Measurement of the interfacial normal strength in single fibre transverse tensile tests

MEASUREMENT OF THE INTERFACIAL NORMAL STRENGTH IN SINGLE FIBRE TRANSVERSE TENSILE TESTS

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A technique is presented which can be used to measure the interfacial normal strength in transversely loaded composites. The technique combines the measurement of local deformations during tests in a Scanning Electron Microscope and numerical simulations, in which the measurements are used as boundary conditions. Deformations are measured by placing a grid of markers on the surface of single fibre specimens. The displacements of these markers are measured during loading of the specimen. Numerical simulations show that the radial and tangential interface stresses increase towards the specimen surface, causing the initiation of debonding at the surface. This effect is supported by tests under an optical microscope. The interfacial normal strength is defined to be the maximum radial interface stress just before the initiation of debonding. Based on the presented results, it can be concluded that the presented technique is a promising tool for the measurement of the interfacial normal stress.

Introduction

The mechanical properties of interfaces or interphases in composite materials are difficult to determine. Until recently, most of the work to characterize interfaces was performed in shear loading conditions. Experiments such as pull-out, microdebond, fragmentation or microindentation tests in combination with numerical models of these experiments result in a value for the interfacial shear strength (ISS) [1, 2]. These experiments exist already for a long time, but accurate values for the interfacial shear strength were obtained only recently due to the developments in laser Raman spectroscopy, which is used to measure field information during the experiments [3, 4]. However, since the first failure mechanism in composite structures is debonding in the transverse plies, not the interfacial shear strength but the strength of interfaces in normal loading conditions is the most critical parameter.

Increased attention for microphenomena in composite materials resulted in an increased use of microscopic techniques in a wide range of experiments. For example, optical microscopes in combination with miniature tensile stages are used to study fibre breaking and the growth of debonding cracks along fibres in single and multiple fibre fragmentation tests [5, 6]. Daniel *et al.* [7] and Varna *et al.* [8] use the optical microscope to study the failure process of unidirectional composites under transverse and longitudinal loading. Using such techniques a qualitative understanding of microphenomena can be obtained, but for quantitative measurements of microscopic strains Scanning Electron Microscopy (SEM), in combination with a method for measuring local deformations, is a more suitable technique. In situ observation of fracture and damage propagation in ceramic matrix composites using a SEM has been performed by several researchers [9, 10]. A SEM also offers the opportunity to use the electron beam to produce line or dot patterns on the surface of specimens. The deformation of these patterns during loading of the specimen is a measure for the deformation of the material [11, 12]. The use of field information, measured during experiments offers the means to derive an accurate value for the interfacial normal strength.

In this study, a measurement technique for the micronscale is used to measure the interfacial normal strength (INS). Markers are placed on the surface of transversely loaded single fibre specimens using the electron beam of a SEM. Displacements of these markers are measured during a tensile test in the SEM, for which a specially designed tensile stage is used. Debonding cracks initiate at the specimen surface, which can be observed with the SEM. The measured displacements are used as input for finite element simulations of the experiment which give the maximum stresses at the interface. This experimental-numerical method is described in the next section.

Subsequently, the interfacial stress state at the surfaces of composite specimens is described, after which the results for single E-glass-fibre reinforced epoxy specimens are given.

The experimental-numerical method

◇ *A measurement technique for the micronscale*

A procedure has been developed to conduct near real-time measurements of deformations of composite materials, and to subsequently obtain measurements of displacements on the micronscale. A grid of markers on the surface of test specimens can be generated in the SEM, as is described in this section. For analysis in the SEM, polished composite specimens are used. To avoid charging, a ± 100 Å thick electrically conductive gold-palladium coating is applied. To obtain markers, the electron beam of the SEM is used to concentrate an amount of contamination, present in the vacuum chamber of the SEM, on one spot on the specimen surface. This results in a black spot. Repeating this procedure produces a marker field (Figure 1). The dots are placed on a part of a glass fibre cross-section and on the epoxy matrix. The diameter of the dots is 0.2 μm.

