Transverse failure initiation in polymer composites

Leif E. Asp
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Leif E. Asp
To

Mom and Dad
I enjoy to get an idea, not to have one

Lens P. Ås
1995
ABSTRACT

Transverse failure is one of the most important failure modes in polymer composites. The phenomenon often causes the first deviations from non-linear laminate behavior. Also, in pressure vessels and pipes, fluid leakage through a path of transverse cracks is often the limiting design criterion. In the present work, experimental and theoretical studies focused on the micromechanical level have been carried out. The objective was to investigate transverse failure initiation in the matrix. The other major mechanism of failure initiation, fiber/matrix debonding, was not considered. The triaxial nature of the matrix stress state in glass fiber/epoxy was confirmed by finite element analysis. Experimental results for glassy epoxies subjected to composite-like stress states demonstrated large reductions in strain to failure as compared with uniaxial loading. The triaxial stress state is therefore by itself a sufficient explanation for the low transverse strain to failure in polymer composites. Plastic yielding in the matrix was demonstrated not to be the cause of failure initiation. Instead cavity induced cracking was suggested as a failure mechanism. A criterion was proposed based on a critical value for the dilatational energy density. Comparison with experimental results for epoxies subjected to a variety of multiaxial load-cases supported the criterion. Additional support was obtained from comparison with experimental results in the literature for transverse failure of glass fiber/epoxy at different fiber contents. Although the epoxy matrix was different from those in the present study, general trends in data were supported by predictions based on the criterion and finite element analysis. Thermal residual stresses were found to be important for high fiber contents. Based on the criterion, a conservative estimate of composite strain to failure was obtained. This is reasonable since the criterion predicts initiation, not final failure. Based on the model, effects from changes in constituent properties were examined in a parametric finite element analysis. Fiber modulus was found to strongly influence transverse failure. Introduction of a third phase interphase between fiber and matrix was also investigated. Beneficial results on transverse failure strain caused by matrix initiation was observed for thin rubbery interphases.
PREFACE

During the years 1992-95 I have had the opportunity to work in the division of Polymer Engineering at the department of Materials and Manufacturing Engineering. My work has been in the area of failure initiation in transversely tensile loaded unidirectional glass fiber/epoxy composites. Initiation of transverse cracks in a unidirectional composite is, indeed, a complex and interesting phenomenon. The investigation has been restricted to concern matrix initiated transverse failure, only.

There are a number of people who deserves my gratitude as they have been important to my thesis work. I sincerely thank my advisor Professor Lars Berglund for his inspiration and support. Professor Berglund creates a magic atmosphere in which research becomes a pleasure, and I addicted. All my colleagues at the division of Polymer Engineering along with my fellow postgraduates are acknowledged for helping and for offering me their friendships. Special thanks are due to Professor Peter Gudmundson, at the Royal Institute of Technology, and Professor Ramesh Talreja, at Georgia Tech. for their scientific contributions and constant interest in my work.

Finally I would like to express my gratitude to my fiancée Pia for always supporting me and for putting up with my absence.

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Leif Asp
This thesis is based on the following papers:


II  L.E. Asp and L.A. Berglund, A biaxial thermo-mechanical disk test for glassy polymers, submitted to *Exp. Mech*.


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Introduction
INTRODUCTION

General Background
Composite materials used for aerospace applications usually consist of continuous high performance fibers, i.e., carbon fibers, in a polymer matrix, e.g., a thermoset such as epoxy or a thermoplastic such as PEEK. These types of composite materials are characterized by high specific longitudinal stiffness and strength. High performance composites are manufactured by stacking of thin prepreg plies of unidirectional fiber orientation. These plies are stacked at different fiber directions to a laminate that meets required mechanical property specifications.

