_{p-v} = z_{\max} - z_{\min}$$

Mean height: The mean height, $\bar{z}$ is given by the average height within a selected profile with N data points.

$$\bar{z} = \frac{1}{N} \sum_{n=1}^N z_n$$

Root Mean Squared Roughness: For a profile containing N data points, the root mean squared roughness, R_{rms} , is given by the standard deviation of the data, determined from:

$$R_{ms} = \sqrt{\frac{\sum_{n=1}^N (z_n - \bar{z})^2}{N-1}}$$

where $\bar{z}$ = mean z height.

Average Roughness: The average roughness is given by the mean deviation of the data from the average of the data. For a line containing N data points, the average roughness is determined from:

$$R_{ave} = \frac{\sum_{n=1}^N |z_n - \bar{z}|}{N}$$

where $\bar{z}$ = mean z height.

Sample Measurement:

Three kinds of fibre reinforcements typically used in composite manufacture were studied.

Carbon fibre (treated and untreated), Silicon Carbide (SiC) fibre and E-Glass fibre were scanned by AFM and the results are discussed here to highlight the capabilities and limitations of the technique.

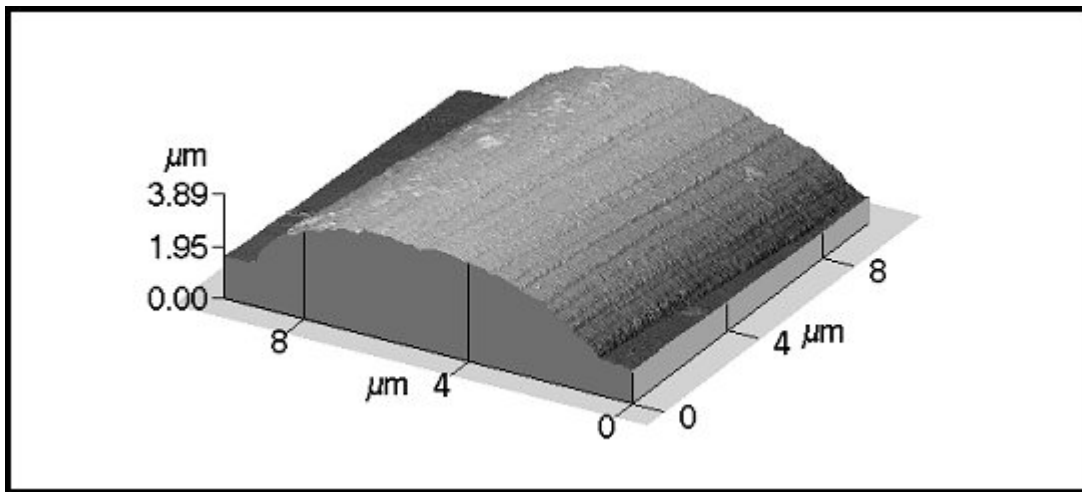


Figure 1: 3D image of treated carbon fibre

Three fibres of each kind were scanned in two directions left to right and right to left, at arbitrarily chosen positions along the length. The data was processed to remove the form of the fibre and line profiles were analysed at 8 roughly evenly spaced positions over the scan. Altogether this yielded 48 measurements of the roughness parameter for each fibre sample.

Carbon Fibre: Carbon fibre used as reinforcement in polymer matrix composite materials are generally given an oxidative surface treatment and a chemical sizing to enhance the adhesion between the fibre and resin matrix. In the present study, samples of untreated and treated fibres were analysed.

Figures 1 and 2 show the 3-dimensional images of a treated and untreated carbon fibre respectively. Figure 3 shows line profile measurement for treated carbon fibre.

Assuming the fibre to be cylindrical, the diameter of the carbon fibre can be calculated and was found to be 9 μm. The Surface Roughness measurements obtained are given in Table 1. Measurable differences were observed between treated and untreated carbon fibre surface parameters. A visual inspection of the 3D images reveals a qualitative difference between the topographies.