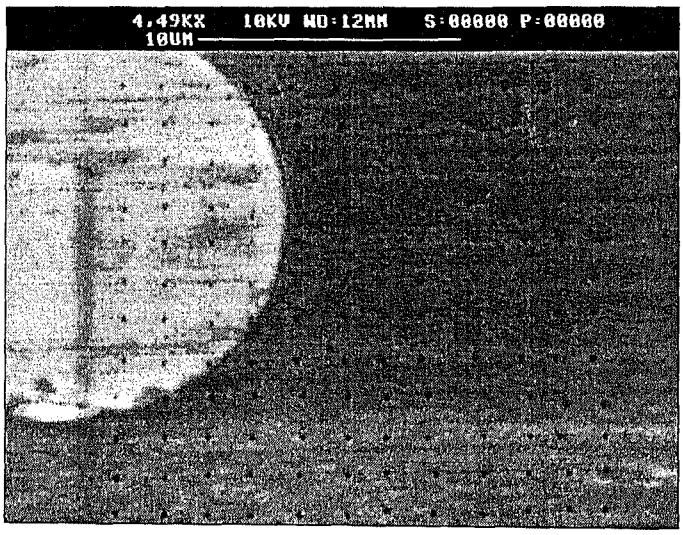

Figure 1: *Example of a marker field of black spots on a glass/epoxy composite.*

Displacements are measured by comparing the coordinates of the dots in reference and deformed situation. The coordinates are determined from directly digitized images. After isolating the markers from the images, the coordinates were defined to be their centre of gravity.

◇ *Numerical modelling of experiments*

The experiment, in which the displacements of markers are measured, is simulated with a finite element model. The geometry of this model is defined by the positions of the boundary markers. The displacements of these markers define the measured kinematic boundary conditions [12]. This method offers the advantage that the local stress-strain state in the experiment can be modelled accurately. In the experiments, the displacements of the markers are recorded at several load steps. The numerical model is then used to calculate the radial and tangential stresses at the fibre-matrix interface. The interfacial normal strength (INS) is then defined by the maximum stress at the interface just before debonding.

However, to give an exact description of the experiment, the finite element model has to give an accurate description of the stress-strain state in the experiment. In the next section, the local stress-state at the surface of a transversely loaded composite is considered.

Interfacial stresses at the surface of specimens

Because displacements are measured at the surface of a transversely loaded specimen, a 3D finite element model consisting of a quarter of a glass fibre and the surrounding epoxy matrix has been used for the analysis of 3D effects. Figure 2 shows the radial and tangential interface stresses, as a function of θ and the distance to the specimen surface z.

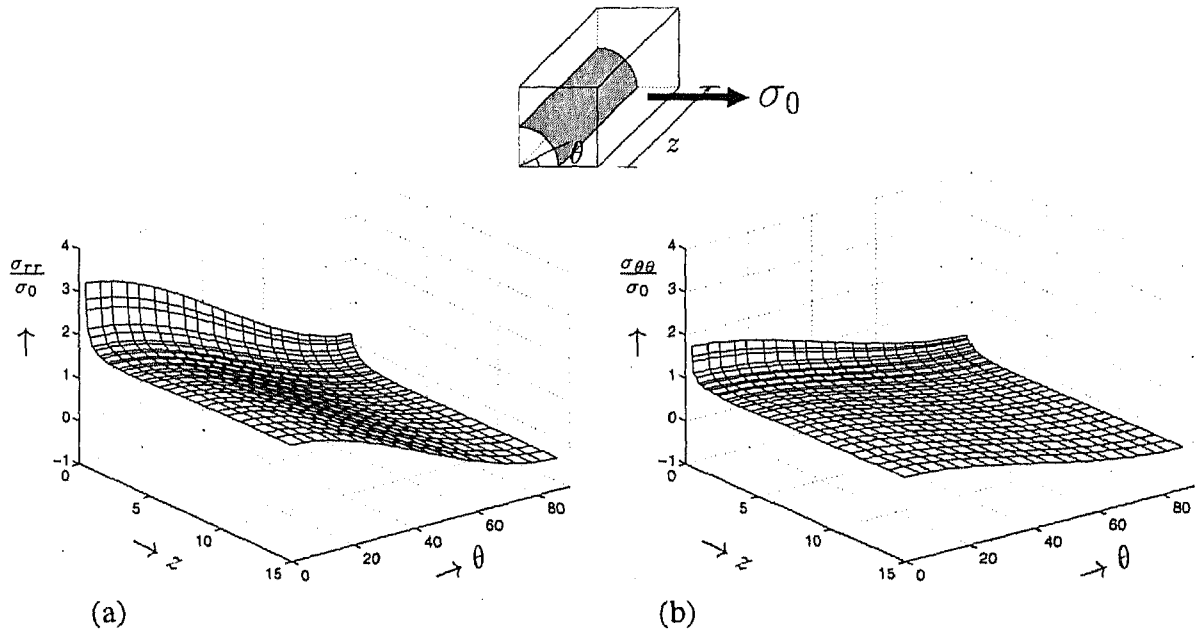

Figure 2: *Radial and tangential interface stresses.*

Figure 2a shows that the radial stresses at the interface reach a maximum value for $\theta = 0°$, and that the radial stresses increase significantly towards the specimen surface. The tangential interface stresses are lower than the radial stresses, as can be seen from Figure 2b. Additional analyses showed that the stress built-up towards the specimen surface increased for decreasing fibre volume fraction, indicating that for single fibre specimens the stress built-up is even larger than shown in Figure 2. To incorporate this effect, the experiments used in this study have to be modelled with 3-D finite element models.