Due to material heterogeneity and anisotropy, cracks parallel to the fibers will appear in plies oriented perpendicular to the load direction, even at low load levels. Intralaminar transverse cracking in off-axis plies is one of the first failure modes\textsuperscript{1,2}. Although final failure of polymer composites with continuous fibers usually involves fiber fracture, transverse cracking is also of great importance. Transverse cracks reduce laminate stiffness and are also known to initiate other types of damage such as local delamination and fiber fracture. The investigation by Spencer and Hull\textsuperscript{3} on pressurized glass fiber/polyester pipes provides an example of how transverse cracks form early in the deformation process of a composite structure. Onset of weepage due to transverse cracks occurred at transverse strains of about 0.2% whereas final failure occurred much later.

The effect of the matrix on transverse failure is of interest. In the literature, studies have compared the strain to failure in transverse tension and the strain to failure of the pure matrix loaded in uniaxial tension\textsuperscript{1,4-7}. Uniaxial matrix strain to failure varied from 1.5 to 70%. Transverse strain to failures of corresponding fiber composites were dramatically smaller and varied only in the range 0.2 to 0.9%. There are many explanations for the discrepancy in strain to failure between the transversely loaded composite and the uniaxially loaded neat resin. Transverse composite failure may initiate by debonding due to a weak interface, by presence of voids or in regions between fibers in contact with each other. Also, presence of stiff fibers causes a triaxial stress state\textsuperscript{8-10} as well as stress\textsuperscript{8} and strain\textsuperscript{11} magnification in the matrix. Agarwal and Broutman\textsuperscript{12} pointed out the state of stress to be the most important factor influencing initiation of failure. Highly magnified stresses or triaxial stresses may initiate failure in the matrix, even at low global composite loads. Transverse failure initiated by the triaxial stress state is likely in materials
with a strong and tough fiber/matrix interface. This thesis concerns transverse failure initiated in the matrix by presence of a triaxial stress state.

Several studies suggest the triaxial stress state to be important for initiation of transverse failure in the matrix at low strains\textsuperscript{10,13,14}. Gaggar and Broutman calculated a strain magnification from the triaxial stress state in a homogeneous matrix produced by the inhibition of Poisson contraction\textsuperscript{10}. A failure criterion based on distortion energy theory was chosen. By use of this criterion, the calculated strain to failure of a ductile matrix was 1.6\%. However, the strain to failure for a brittle matrix was predicted to be larger and as high as 3\%. Also, the analysis of de Kok et al.\textsuperscript{13} is of interest. Finite element calculations show high local strains in the matrix at low global composite strains, due to the triaxial stress state. The von Mises yield criterion is applied and local shear strains are shown to concentrate in a thin band. Local yielding occurs at low global strains. The matrix is assumed to be ideal elasto-plastic. Furthermore, experimental results by Nicholls\textsuperscript{14} demonstrate low strain to failure for polymers subjected to a biaxial tensile load.

In order to investigate the effects from the triaxial stress state, an understanding of the stress state in the matrix of a transversely loaded composite is needed. In literature, stress analyses on transversely loaded polymer composites have been performed analytically\textsuperscript{8} as well as numerically, using finite difference or finite element methods\textsuperscript{9,15-18}. The stress analyses of micromechanical models provide information such as; normal and shear stresses, maximum principal stress, and von Mises effective stress. Stress analysis combined with failure criteria predict failure initiation. The maximum principal stress criterion has been used for polymer composites\textsuperscript{9,19}. However, since the stress state in the matrix is highly triaxial, the maximum principal stress criterion will lead to erroneous and optimistic predictions. The von Mises criterion has also been applied to polymer composites\textsuperscript{9,15}. The von Mises effective stress based on the second invariant of the deviatoric stress tensor is used to predict failure initiation in regions with high shear stresses.

