3D Characterisation of Glass Fibres in Composites by Confocal Microscopy

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SUMMARY: The development of instrumentation to reconstruct the orientation of fibres in polymer composites has come a long way over the past ten years, mainly due to the improvements in computer-assisted microscopy and the implementation of novel microscopic techniques. In this paper, both 2D reflection mode optical microscopy and also confocal laser scanning microscopy (CLSM) applications to specific problems associated with glass fibre reinforced polymer composites are discussed. The ability of the CLSM to throw optical sections within semi-transparent materials allows us to reconstruct fibre orientations and spatial distributions over large sample regions (mms x mms x 50 μm) within reasonable timescales. Both continuous glass fibre composites and discrete, injection moulded composites are discussed and new characterisations are proposed e.g. fibre curvature, torsion and curl of the vector field. Complex vortex regions and fracture regions are now accessible to 3D reconstruction using the CLSM.

KEYWORDS: confocal laser scanning microscopy, 3D reconstruction, glass fibres.

INTRODUCTION

Many books have been written about the confocal laser scanning microscope, (CLSM) e.g.[1] and [2] and the technique has been used extensively in biological and physiological research. Over the past five years, work at Leeds has shown that confocal laser scanning microscopy (CLSM) is a technique which enables high spatial resolution, 3D reconstructions of glass fibre reinforced composites to be undertaken. At typical glass fibre packing fractions of 40% by volume, fibre orientations may be acquired down to depths of 50 μm [3].

There are many commercial designs on the market, but the Noran 'Odyssey' CLSM is one of the few with videorate image acquisition capabilities and whose functions are fully automatable by linking with a standard microcomputer. As shown in Fig. 1, an image is built up by scanning over a sample and an acousto-optic deflector is used for one of the scan directions, rather than a galvanometer. Note that the sample may be viewed in reflection mode or fluorescence mode. Fibre orientation research is best conducted in fluorescence mode[4], using very high numerical aperture (oil immersion) objective lenses in order to improve the optical sectioning.

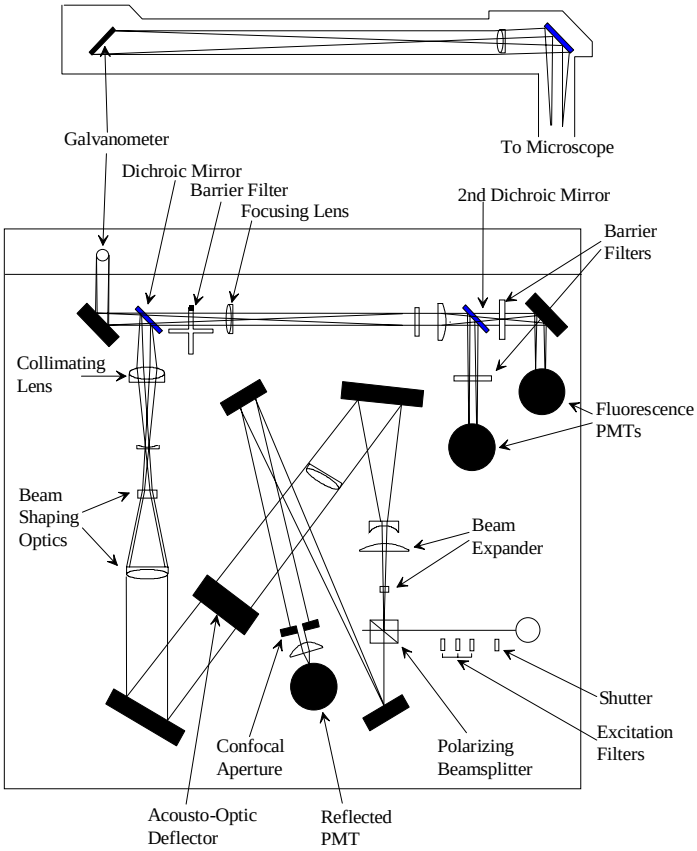


Fig. 1: Schematic diagram of the NORAN 'Odyssey' CLSM optical arrangement which may be attached to standard laboratory microscopes.