The increase of stresses towards the specimen surface will cause debonding cracks to initiate at the specimen surfaces. Therefore, single fibre specimens were used for the observation of crack initiation under transverse loading. Dumpbell shaped miniature specimens with a small gauge length were designed. For the matrix material, an epoxy system consisting of a common diglycidyl ether of Bisphenol A (DGEBA, Ciba Geigy, LY 556) and a stoichiometric amount of of poly(oxypropylene)tri-amine curing agent (Texaco Jeffamine, T-403) was chosen. A single E-glass fibre was positioned and fixed in the cavity of a dumpbell-shape silicone mould. After degassing, the resin mixture was carefully injected into the mould with a syringe. The samples were cured at room temperature for 24 hours, and post-cured at 75°C for 16 hours, resulting in a single fibre specimen with the fibre positioned in the center of the gauge section. The curved edges of the specimens were carefully polished, until both fibre ends were free of surface damage.

To observe the initiation of debonding, the specimens were tested in a MINIMAT miniature materials tester, which was mounted on the x-y-table of an optical microscope equipped with a video camera. Transverse tensile tests were performed with a cross-head speed of 0.10 $mm \cdot min^{-1}$, during which the specimen surfaces were observed as indicated in Figure 3.

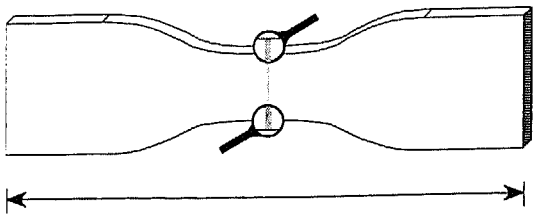

Figure 3: *Observation of the specimen surfaces during transverse tensile testing.*

The vicinity of the upper and lower specimen surfaces are shown in Figure 4 for four increasing load steps (σ_1 to σ_4).

