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## Accepted Manuscript

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Abstract:

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calculated. Furthermore in the presence of a fibre-break, interfacial debonding and matrix yielding during stress transfer was investigated.

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**Six image phase stepped photoelasticity for the quantification of the stress field  
around 25  $\mu\text{m}$  reinforcing fibres**

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**Abstract**

A six image phase stepped photoelastic technique has been applied for the measurement of the interfacial shear stress distribution in the matrix resin along a reinforcing E-glass fibre of  $\sim 25 \mu\text{m}$  at its end and at a fibre-break. Contour maps of the isochromatic fringe order and its corresponding isoclinic angle, which are related to the principal stress difference and direction relative to the fibre axis, could be obtained continuously using this photoelastic technique. Thus the actual interfacial shear stress along the fibre can be calculated. Furthermore in the presence of a fibre-break, interfacial debonding and matrix yielding during stress transfer was investigated.

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Photoelasticity

**1. Introduction**

The photoelastic method has been used for analysis of interfacial stresses in fibre composites. Schuster and Scala [1] reported on its application to sapphire whiskers in a birefringent resin. More recently, Fiedler and Schulte [2] applied this technique to glass fibres in a resin. In recent studies, Zhao et al. [3 - 8] have applied the 4 image phase stepping automated photoelastic technique of Patterson and Wang [9] to single embedded fibres. This technique can provide full field maps of the relative retardation and the isoclinic angle. By the application of an unwrapping algorithm [10], a continuous map of isochromatic fringe order can be obtained. It has been used to investigate the stress field in a matrix and the stress distribution at the interface in model composites [5, 6, 9]. In the previous studies, the photoelastic technique has been limited to relatively large diameter fibres of  $> 80 \mu\text{m}$  because of the loss of light intensity through the optics and the high stress fringe constant of the matrix resins. Patterson and Wang [11] have developed a four image phase-stepping automated photoelastic camera for the simultaneous observation and recording of the four images required to solve the equations for quantification. Zhao and Jones [3, 5-7] succeeded in applying this technique to investigate the interfacial shear stresses in model single fibre composites. Full profiles of the interfacial shear stress could be determined by assuming that the principle stress at the interface is one of shear. Thus the initiation and propagation of interfacial debonds at fibre-breaks and matrix cracks have been studied. The technique records the principle stress difference ( $\sigma_1 - \sigma_2$ ) so that within the matrix full quantification is not possible, however Zhao et al. [3] were able to observe trends in the radial direction from fibre which showed that interfacial bond formation was a curing temperature dependent. For a fibre of a few micrometers in diameter, a smaller region of the matrix at the interface is perturbed so that a higher resolution microscope and increased light transmission are needed. However in the 4 image phase stepped system,

the polarized light is divided into four beams by Cubic beam splitters, at which losses in light intensity are significant, which leads to difficulties in processing the image by the multiplex software [3, 11]. This limits the application of four image phase-stepping photoelasticity to relatively low magnifications where the signal remains intense.

In this present work, a six image phase-stepping photoelastic technique has been developed to investigate the interfacial shear stress at the surface of fibres of smaller diameter: In this case a glass fibre of 25  $\mu\text{m}$  in an epoxy resin. Each of 6 images can be acquired directly using an elliptically polarized light beam by a digital camera without the need for the cubic beam splitters, to achieve a high quality images of  $720 \times 720 \mu\text{m}^2$  in size for photoelastic analysis .

## 2. Experimental

Unsize and unsilanised E-glass fibres used in this study were provided by Owens Corning (Battice,Belgium) with the diameter of  $25 \pm 1.2 \mu\text{m}$ , during manufacture, only water was used to cool the fibres.

The matrix resin used in this study was a mixture of Araldite LY 5052 epoxy resin, and 38 phr (parts per hundred resin by weight) Aradur HY 5052 hardener, produced by Huntsman (UK), which were supplied by Advanced Composites Group (UK). The resin blend was cured at room temperature for at least 7 days. The room-temperature cured epoxy resin was chosen in order to avoid the induction of a residual thermal stress on cooling from a curing temperature [7, 8]. The properties of the fibre and cured resin are shown in Table 1.

The stress-fringe constant,  $f_\sigma$ , was determined in a tensile test on the epoxy resin casting. Under monochromatic polarised light the specimen was either completely dark or bright (i.e. fringe order  $N = 0, 0.5, 1, 1.5 \dots$ ) as the applied stress was increased, as shown in equation 1.

$$f_\sigma = \frac{(\sigma_1 - \sigma_2)t}{N} \quad (1)$$

where  $t$  is the thickness of the specimen,  $(\sigma_1 - \sigma_2)$  is the principal stress difference,  $f_\sigma$  is the stress-fringe constant of the resin, i.e. the slope of the curve of  $(\sigma_1 - \sigma_2)t$  against fringe order  $N$ , which is 20.23 MPa/fringe/mm in the elastic range of the resin (Table 1), the same as that measured by Zhao et al [3].

### 3. Six image phase-stepping photoelastic technique

A phase-stepping automated polariscope was combined with a horizontal tensile mini-tester with a 2 kN load cell and microscope during fragmentation for the measurement of the stress field in an epoxy resin around fibre-breaks. The principle of the instrument is shown in Figure 1. It is equipped with a digital camera (Panasonic WV-BP330) for collecting six photoelastic images by rotating the positions  $(\alpha, \beta)$  of a quarter wave plate ( $Q_2$ ) and the analyser (A). The relative position of  $Q_2$  and A are given in Table 2, thus generating six different phase-stepped images. Table 2 also shows the position of the other optical elements, the polariser and the quarter wave plate ( $Q_1$ ), which were kept at  $0^\circ$  and  $-45^\circ$ . A monochromatic light made up from deep yellow wavelength at  $589.3 \pm 0.3$  nm was used. The light is elliptically polarised, and then passes through the specimen, which is subjected to a tensile load, quarter-wave plate and analysers, and is focused by objective lenses, on to the digital camera. By rotating the angles of the  $Q_2$



quarter wave plate and the analyser, the six images can be obtained, which can be processed by a multiplexer to combine them into a single composite image that can be digitized by a single frame-grabber.