Analysis of a transversely loaded composite reveals variations in the matrix stress state with position. Regions with high shear stresses as well as regions with high dilatational stresses are active in the polymer matrix\textsuperscript{9}. It is therefore of interest to investigate the mechanical behavior of polymers subjected to either high dilatational or high distortional stresses. High distortional stresses lead to plastic yielding. The effect of stress state on yielding in glassy polymers is well understood\textsuperscript{20}. For glassy polymers, shear
driven yielding but also crazing\textsuperscript{21} has been emphasized with attention given to the associated influence of hydrostatic stress. A number of yield and craze criteria have been proposed for this purpose\textsuperscript{21-24}. The present study will focus on yield and failure criteria for epoxies subjected to different stress states.

In order to subject glassy polymers to high dilatational stresses, multiaxial tensile tests are needed. Several multiaxial tensile tests have been proposed in the literature\textsuperscript{14,25-26}. Mönch et al\textsuperscript{25} developed the biaxial tension cruciform test method which has been successfully applied to metals and composites. However, the cruciform test is difficult to perform, especially for brittle materials. The corners of the cruciform specimen act as stress raisers and are likely to initiate fracture. Sultan and McGarry\textsuperscript{26} performed biaxial tensile tests on pressurized epoxy tubes. In their study a pressurized silicone oil inside the cylinder provides the hoop stress while a tensile test machine applies the axial stress. Both in the cruciform and the pressurized tube test, complicated experimental set-ups are needed. A simpler test method was suggested by Nicholls\textsuperscript{14}. He applied biaxial tensile load in order to investigate the effect of the biaxial stress state on the strain to failure of neat resins. Nicholls clamped a short and wide specimen in a tensile tester, creating a biaxial stress state. However, the stress state is difficult to analyze as clamping conditions are critical in this type of test. Nevertheless, all test methods described above subject the specimens to biaxial tensile stress states. To further enhance dilatational stresses and reduce distortional contributions to the stress field, triaxial tensile tests are desired. The poker-chip test method subjects the specimen to a triaxial tensile stress state\textsuperscript{27,28}. In this test a poker-chip shaped specimen is bonded between two rigid cylindrical substrates. Load is applied in the direction of the cylinders. As a consequence, a triaxial tensile stress state is activated in the polymer specimen. The poker-chip test was applied to rubbers in the late fifties and the sixties\textsuperscript{27,28}.

In the present study glassy epoxies were subjected to an almost equitriaxial tensile (hydrostatic tensile) stress state by the poker-chip test method. Results suggest failure to initiate by cavity-induced brittle failure rather than by yielding. For this reason a failure criterion based on a critical value for the dilatational energy density was developed. Predictions made based on the dilatational energy density criterion are supported by experimental data\textsuperscript{9} for transverse stresses at and strains to transverse failure initiation in glass fiber reinforced epoxies.
Objective of the thesis
The objective of the thesis is to determine the effects of the triaxial stress state in the matrix on transverse failure initiation in continuous glass fiber reinforced epoxies.

Summary of the papers
To evaluate the influence of triaxial stresses on transverse crack initiation in the matrix of a polymer composite, analysis of the stress state is needed. In Paper I a preliminary stress analysis of a square (quadratic) fiber distribution is performed using FEM (Finite Element Modelling). The analysis does not take residual thermal stresses into account. A region of high dilatational stress is observed at the fiber poles, see Paper I. This triaxial tensile stress state is mimicked in the poker-chip test. The poker-chip strains to failure in the primary loading direction were 0.5 to 0.8 %, whereas strains to failure in the uniaxial tests were 1.8 to 7 %. The triaxial stress state in composite matrices may therefore by itself be a sufficient explanation for low values of transverse composite strains to failure. Additional tests were required to evaluate the influence of dilatational stresses on the strength of polymers. A method for testing glassy polymers under biaxial tensile loading was developed, see Paper II. In the test method, a disk of epoxy is bonded between a steel ring and a steel disk. The test method is designed to subject the specimen to a biaxial tensile stress state under cooling. An approximate analytical model was developed for stress analysis of the disk under cooling. The results from this test in combination with those from the poker-chip test provide failure data for glassy polymers subjected to high dilatational stresses.