At Leeds, the Odyssey is attached to a standard Nikon microscope and an oil immersion objective, x 60, NA 1.4 lens has been used to reconstruct fibre orientations at the highest spatial resolution (approximately 0.25 μms in X, Y and Z).
The 3D reconstruction of fibre orientations is achieved in one of two ways: by taking a set of XY sections at the surface and different depths, Z within the sample and by matching fibre centre coordinates between XY planes *or* by taking a set of XZ sections at different Y positions and by matching fibre centre coordinates between XZ planes. A typical fluorescence mode, XZ section of a continuous glass fibre composite is shown in Fig. 2 and it is clear that robust software has to be written to automatically locate fibre images at different depths (because of the increasingly fuzzy images and poorer signal to noise as the depth increases). The fibres in this section were lying essentially parallel to the sample surface and aligned along the Y direction - hence the circular fibre images.

CONTINUOUS GLASS FIBRE COMPOSITES

Compression moulded, glass fibre reinforced, epoxy matrix samples

Recently, semi-automated software has been written for the Noran 'Odyssey' CLSM which allows unidirectional fibre composite volumes of typically 5 mm x 1mm x 50μm in XYZ to be analysed within one working day, as shown in Fig.3(a). The 3D reconstruction results from identifying fibre cross-sections on one hundred XZ section planes, each separated by 50 μm in

the Y direction. Note that the sample has been cut with the fibres aligned essentially parallel to the sample surface. In this way, individual fibres are followed in space for distances of mms (i.e. the fibres stay within 50 μm of the sample surface, despite their intrinsic waviness). Hence novel 3D characterisations of the fibres within unidirectional composites are possible e.g. the determination of fibre waviness [5], fibre curvature, κ and torsion, τ [6], unambiguous fibre segment orientations (as shown in Fig.3(b)), the clustering of fibres, also ply misalignments and even the 3D angular divergence of nearest neighbour fibre segments may be derived.

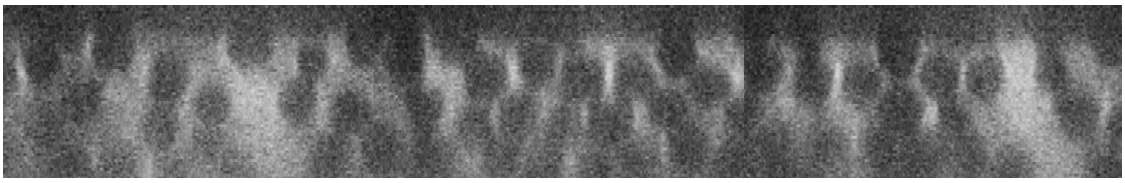
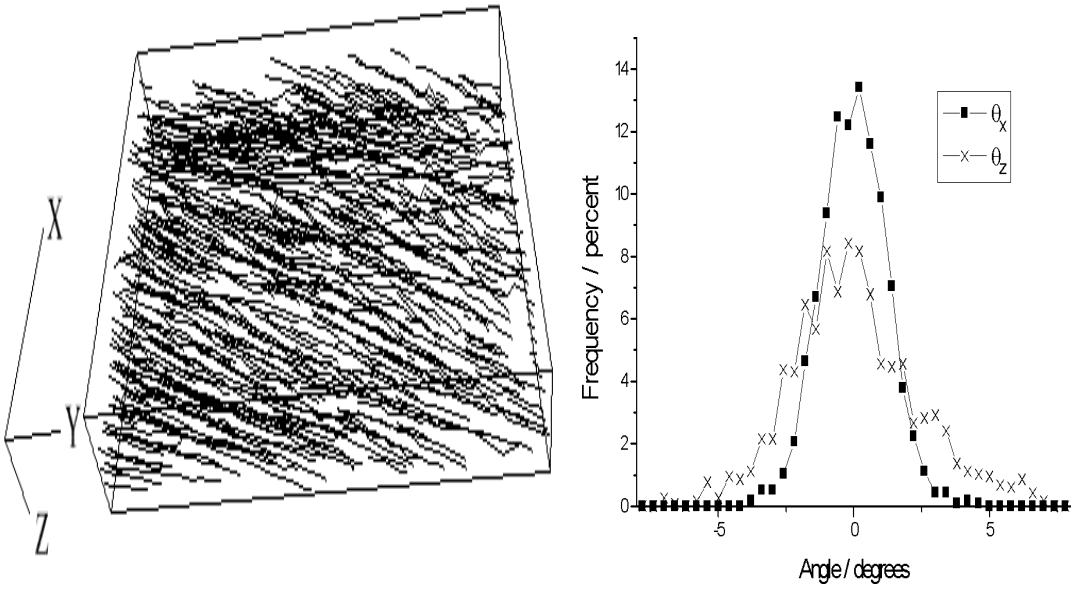


Fig. 2: A typical XZ optical section acquired by the CLSM, showing the fluorescing matrix and the circular cross-section fibre images within the sample. The dimensions of this section are 200 μm x 50 μm in XZ.