The automated polariscope allowed continuous monitoring of the stress build-up at fibre-breaks and fibre-ends, which enable the load transfer at the interface to be stimulated. The magnitude and direction of stresses at any point can be determined by examination of the fringe pattern, *i.e.* isochromatic and isoclinic fringes. Photoelastic analysis does not require the necessary saturation in fibre fragmentation to be reached [3, 11]. The polariscope uses the method of phase-stepping to determine the relative retardation and isoclinic angles. Table 2 shows the rotating angles of analyzer and  $Q_2$  quarter-wave plate to capture six images. When the analyzer and its associated quarter-wave plate are set to produce circularly polarised light, the intensity of light emitted from a polariscope can be described by the following equation [9],

$$i_n = i_m + i_v \{ \sin 2(\alpha - \theta) \cos \delta - \sin 2(\theta - \beta) \cos 2(\alpha - \beta) \sin \delta \} \quad (2)$$

where  $n = 1, 2, \dots, 6$ ,  $\beta$  and  $\alpha$  are the angular orientations of the analyser and its associated quarter-wave plate, respectively, with respect to the polariser.  $\theta$  and  $\delta$  are the isoclinic angle and the relative retardation, respectively. The isoclinic fringe pattern represents the contours of equal angles between the direction of the highest principal stress,  $\sigma_1$ , and the fibre axis. It depends only on the angle between the specimen or fibre axis and the optical set-up.  $i_m$  and  $i_v$  are the intensities of stray light observed when all the optical axes of all the elements and the specimen are parallel. The unknown isoclinic angle and relative retardation are found from six light intensities,  $i_1, i_2, \dots, i_6$  measured for each point  $(x, y)$  at different values of  $\beta$  and  $\alpha$ , and using following equations:

$$\theta = \frac{1}{2} \tan^{-1} \left( \frac{i_5 - i_3}{i_4 - i_6} \right) \quad (3)$$

$$\delta = \tan^{-1} \left( \frac{i_4 - i_6}{(i_1 - i_2) \cos 2\theta} \right) \quad (4)$$

The automated system supplied by Wang's DTS Ltd. (UK) [9] possesses unwrapping algorithms which are used to produce a continuous map of relative retardation. The map of relative retardation can be converted to isochromatic fringe order by a software operation [9]. The isochromatics only depend on the applied stress, which have nothing to do with the fibre centre axis and the optical set-up. The relative retardation,  $\delta$ , in the specimen is related to the fringe order,  $N$ , [12], as follows:

$$\delta = 2\pi N \quad (5)$$

In photoelasticity, the relationship of the isochromatic fringe order,  $N$ , the principal stresses  $\sigma_1$  and  $\sigma_2$ , and maximum shear stress,  $\tau_{\max}$  in a two dimensional model as described by the following equation:

$$\tau_{\max} = \frac{1}{2} (\sigma_1 - \sigma_2) = \frac{f_{\sigma} N}{2t} \quad (6)$$

where  $t$  is the thickness of the specimen. In fact, the stresses are not constant through the thickness of a resin matrix specimen containing a high Young's modulus fibre, and the observed values of the principal stress difference or maximum shear stress are an average. To correct for the depth of the resin at the interface, equation 7 is used [1, 2].

The corrected maximum shear stress,  $\tau_{c\max}$ , can be calculated by the following equation [1],

$$\tau_{c\max} = 0.5 \left( \frac{t(\sigma_0 - \sigma_n)(r_1 - r_0)}{2 \left\{ r_1 a - 0.5 \left[ r_1 a + (r_0 + y)^2 \log \left( \frac{a + r_1}{r_0 + y} \right) \right] \right\}} + \sigma_n \right) \quad (7)$$

Where  $a = \sqrt{r_1^2 - (r_0 + y)^2}$ ,  $\sigma_0$  is the observed principal stress difference at the interface.

$\sigma_n$  is normal applied matrix stress. The other variables are defined in Figure 2.  $r_0$  is the fibre radius.  $r_1$  is the distance from centre of fibre to the point where the radial distribution of applied matrix stress falls to zero.  $y$  is the distance from edge of fibre,  $y = 0$  at the interface.  $z$  is a dummy variable, defined by  $z = r_1 \cos \theta$  at  $r = r_1$ ,  $z = 0$  at  $r = r_0 + y$ .  $t$  is thickness of the specimen.

The actual interfacial shear stress  $\tau_i$  between the fibre and matrix resin can be calculated from the corrected maximum shear stress,  $\tau_{cmax}$ , and the isoclinic angle,  $\theta$ , according to Mohr's circle [2].

$$\tau_i = \tau_{cmax} \sin 2\theta \quad (8)$$

## 4. Results

### 4.1 Stress near a fibre-end

Figures 3-5 show the contour maps of fringe order in the matrix resin around a 15.1 mm long E-glass fibre-end at the applied matrix stresses of 18.38, 25.61 and 32.53 MPa.

The dimensions of the matrix region in the contour map of fringe order are 0.51 mm by 0.31 mm. It can be seen that there are regions of high fringe order in the vicinity of the fibre, indicating the existence of a high stress concentration zone. At an applied stress of 18.38 MPa in Figure 3, the stress concentration region appeared at the rim of the fibre-end and at a short distance along the fibre. On increasing applied load to 25.61 MPa, the region of high fringe order extended along the fibre. The maximum fringe order occurred in the matrix near the interface. As the applied load was further increased to

32.53 MPa, the region of high fringe order propagated along the fibre, away from the fibre-end. This indicated that a debond had been initiated.