In Paper III a dilatational energy density criterion is developed for prediction of failure initiation in glassy polymers subjected to a composite-like stress state. In the analysis of the poker-chip data, additional thermal residual stresses are taken into account. This dilatational energy density criterion is found to give critical energy densities for a triaxial composite-like stress state that correlate well to those of biaxial tensile load cases. Also, as polymers are known to be sensitive to hydrostatic pressure, the possibility of yield initiated failure is examined. As a result, yield initiated failure in glassy epoxies subjected to a composite-like stress state is ruled out. The results therefore imply that cavity-induced brittle failure in glassy epoxies subjected to a composite-like stress state is indeed taking place and can be predicted by the suggested dilatational energy density criterion.

Numerical analyses of three different fiber distribution geometries are performed in Paper IV. The objective is to predict transverse stress at and
strain to failure initiation of unidirectional glass fiber/epoxy (GF/EP) composites. All analyses are restricted to glass fiber composites because of their isotropy. All analyses in Paper IV include thermal residual stresses due to differences in thermal coefficient of expansion between fiber and matrix. Transverse failure initiation is predicted by the dilatational energy density and the von Mises yield criteria. In all cases, independent of fiber distribution or fiber volume fraction, the numerical results suggest failure to initiate due to high dilatational energy density. The transverse strengths of a square (quadratic) fiber distribution are compared to experimental data by deKok. The comparison supports the ability of the dilatational energy density criterion to predict transverse failure initiation in continuous GF/EP composites.

Paper V contains modeling results based on FEM-analysis and the dilatational energy density criterion. The objective is to study effects of constituent properties on failure initiation as predicted by the model. It is assumed that the fiber/matrix bond remains intact. In the first part, a parametric study is conducted in order to demonstrate how mechanical properties of the fibers affect the stress at and strain to failure initiation. Specific examples for carbon fiber and glass fiber reinforced epoxy is presented. In the second part, a parametric study is conducted to demonstrate how mechanical properties and thickness of a third phase interphase affect the stress at and strain to failure initiation of a composite loaded in transverse tension. Specific examples of rubber, thermoplastic, and intermediate modulus interphases are presented. Numerical stress analysis by the finite element method is conducted on a square fiber array. The von Mises yield criterion and the dilatational energy density criterion are applied to locate the zones of yielding and cavitation-induced brittle failure. Also, the position of maximum radial stresses at the fiber/interphase and interphase/matrix interfaces are examined. Fiber modulus is shown to have a large influence on transverse composite stress at and strain to failure initiation. Introduction of a third phase interphase between fiber and matrix is shown to increase stress at and strain to failure initiation for thin, low modulus, high Poisson's ratio interphase composites. Hence, in order to improve failure initiation properties, application of thin rubber interphases is suggested. Finally, position and mode of failure initiation is found to depend strongly on fiber and interphase properties.
REFERENCES


Paper I
Effects of composite-like stress state
on the fracture of epoxies

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Abstract
The strain to failure of a transversely loaded composite is much lower than for the pure matrix in uniaxial tension. Several studies of composites suggest the triaxial matrix stress state as one of the explanations. In order to investigate this experimentally, a triaxial tensile test previously used for rubbers (poker-chip test) was successfully applied to four epoxies in the glassy state. The chosen specimen geometry mimicked the most severe stress state in the matrix as determined by finite element analysis of a transversely loaded glass fiber/epoxy (GF/EP) composite. The poker-chip strains to failure in the primary loading direction were 0.5 to 0.8 %, whereas uniaxial strains to failure were 1.8 to 7 %. The triaxial stress state in composite matrices may therefore by itself be a sufficient explanation for low values of transverse composite strains to failure.