(a) (b)

Fig.3: (a) 3D reconstruction of continuous, highly aligned, fibres in a 5mm x 1mm x 50μm epoxy sample volume and, from these data, (b) the θ_X and θ_Z angular distributions of fibre segments in two orthogonal planes are plotted.

Our latest work has been to look at nearest neighbour fibres and to discover whether or not the 3D separation angles between adjacent fibres could be linked to the curious anisotropy[7], seen in the nearest neighbour, in-plane angular distribution, Φ_{NN}. A paper addressing this effect is currently in preparation[8].

Fracture of continuous glass fibre reinforced samples

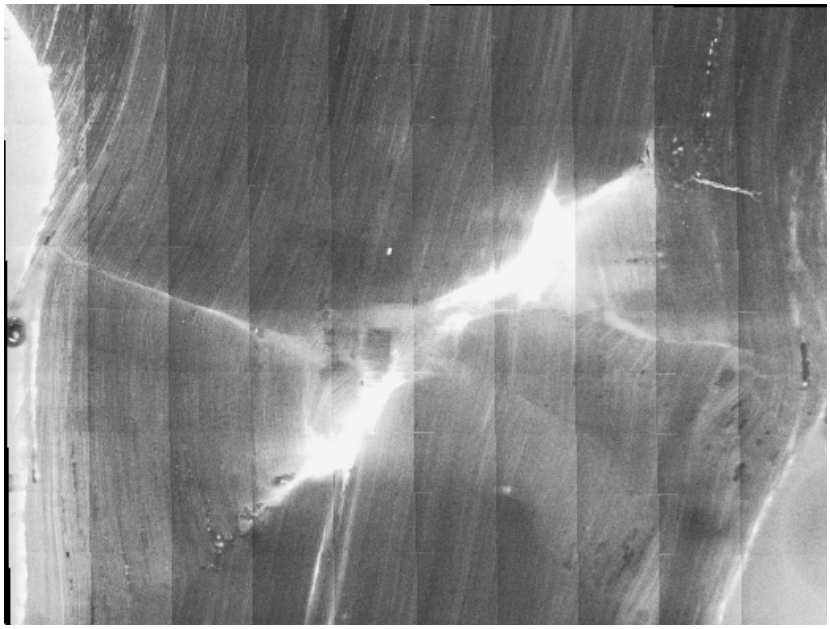
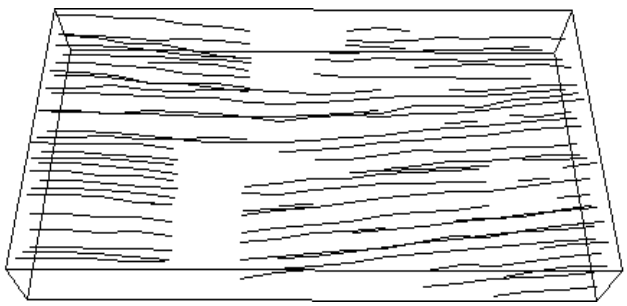
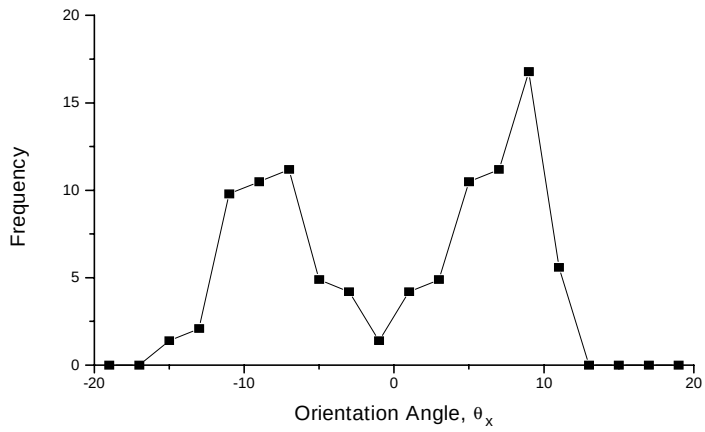


Fig. 4: Large area overview of a fracture region seen in fluorescence mode.