These images can be quantified by applying the analysis described in equations 3 - 8. At the fibre-matrix interface, a shear stress dominates so that the interfacial shear stress can be calculated from the principal stress difference at the interface. Figure 6 shows the profiles of interfacial shear stress for the 25  $\mu\text{m}$  fibre in the epoxy resin at the three different applied stresses. It can be seen that on increasing the applied load, the maximum interfacial shear stresses increased. At an applied stress of 18.38 MPa, the maximum value of interfacial shear stress is very close to fibre end, however on increasing the applied load, the maximum value moved along the fibre. Since the maximum can be associated with the bonded interface we can see that at a high load, a debond forms and propagates along the fibre. The shear stress decays according to that of a well bonded fibre [6]. The peaks in the shear stress curves in the bonded region occurred at  $\sim 7, 18$  and  $33 \mu\text{m}$  away from the fibre-ends at the loads of 18.38, 25.61 and 32.53 MPa, respectively, which correspond to the maximum interfacial shear stresses: 26.87, 30.22 and 33.20 MPa. The maximum interfacial shear stresses at the applied stresses of 25.61 and 32.53 MPa, are higher than the matrix shear yielding stress of  $27.7 \pm 1.4$  MPa (Table 1), so that the debond happened at the fibre-end. Therefore, the interfacial bond shear strength could have a value between 26.87 and 30.22 MPa.

#### 4.2 Stresses near a fibre-break

Figure 7 shows the contours of the fringe order at the first fibre-break at an applied stress of 24.69 MPa. The area examined has the dimensions of 0.64 mm by 0.50 mm. The left fragment associated with the break is 4.65 mm long, the right fragment is 4.71

mm in length. From Figure 7 it can be seen that the highest fringe order appears at the end of the fibre-break and extends along the interface with the fibre. On increasing the applied stress to 33.09 MPa, the interfacial shear stress exhibited a symmetrical distribution at the fibre-break. The region of high fringe order propagated along the fibre away from the fibre-break (Figure 8). In order to simplify the calculation of the interfacial shear stress, the only top right areas in Figures 7 and 8 were quantitatively analyzed. The interfacial shear stress profile was calculated from the fringe order directly at the interface along the fibre from the end of the fibre-break (Figure 9). With the reloading of fibre after fracture, a high region of fringe order appeared at the interface near the fibre-break at an applied stress of 24.69 MPa. The maximum interfacial shear stress of 30 MPa was observed to occur at  $\sim 13 \mu\text{m}$  away from the fibre-break. There was no evidence of a debond in the optical micrograph at this axial load. Therefore yielding of the interphasal matrix appears to have arisen from the resolved shear stress reaching 30 MPa. Compared with the above analysis, the interfacial bond shear strength could be  $\sim 30$  MPa. At the higher applied stress of 33.09 MPa, the interfacial shear stress reached a maximum of 36.3 MPa at a distance of 39  $\mu\text{m}$  away from the fibre-break. The minimum in the shear stress profile is at 12  $\mu\text{m}$  in Figure 9, which corresponded to the observed interfacial debonding length, recorded in the optical micrograph shown in Figure 10.

## 5. Discussion

In this work, 6 image phase stepped photoelasticity was introduced to successfully measure the stress field around 25  $\mu\text{m}$  diameter fibres. By increasing the resolution of microscope and light transmission, the change in fringe order in the small region of  $720 \times 720 \mu\text{m}^2$  at the interface was clearly recorded. This enabled the variation in interfacial

shear stress with applied load to be studied. Using this technique, real-time data could be obtained for the strain field in the matrix resin at the fibre-end and fibre-break for a fibre about 20 microns in diameter, which is of typical dimensions of an industrial reinforcement. This therefore, has an advantage over common micro-mechanical techniques, such as fibre fragmentation or pull-out for investigating the interfacial properties because the data analysis does not require the assumptions associated with the theoretical models employed. The micro-mechanisms of stress transfer in fibre composites can be observed by this technique. The main disadvantages are associated with the intensity of the transmitted light under high magnification. The similarity of the matrix stress profiles at fibre-breaks and fibre-ends obtained by photoelasticity with those from embedded carbon nanotubes-Raman sensors demonstrates the advantage of mapping the strain in the interfacial matrix of model fibre-matrix composites [13-16].

Fibre fracture, interfacial debonding and propagation, and interfacial yielding are all involved in the micromechanics of the fracture of a fibre composite. These have been shown to significantly influence the matrix stress field and hence the magnitude of the interfacial shear stress which results from stress transfer into the unbroken fragments [3, 6]. In this study, interfacial debonding was found to be initiated after matrix yielding and propagate along the fibre from the fibre-end, during fragmentation despite the lack of a fibre-surface treatment. However at a free fibre-end, the maximum shear stress was shown to move away from the fibre-end on increasing the axial load (Figure 6). This was an indication of debonding which resulted from the stress concentration at the free-end. With the applied stress increasing, the frictional shear stress at the debonded interface has a range of values, because of the complex micromechanics of failure, involving partial and complete debonding [4].

At a fragment-break, debonding did not occur immediately under an axial stress of 24.69 MPa. However shear yielding of the matrix in the vicinity of the fibre/matrix interface was observed to proceed debonding which occurred at the higher applied stress of 33.09 MPa, as shown in Figure 9. The stress transfer profile at an applied stress of 24.69 MPa did not exhibit features typical of debonding but showed a constant shear stress indicative of interphasal matrix yield. However on increasing the applied stress from 24.69 to 33.09 MPa, interfacial debonding at the fibre/matrix interface clearly occurred. The minimum in the interfacial shear stress profile was shown to correspond to a debonded region as shown in Figure 10.

The most significant result from this study is the differing interfacial response to stress transfer at a free fibre-end and at fibre-break. The fibre used had not had a silane or other surface treatment. Furthermore the resin was cured at room temperature over a long period of 7 days. Residual stresses in the sample can be considered to be negligible, since relaxation of cure shrinkage stresses can be anticipated during the long-term sample preparation. Firstly the long-considered adhesion of bare glass to epoxy resin is demonstrated. However, debonding could be initiated at a free-end by the stress concentration associated with the free edge. However during fragmentation the stored energy which is released leads to matrix yield which dissipates the energy. Debonding in this case only occurs when the interfacial shear bond strength is exceeded. This observation may explain some discrepancies from calculating bond strengths from the average fragment length. An interfacial bond strength was found to be  $\sim 30$  MPa which is close to the matrix yield strength of  $27.7 \pm 1.4$  MPa, explaining the complex

micromechanics. 6-phase stepped photoelasticity has also proved successful in identifying these micromechanics.