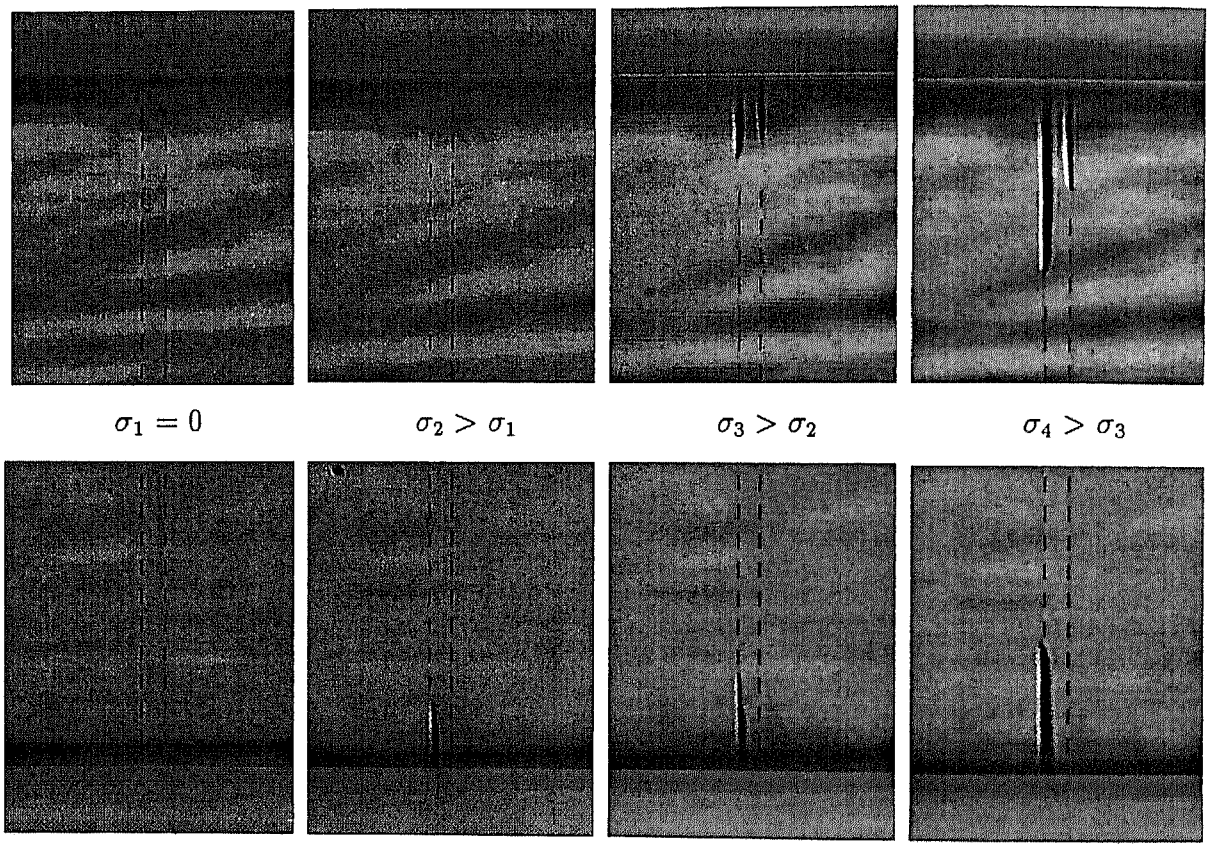

Figure 4: *Optical micrographs of initiation of debonding in a transverse tensile test.*

At the second load step, a debond crack initiates at the lower specimen surface. Increasing the load causes debonding initiation at the upper surface, followed by crack growth until macroscopic failure.

Measurement of the interfacial normal strength

During transverse loading of the specimens in the SEM, initiation of debonding and crack-growth can be observed at the surface of the specimens. In the previous section it was shown that cracks initiate at the surface, so that the local stress-strain state in the vicinity of the normally loaded interface can be measured with the presented measurement technique. Figure 5 shows a measured displacement field, and the initiation of debonding just after that load step.

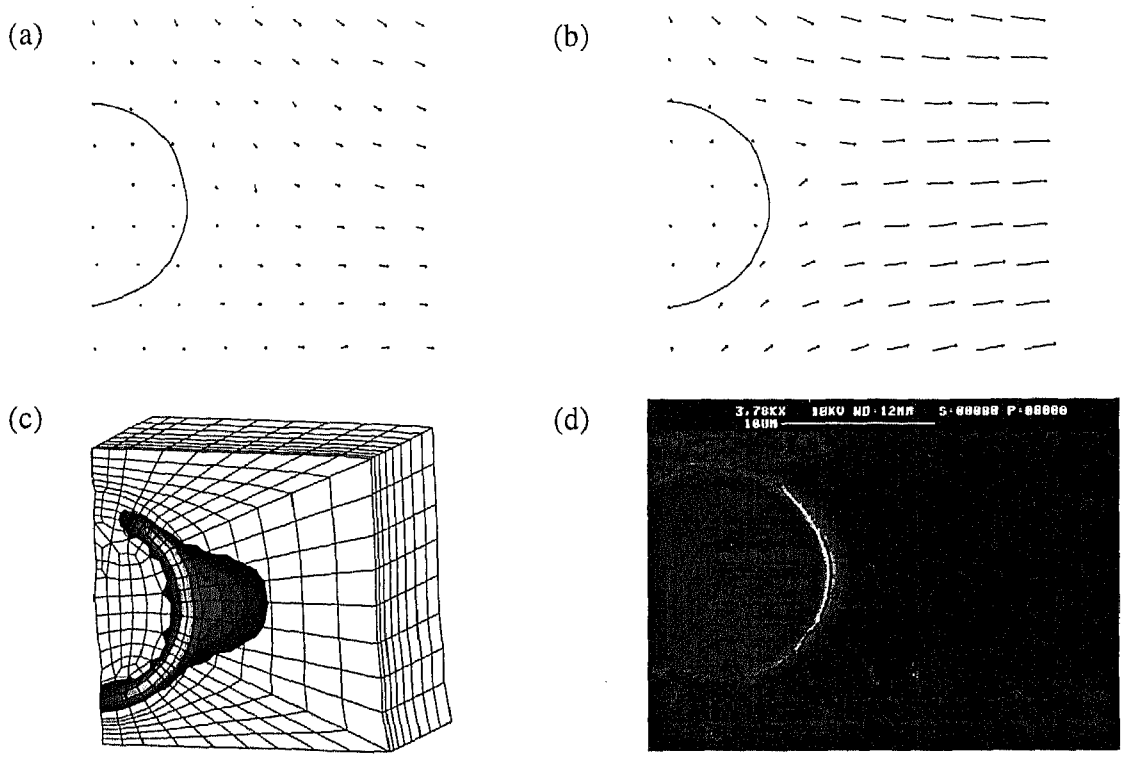

Figure 5: *Experiment in SEM: (a) displacements (×5) at small load, (b) displacement field before debonding (×5), (c) equivalent plastic strain, (d) initiation of debonding.*