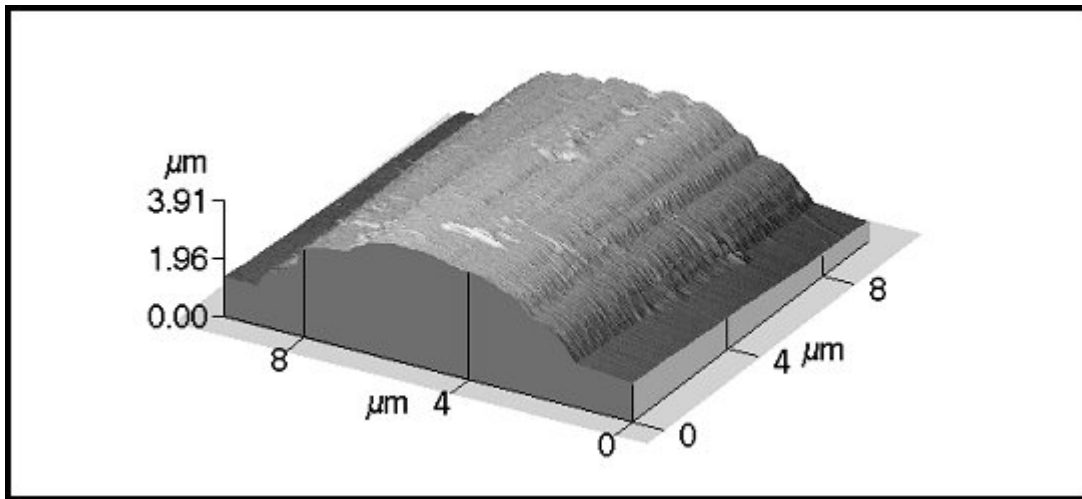


Figure 2: 3D image of untreated carbon fibre

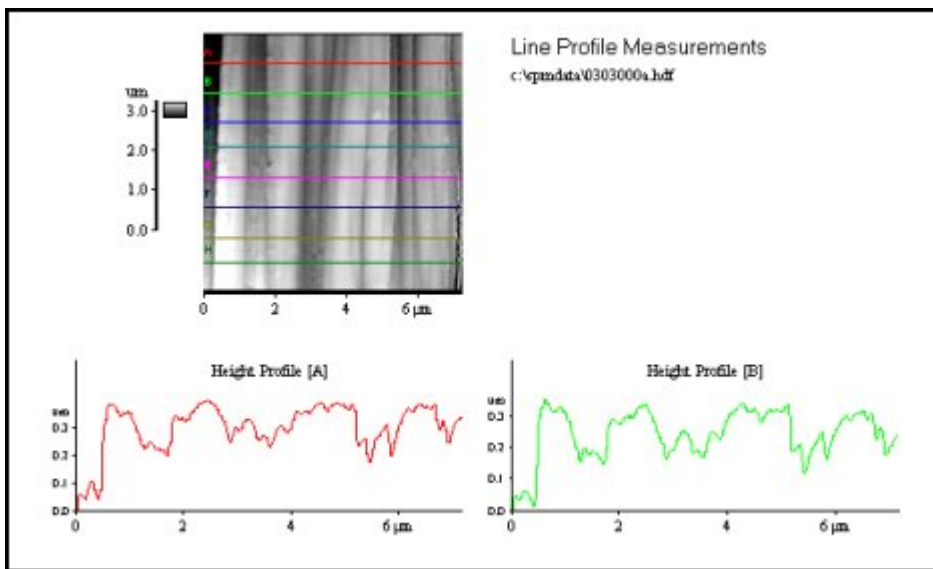


Figure 3: Line profile measurements for treated carbon fibre

The treated sample (Figure 1) has a form with low amplitude and high frequency undulation compared to the untreated sample in Figure 2 which shows a high amplitude low frequency undulation. The surface roughness parameters were also determined along the fibre axis. From the 3D image it is obvious that the line at which the profile is to be generated must be carefully chosen so that it runs exclusively along a ridge or valley. In fact, it was not possible to choose a line that ran the full length of the scan as was done in the 90° direction and shorter lines were used to determine the roughness parameters in the 0° direction. The roughness parameters for a treated fibre (axial direction) were: $R_{p-v} = 50$ nm, $R_{rms} = 13$ nm and $R_{ave} = 11$ nm.

Table 1 Surface Roughness Parameters for Carbon Fibre (Circumferential) (nm) *

	R_{p-v}	R_{rms}	R_{ave}
Treated Fibres			
Average	319	67	53
Std. Dev	±68	±11	±7
Untreated Fibres			
Average	724	142	109

Std. Dev	± 129	± 21	± 2
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* Each value is the average of 48 measurements

SiC Fibre: The 3-dimensional image of a SiC fibre is given in Figure 4.