## 6. Conclusions

A six image phase-stepping photoelastic analysis is a feasible method for the quantitative determination of stress distributions in fine diameter single fibre composites. It can be directly applied to an investigation of the in-situ matrix stress or interfacial shear stress distributions in low diameter fibre / epoxy system. The six image phase-stepping photoelasticity is able to quantify the propagation of interfacial debonding, as well as identify delays associated with matrix yielding. This technique allows a full study of interfacial micromechanics in model fibre composites. This study has shown that bonding of unsilanised glass fibre to epoxy resin occurs and that a mixed micromechanics of interphasal yield and debonding occur at fibre-ends and fragments.

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### Figures Captions

Figure 1 Experimental arrangement for 6 image phase stepped Photoelasticity

Figure 2 The geometrical correction of the principal stress difference as a function of resin thickness ( $t$ )

Figure 3 Contour maps of fringe order in the matrix resin at the free-end of an E-glass fibre at 18.38 MPa

Figure 4 Contour maps of fringe order in the matrix resin at the free-end of an E-glass fibre at 25.61 MPa

Figure 5 Contour maps of fringe order in the matrix resin at the free-end of an E-glass fibre at 32.53 MPa

Figure 6 The profiles of interfacial shear stress determined by photoelastic analysis for E-glass fibre-end embedded in epoxy matrix at 18.38, 25.61 and 32.53 MPa

Figure 7 Contour maps of fringe order in matrix resin around the fibre-break of an E-glass fibre at 24.69 MPa

Figure 8 Contour maps of fringe order in matrix resin around the fibre-break of an E-glass fibre at 33.09 MPa

Figure 9 The profiles of interfacial shear stress determined by photoelastic analysis at a fibre-break in an E-glass fibre embedded in epoxy matrix at 24.69 and 33.09 MPa

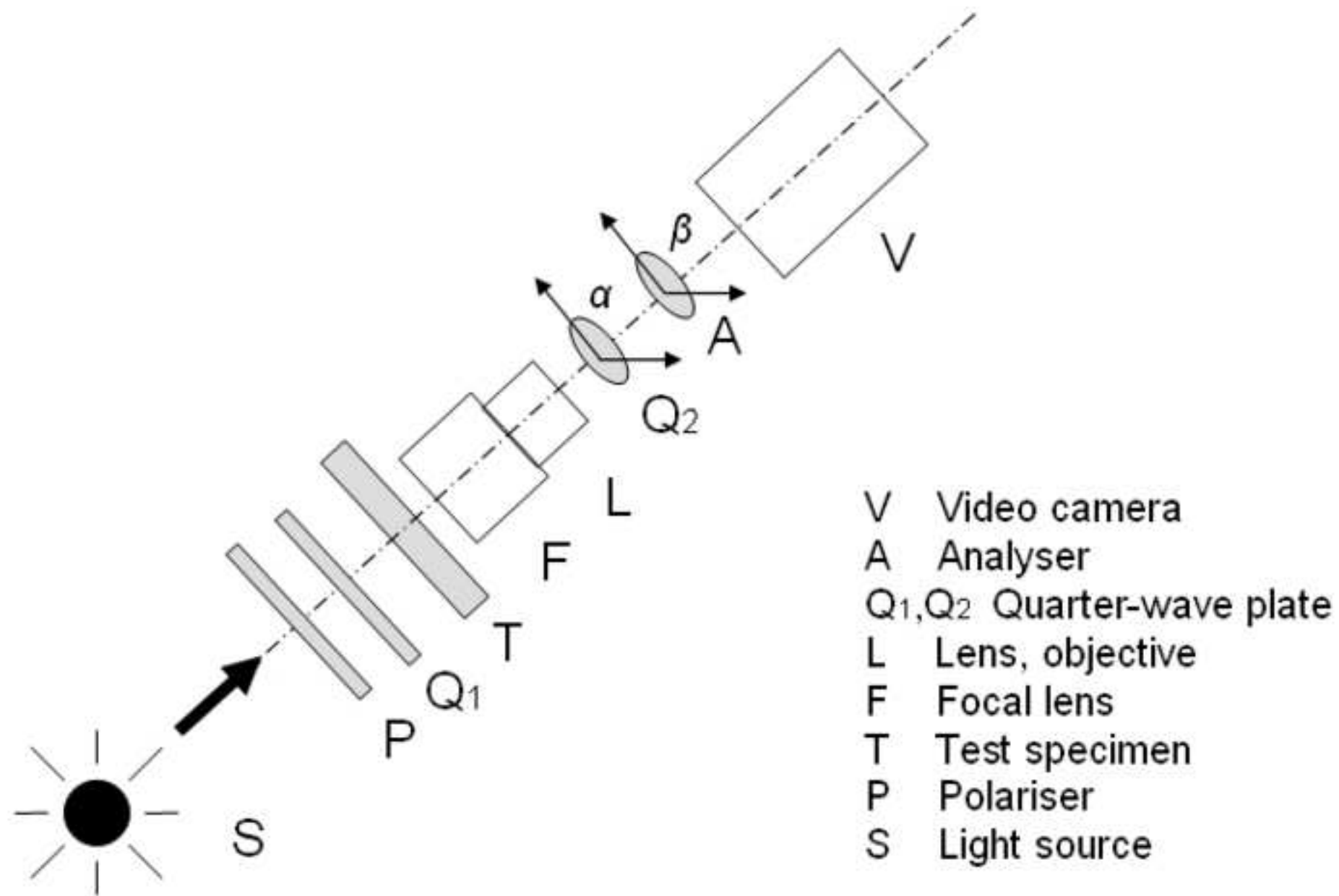
Figure 10 Micrograph showing interfacial debonding around a fibre-break under the optical microscope (POLYVAR) after loading axially to 33.09 MPa