1. INTRODUCTION

Final failure of polymer composites with continuous fibers usually involves fiber fracture. However, transverse cracking parallel to the fiber direction is also of great importance. Transverse cracks reduce laminate stiffness and are also known to initiate other types of damage such as local delamination and fiber fracture. The investigation by Spencer and Hull on pressurized glass fiber/polyester pipes provides an example of how transverse cracks form early in the deformation process of a composite structure. Onset of weepage

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due to transverse cracks occurred at transverse strains of about 0.2% whereas final failure occurred much later.

The effect of the matrix on transverse failure is of interest. Several studies have compared the strain to failure in transverse tension and the strain to failure of the pure matrix loaded in uniaxial tension\textsuperscript{2-6}. Uniaxial matrix strains to failure varied from 1.5 to 70%. Transverse strain to failures of corresponding fiber composites were dramatically smaller and varied only in the range 0.2 to 0.9%.

We suggest a division of explanations for this phenomenon into two categories. The first category of explanations is based on the more severe stress state in the composite matrix or at the fiber/matrix interface as compared with the uniaxial pure matrix case. The non-uniform fiber distribution in commercially processed materials magnifies this effect. The second category of explanations is based on the existence of material flaws such as voids or interfacial debonds. This explanation can also include a consideration of the stress state in a composite.

As a starting point for an analysis of transverse failure, we consider two major mechanisms for failure initiation. One is fiber/matrix interfacial debonding. Debonding is the first event and final failure occurs by linking of debonded sites. Debonding during transverse loading has been demonstrated for glass fiber/polyester\textsuperscript{3}. The other major mechanism is failure initiation in the matrix. This mechanism is most likely in materials with a strong and tough fiber/matrix interface. It is the mechanism to which the present study relates. A schematic of the two mechanisms is presented in Figure 1.

Let us consider theoretical treatments of transverse failure with the exception of studies related to failure initiation by interfacial debonding. Christensen and Rinde analyzed the transverse strength using macromechanical fracture mechanics\textsuperscript{5}. Although this approach has some theoretical justification, the inherent flaw size of the material is used as a fitting parameter. The approach is not applicable to tough matrices\textsuperscript{7}. Another problem is lack of connection between such a macroscopic model and micromechanical models. Micromechanical models are desirable since they may improve our understanding of the physical mechanisms involved.

Kies considered non-uniform strain distribution in the matrix using a square array of fibers\textsuperscript{8}. The strain concentration factor is an average quantity found to be a function of fibre volume fraction and fiber and resin moduli. From his equation, a number for the strain magnification can be obtained as a
function of fiber volume fraction. Chamis developed a related model, where the magnification in matrix stress due to the fibers is expressed. His model relates the transverse composite strength to the uniaxial matrix strength. Garrett and Bailey used the theory by Kies to explain the relative insensitivity of transverse composite strain to failure to uniaxial strain to failure of the matrix. The major limitation of such approaches is obvious from the final result. It is in the form of one average strain or stress magnification number, unable to express local changes in strain with position.

More advanced stress analyses have also been performed. Tirosh et al determined the stress distribution around a single fiber embedded in a linear elastic matrix. With the center of the fiber as a starting point, the highest stress was found at a distance of 1.2 times the fiber radius. The stress distribution in a single fiber material is, however, very different as compared with a real composite. Greszczuck developed an approximate analytical elasticity solution for the stress distribution around an idealized distribution of fibers. In a widely quoted study, Adams and Doner used finite difference analysis in order to solve the plane elasticity problem for a square array of circular fibers. The purpose, however, was to calculate the transverse modulus rather than to estimate consequences for transverse failure.

Gaggar and Broutman calculated a strain magnification from the triaxial stress state in a homogeneous matrix produced by the inhibition of Poisson contraction. A failure criterion based on distortion energy theory was chosen. By use of this criterion, the calculated triaxial strain to failure of a ductile matrix was 1.6%. However, the triaxial strain to failure for a brittle matrix was predicted to be larger and as high as 3%.