The maximum measured displacement in this experiment was approximately 0.4 μm, resulting in plastic deformations around the fibre, which can be calculated in the numerical simulation of this experiment. In the finite element model, ideal elasto-plastic behaviour was assumed for the matrix material with a Young's modulus of 2.8 GPa and a Von Mises yield stress of 66 MPa. The measured displacements of the boundary markers at five load steps were used as boundary conditions for the simulations. The equivalent plastic strain in Figure 5c varies from 0.5% to 6% at the fibre-matrix interface. Although plastic deformation was observed around the fibre, macroscopic yielding of the single fibre specimen did not occur. The debonding crack grew over over the length of the fibre, after which crack propagation into the matrix was observed.

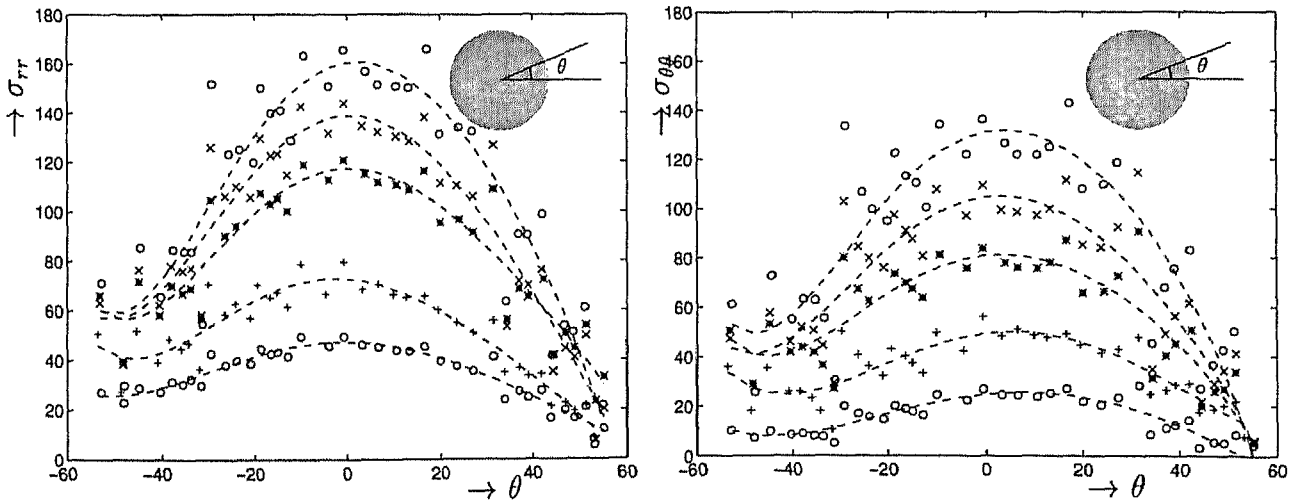

Figure 6: *(a) Radial and (b) tangential interfacial stresses for increasing global load.* o o o*=75 N,* + + +*=101 N,* * * *=153 N,* × × ×*=178 N,* o o o*=218 N.*

Because the interface stresses are the highest at the specimen surface, they were calculated at the surface of the 3-D finite element model. The interfacial stresses for five different load steps are given in Figure 6. For all load steps the radial stresses are higher than the tangential stresses, which results in initiation of a crack along the fibre surface. The trend of the interfacial stresses is in qualitative agreement with earlier studies [13]. For the five increasing load steps, the radial interface stress increases to a maximum value of 160 MPa at θ=5°.

In future research, the mechanical behaviour of the matrix material will be modelled with a more accurate constitutive model, and an interphase between fibre and bulk-matrix will be included.

Conclusions

A technique for the measurement of the interfacial normal strength has been presented. Numerical simulations of the stress-strain state at the surface of transversely loaded composites revealed a stress concentration towards the specimen surface, which causes debonding cracks to initiate at this location. Observation of crack initiation with an optical microscope supported these results. Measurement of deformation around a fibre in a single E-glass fibre specimen in combination with finite element modelling, in which the measurements are used as boundary conditions, resulted in radial and tangential interface stresses at the fibre surface. Based on the presented results, it can be concluded that the presented technique is a promising tool for the measurement of the interfacial normal stress.

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