Table 1 Properties of the E-glass fibre and the matrix resin

Matrix resin		E-glass fibres	
Young's modulus (GPa)	$3.2 \pm 0.4$	Young's modulus(GPa)	$72 \pm 2.7$
Tensile strength (MPa)	$72 \pm 3.5$	Diameter ( $\mu\text{m}$ )	$25 \pm 1.2$
Tensile yield stress	$48 \pm 2.3$		
Shear yield stress (MPa)	$27.7 \pm 1.4$		
Tensile yield strain (%)	$1.5 \pm 0.4$		
Stress-fringe constant (MPa/fringe/mm)	$20.23 \pm 0.2$		

Table 2 Orientations of the polarizer (P), quarter wave plates ( $Q_1$ ,  $Q_2$ ) and analyzer (A)

Light intensity of each image	P	$Q_1$	$Q_2 / \alpha$	A / $\beta$
$i_1$			0	$45^\circ$
$i_2$			0	$-45^\circ$
$i_3$	0	$-45^\circ$	0	0
$i_4$			$-45^\circ$	$-45^\circ$
$i_5$			$-90^\circ$	$-90^\circ$
$i_6$			$-135^\circ$	$-135^\circ$



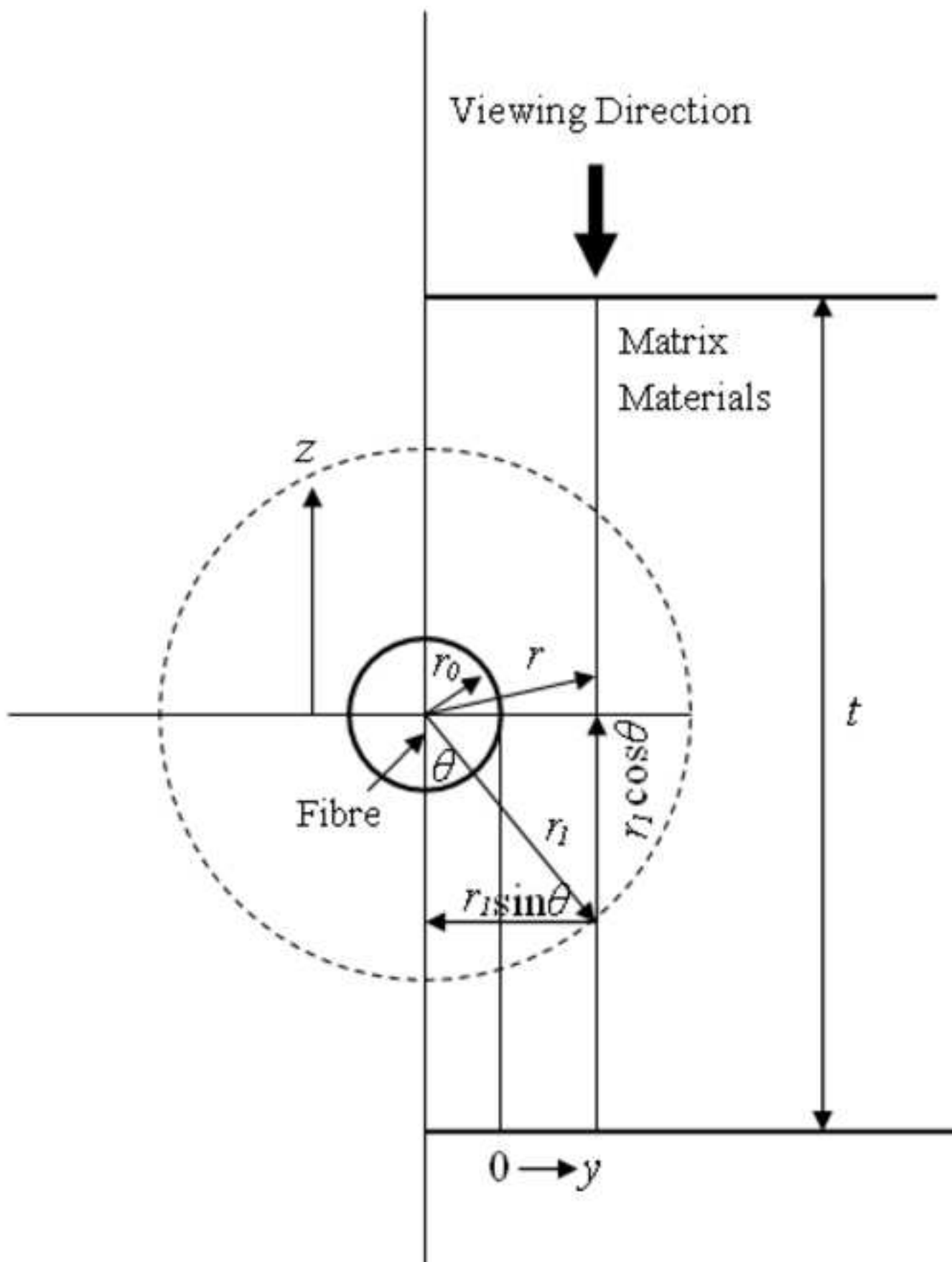


Fig 3

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0.51 mm

0.31 mm

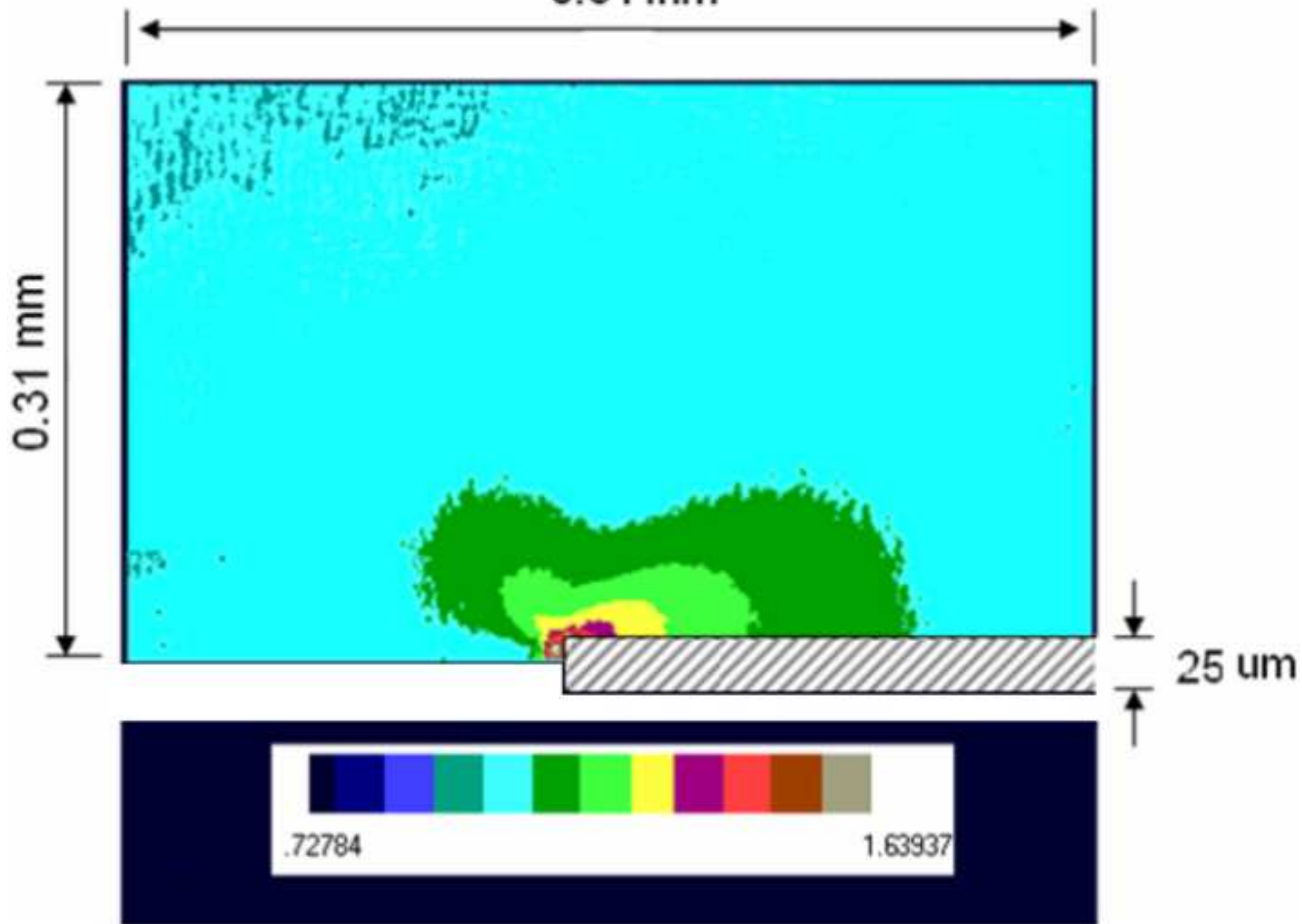


Fig4

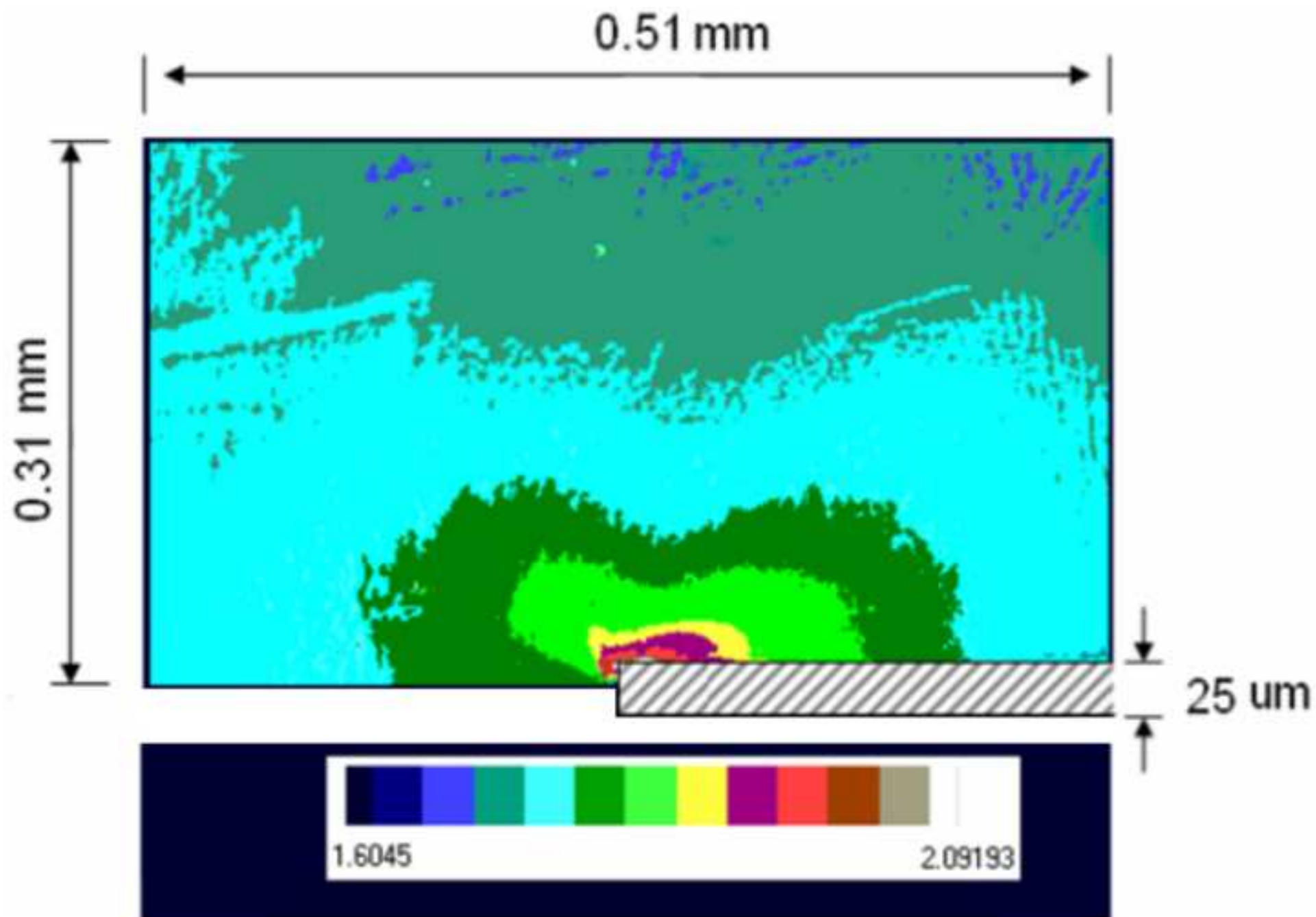
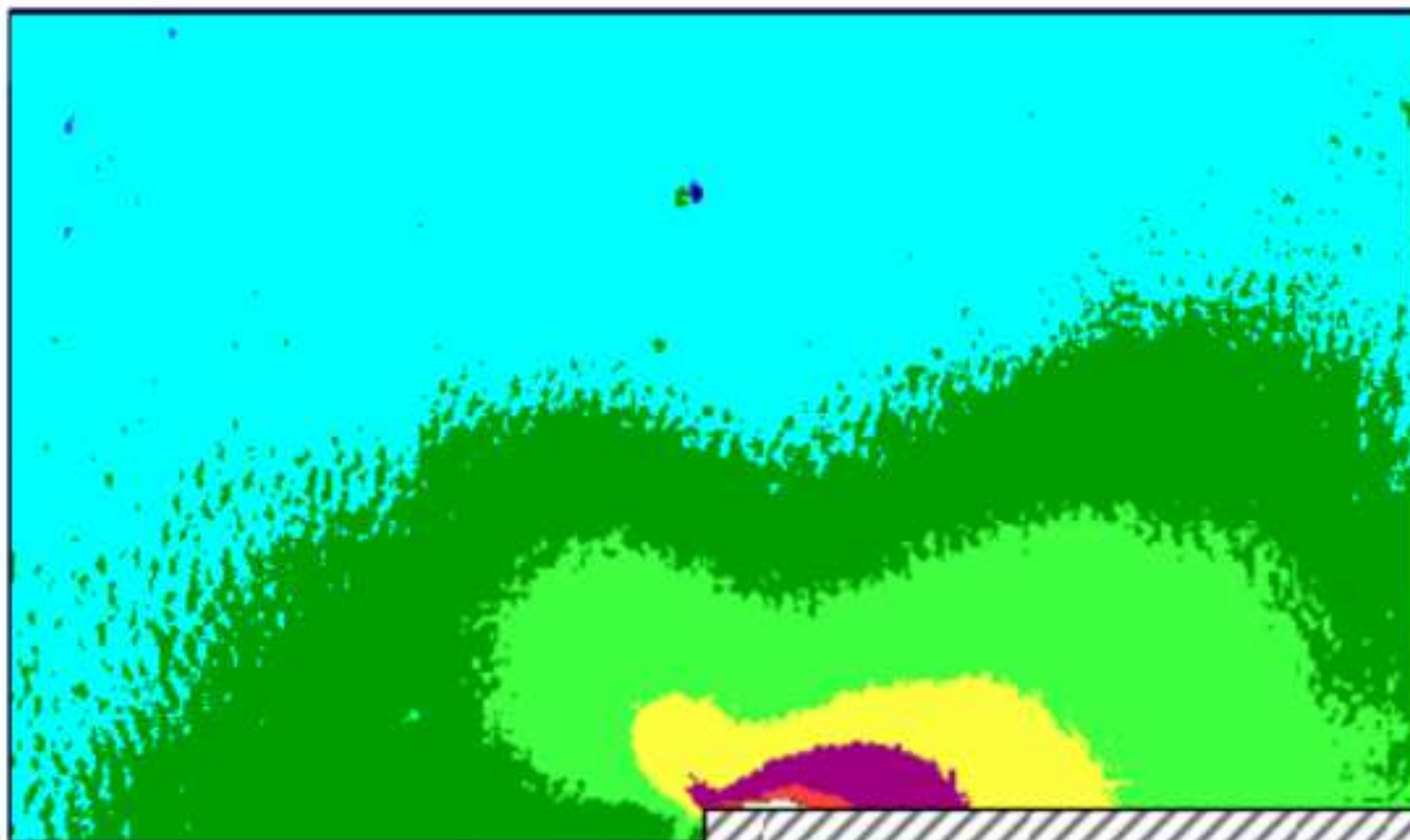