(a)



(b)

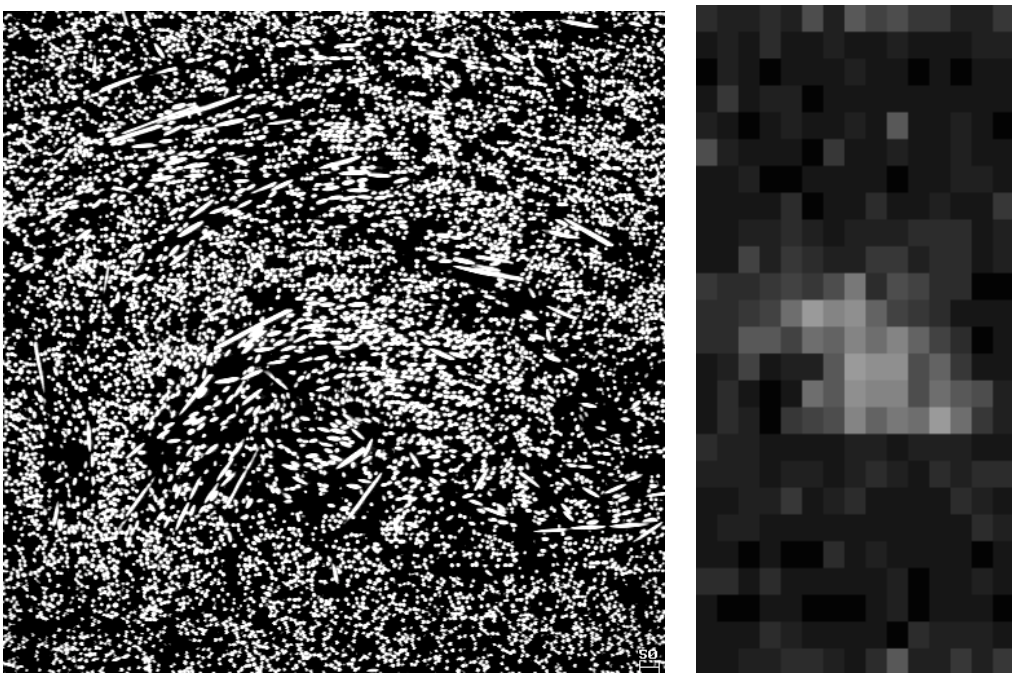
Fig.5: (a) Detailed 3D view of fibre orientations across a fracture.
(b) Frequency distribution of fibre orientations, θ_x across the fracture.

Another study concerns 3D fibre orientations across fractures in continuous glass fibre reinforced composites. As shown in Fig.4, there is considerable complexity in the region of the fracture and the first results of high spatial resolution reconstruction across a fracture line are shown in Fig. 5(a) and (b). Note the few fibres straddling the main fracture line in this region. This application area makes clear the need for rapid automation of the fibre location software, so that large regions can be efficiently mapped at greater spatial resolution.

DISCRETE GLASS FIBRE COMPOSITES

The CLSM research into continuous, glass fibre reinforced samples has been undertaken because the software problems are not as severe as those encountered for discrete fibre composites. In discrete fibre samples, the orientations will in general be more randomised and also fibres appear and disappear within the volume to be reconstructed. Discrete glass fibres in polyoxymethylene (POM) were investigated a few years ago[9], using a BIORAD MRC500 CLSM but the 3D reconstruction was operator intensive and pattern recognition of fibre centres between sections had to be achieved off-line. Recently complex regions of an injection moulded composite have been investigated using the Noran CLSM.

Injection moulded, PushPull sample



(a) (b)

Fig.6: (a) A (3mm x 3mm) region showing vorticity within an Ultramid 12 stroke, push-pull processed plaque and (b) computed spatial variation of the tensor component, a_{22} of fibre orientations (black=0, white=1) for the vortex region using 2D image analyser data (area approximately 1mm x 2mm).

The ‘vortex’ region (which was originally identified by the Leeds large area, 2D image analyser system) within the push-pull processed Ultramid sample, shown in figure 6, has recently been investigated using the CLSM.