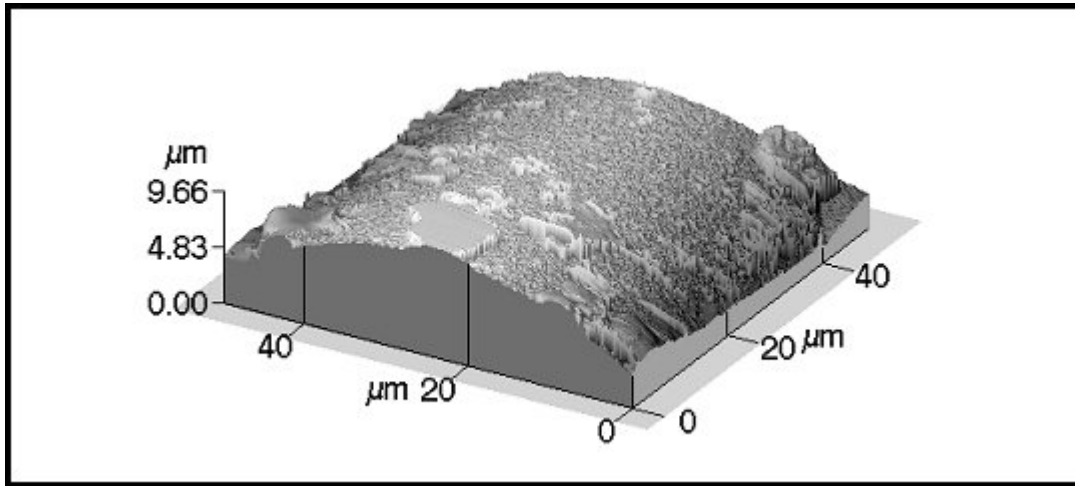


Figure 4: 3D image of silicon carbide fibre

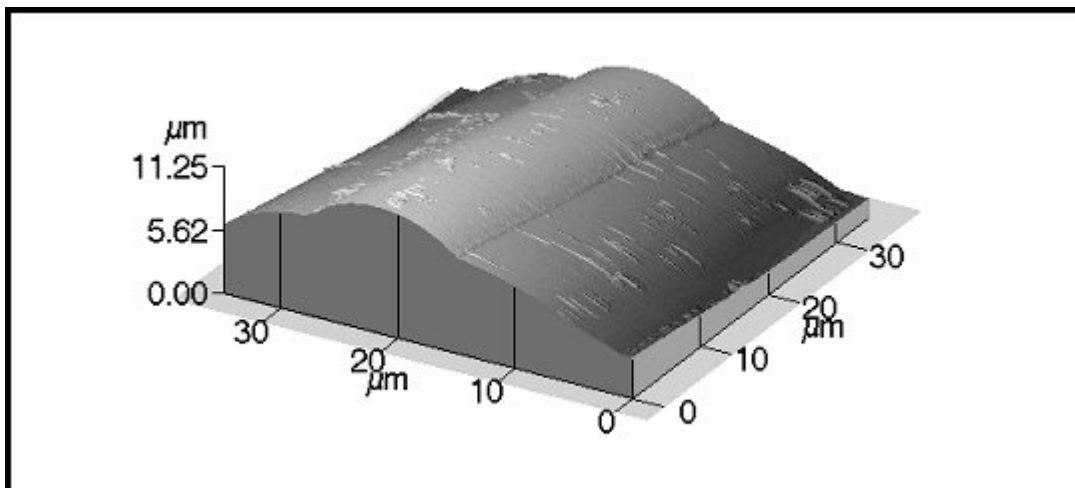


Figure 5: 3D image of E glass fibre

The image illustrates the limitations of the AFM technique. The range on x, y and z axes is insufficient to image the whole of the fibre. The flat portion at the centre front of the scan is where the instrument is out of range. It may be a particle adhering to the sample. In order to derive meaningful roughness parameters for this sample a $14\mu\text{m} \times 14\mu\text{m}$ portion of the image was chosen for analysis. Line profile measurements were done at four cross sections within this area. The resulting surface roughness parameters were: $R_{p-v} = 192\text{ nm}$, $R_{rms} = 38.6\text{ nm}$ and $R_{ave} = 31.1\text{ nm}$. It should be emphasised that these measurements are the average of fewer readings than were used in the study of carbon fibres. Also the pixel density in the square is 72×72 as compared to 256×256 in the earlier study. The pixel spacing is thus about 200 nm compared with about 40 nm in the previous case. There is an obvious qualitative difference between the SiC fibre which has a low amplitude, high frequency non-orientated roughness on a relatively smoother form compared to the carbon fibres in Figures [1](#) and [2](#).