Fig5

0.51mm

0.31 mm



25 μm



2.2760

2.69269

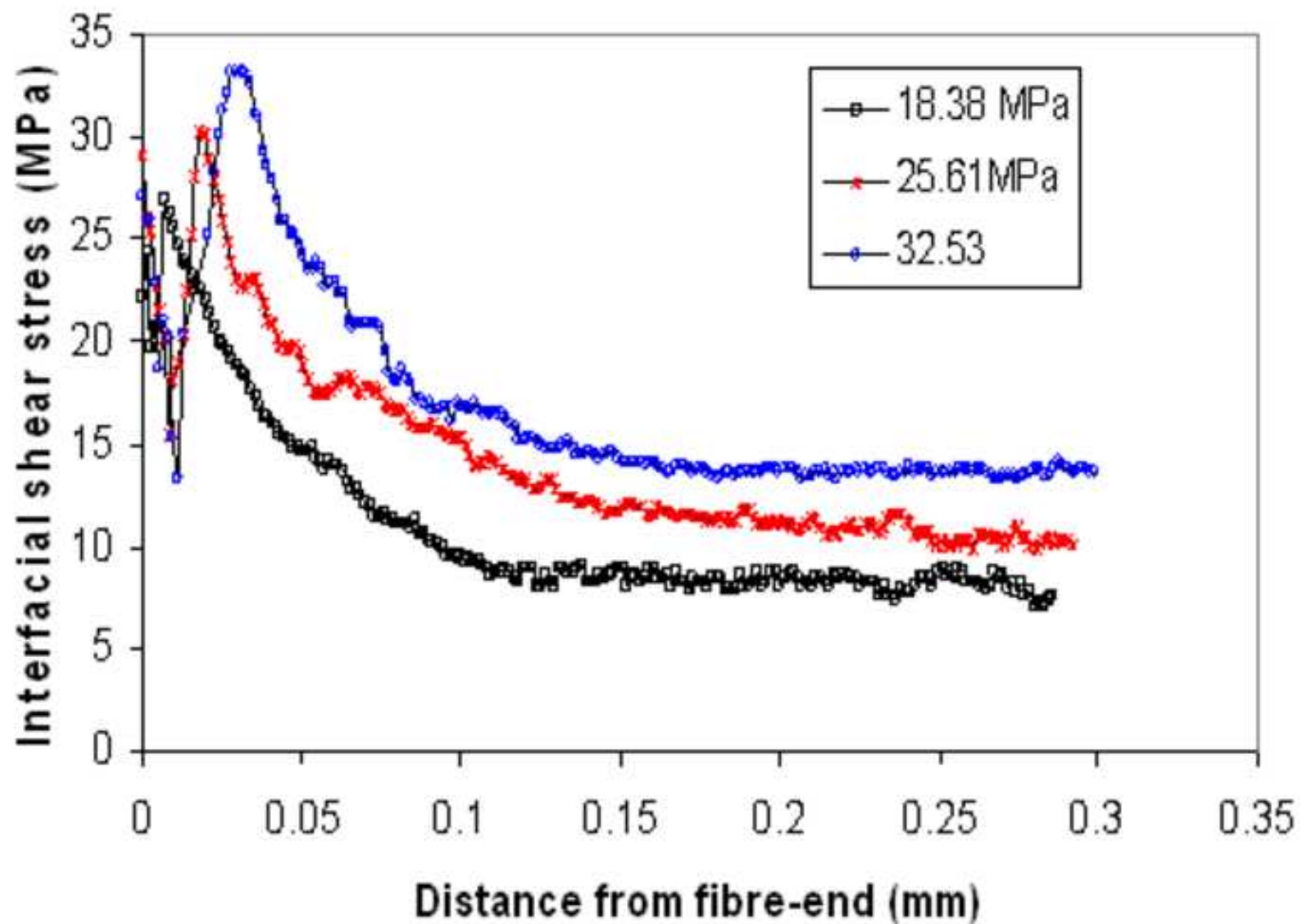


Fig7

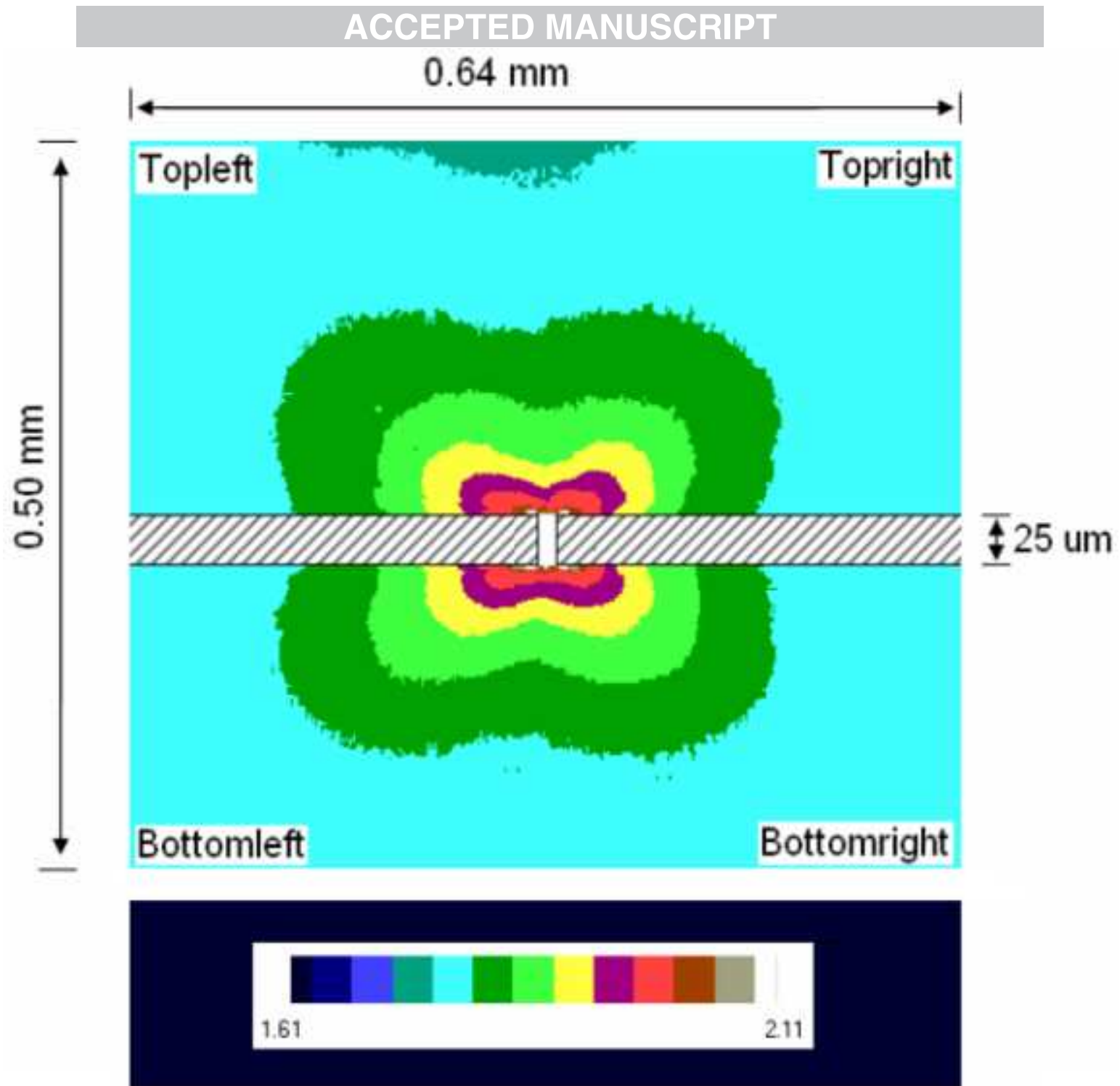


Fig8