By taking sets of XY sections at the surface (Z= 0) and at depths of 5, 10 and 15 μm below the surface, and pattern matching fibre centres between the XY sections, the fibre orientations have been found unambiguously. Figure 7 below shows a large area reconstruction of the fibre orientations where the length of each fibre line illustrates the out of plane angle, θ and the direction of the fibre is denoted by the 'ring' on the fibre line. Note that the CLSM reconstruction is of the same region as Fig. 6(a), but is rotated by 90°. With this 3D information, a number of novel characterisations now become possible.
For example, the curl of the vector field for this region may be derived from the fibre orientation data. Representing each fibre by the vector,

$$\mathbf{P}(x, y, z) = \mathbf{i}.P_x + \mathbf{j}.P_y + \mathbf{k}.P_z \qquad (1)$$

then the curl of the vector field is given by:

$$\nabla \times \mathbf{P} \equiv \text{curl } \mathbf{P} = \mathbf{i}\left(\frac{\partial P_z}{\partial y} - \frac{\partial P_y}{\partial z}\right) + \mathbf{j}\left(\frac{\partial P_x}{\partial z} - \frac{\partial Pz}{\partial x}\right) + \mathbf{k}\left(\frac{\partial P_y}{\partial x} - \frac{\partial P_x}{\partial y}\right) \qquad (2)$$

where each term may be averaged over the fibres within a small region of the image. In figure 8, the magnitude of the curl **P** function is shown, overlaid on the original fibre orientation image.

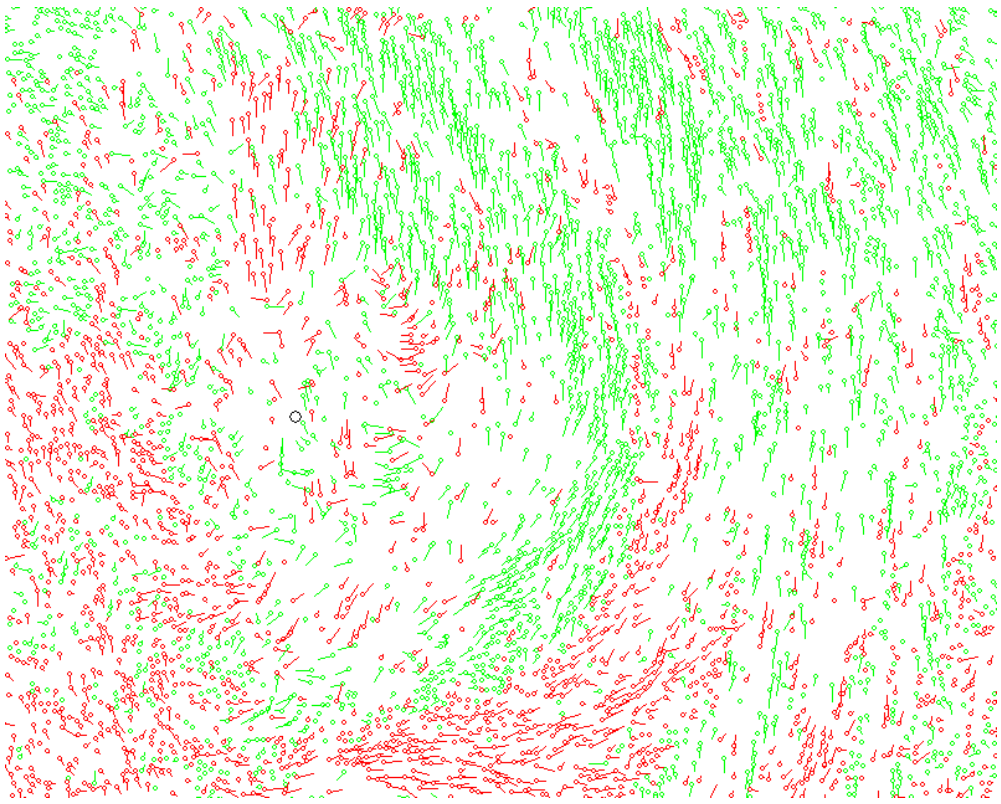


Fig. 7: CLSM reconstruction of the Ultramid 'vortex' region showing sense of fibre rotation about the presumed centre of the vortex (marked by the dotted ring).