E Glass Fibre: [Figure 5](#) is the 3-dimensional image generated of a sample of E glass fibre. At first examination, the fibre appears to have a large cross section featuring many lobes. To verify the finding, the same sample was examined by SEM, where a clump of fibres was observed ([Figure 6](#)). The appearance of the continuous multilobed contour in the AFM is probably an image of the locus of the top fibre with the cantilever arm on which the tip is attached which has contacted the surface of the glass fibres after the tip has moved down the topmost one. As in the case of the SiC fibre, a clear spot on the top fibre was identified for determination of surface roughness and the parameters were found to be: $R_{p-v} = 45\text{ nm}$, $R_{rms} = 10\text{ nm}$ and $R_{ave} = 8\text{ nm}$. These values highlight the

smoothness of the glass fibre surface and the high spatial resolution of the AFM.

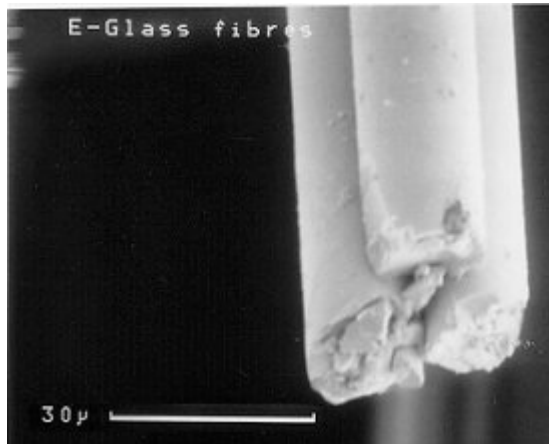


Figure 6: SEM image of E glass fibre

Conclusions and Future Work

This Measurement Note is intended to illustrate some applications of the AFM in determining surface characteristics of reinforcement fibres used in composites.

The technique was found to be valuable in the analysis of carbon fibres where the diameter of about 10 μm and roughness parameters of 100 nm make the AFM especially suitable. The roughness of the carbon fibre was shown to be much higher perpendicular to the axis than along the axis. However, it should be pointed out that the long range wavelength or undulation of the fibre beyond 10 nm cannot be determined by this technique. When scanning large diameter fibres (e.g. SiC), the z range of the instrument limits the section of the fibre that can be sampled for roughness measurements. In such cases, it is appropriate to choose a smaller area for scanning, but conclusions on surface roughness characteristics are correspondingly limited and should be treated with caution. Artefacts as seen in the analysis of glass fibre may require confirmation by other techniques to ensure that improper conclusions are not made.

Atomic Force Microscopy provides a reliable technique for deriving a quantitative measurement of roughness of small diameter fibres. It is also useful when contrasting quantitatively the roughness of the same fibre with different surface treatments. Further, it provides a means for qualitative comparison of fibres with different surface topographies. As mentioned at the outset, mechanical roughness is believed to be one of the parameters influencing interface adhesion efficiency although the degree of importance is not yet known. AFM is a technique which has been shown to be useful in analysing these effects. Further studies will be carried out to assess the correlation between these measurements and the corresponding fibre-matrix systems adhesion strength.

References

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4. ISO 468-1982. "Surface Roughness-Parameters, their values and general rules for specifying requirements"
5. BS 1134:Part 1:1988. "Assessment of surface texture. Part 1. Methods and instrumentation"
BS 1134:Part 2:1990. "Assessment of surface texture. Part 2. Guidance and general information"

BS, EN and ISO standards can be obtained from

BSI Customer Services
389 Chiswick High Road
London W4 4AL
Telephone: 020 8996 7000

Fax: 020 89967001

Acknowledgements

The authors wish to thank Dr. L.N. McCartney, Dr. N.J. McCormick and M.R.L. Gower for valuable assistance in the preparation of this measurement note.

This work is part of the *Materials Measurement* programme of research supported by the DTI.

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May 1998

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