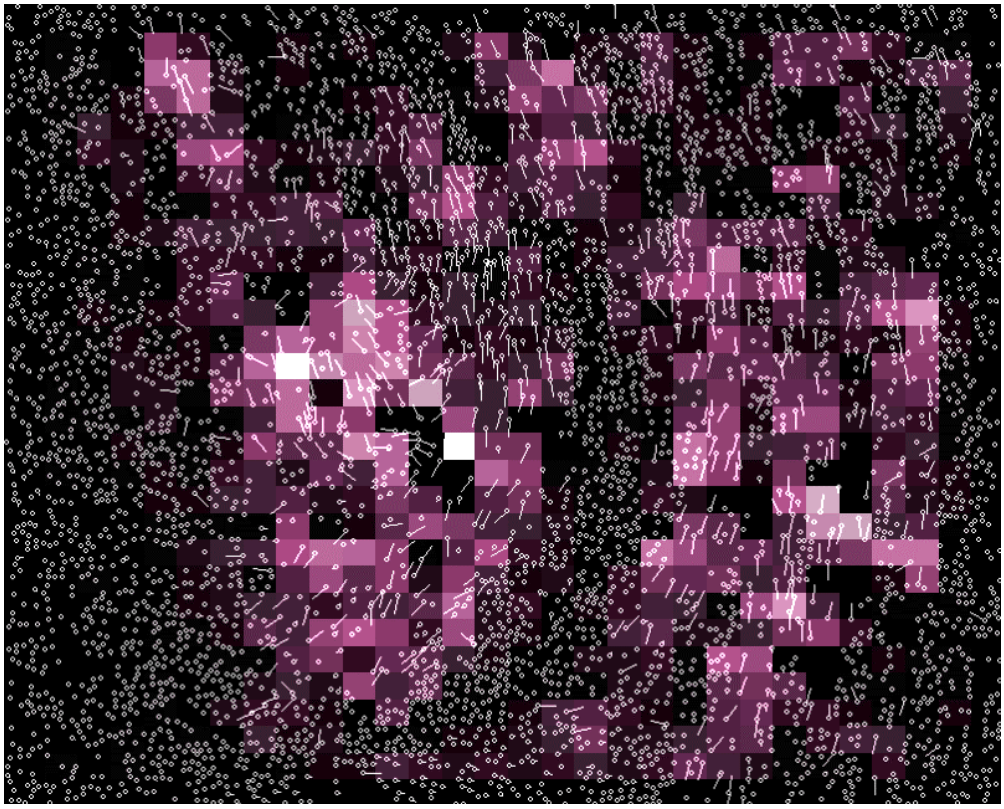


Fig. 8: The average values of the magnitude of curl **P** within the 'vortex' region

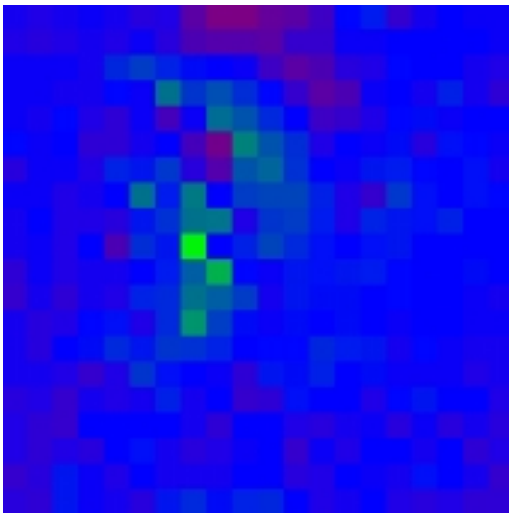


Fig.9: Variation of the off-diagonal, second order, orientation tensor component, a_{23} (= a_{32}) where the intensity is colour coded i.e. red = - 0.5, blue = 0 and green = 0.5. Note that these data cannot be acquired from the 2D analysis of a single section plane.

Following the convention of Advani and Tucker[10], the second rank fibre orientation tensor can now have all of its elements characterised by the unambiguous 3D fibre orientations which have been derived from the CLSM data. As an example of what can be achieved, the fibre orientation data for the vortex region has been used to form one of the off-diagonal tensor components, a_{23}, as shown in Fig. 9, where the tensor is defined by the expression :

$$a_{ij} = \begin{bmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{bmatrix} \qquad (3)$$

These initial data on the vortex region give a tantalising (but incomplete) picture of the 3D flow. Further studies are necessary to resolve the asymmetry of the flow in this region and to establish whether or not the fibres are defining counter-rotating shells around the centre of the 'vortex'.

CONCLUSIONS

In this paper, an attempt has been made to illustrate the potential for the CLSM technique in characterising glass fibre orientations and glass fibre spatial distributions in polymer composites. Sadly, the CLSM technique is of little use for characterising carbon fibres in composites (unless the particular samples of interest have low packing fractions of carbon fibres, say 20% or less by volume).

In order to improve the fibre orientation statistics (and be sure that different regions within the samples behave in the same way) robust software is needed to partially or fully automate the image data acquisition and correct classification of fibre cross-sections within the CLSM images. This software is being developed.

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