For transverse failure initiation in the matrix, the analysis of De Kok et al. is of interest. Finite element calculations show high local strains in the matrix at low global composite strains. The von Mises yield criterion is then applied and local shear strains are shown to concentrate in a thin band. Local yielding occur at low global strains. The matrix is assumed to be ideal elastoplastic.

Still, none of the existing micromechanical theories and/or experimental studies are able to satisfactorily explain the role of the matrix in transverse composite failure and further work is needed. As part of the motivation for the present study, transverse failure is assumed to initiate in the matrix. Experimental studies support the possibility of such a mechanism. Several fractographic investigations report on fracture surfaces where the fibers are
covered by matrix material, see e.g. Bascom et al. (CF/epoxy)\textsuperscript{15} and Purslow (CF/PEEK)\textsuperscript{16}. In CF/PEEK, debonding appears to be unusual during transverse cracking, see Figure 2 where a crack even propagated through some carbon fibers.

Several studies suggest the triaxial matrix stress state to be important for initiation of transverse failure in the matrix at low strains\textsuperscript{13,14}. The effect of triaxial stress states on polymer failure is therefore of interest. Biaxial polymer tests have been reported to result in low polymer strain to failure\textsuperscript{17}. In transverse composite tests, the possibility of crack initiation from a material flaw cannot be disregarded. For this reason it was desirable to find a test method where a pure polymer can be subjected to a triaxial stress state. The poker-chip test is such a method and was applied to rubbers by Gent and Lindley\textsuperscript{18} and by Lindsey\textsuperscript{19}.

The objective is to investigate if the poker-chip test is applicable to glassy epoxies and, if so, compare uniaxial tensile data with those from triaxial poker-chip experiments. Provided failure initiation in the matrix is considered, the importance of the triaxial stress state in the matrix may then be estimated.

2. EXPERIMENTAL

2.1. Materials
The chemical structures of the material components for the four epoxy systems are presented in Figures 3 and 4. In three epoxy systems, presented in Figure 3, the epoxy component is DGEBA, (DER 332, Dow Chem Co). Each system has a different curing agent: (i) DETA (DEH 20, Dow Chem Co), (ii) MHPA, (HY 917, Ciba Geigy) and a methyl imidazole (MI) accelerator, (DY 070, Ciba-Geigy), (iii) APTA, (Jeffamine T-403, Texaco Chem Co). In Figure 4 the fourth system is presented, TGDDM (MY 720, Ciba Geigy) cured by DDS, (HT 976, Ciba-Geigy). TGDDM is an aromatic epoxy and DDS is an aromatic amine.

2.2. Casting procedure
The four different systems were carefully mixed by hand, vacuum was applied to the mixtures ten minutes before casting. The mixtures were then poured into a fluoropolymer coated aluminium mold. Material compositions and cure schedules are presented in Table I.
2.3. Specimen fabrication and testing methods
The cast plates were removed from the mold and machined to the specimen dimensions required for mechanical testing. The specimens designed for uniaxial testing were milled to the dimensions suggested by ASTM D638M-81, type II, in a computer controlled milling machine. The strains in uniaxial tests were measured by a 50 mm gauge length extensometer. A minimum of seven specimens of each material was used.

For the multiaxial poker-chip method, a thin circular specimen was bonded between two aluminium rods and loaded to failure, see Figure 5. The specimens were first cut from the plate to squares 30 mm x 30 mm and bonded to the aluminium rods by 73M OST epoxy adhesive film from American Cyanamid Co. Prior to bonding, the aluminium was degreased and etched in chromic acid. The epoxy surfaces to be bonded were ground and then degreased by the use of acetone. For the strongest epoxy, DGEBA/APTA, the epoxy itself was used as an adhesive since the adhesive film failed prematurely. After bonding, the specimens were ground and polished into circular shape. The diameter and thickness were 30 and 4 mm respectively (aspect ratio 7.5). The aluminium rods had a length of 100 mm. Joints in the free ends of the rods were connected to gripping extensions in a Dartec test machine. The strains in the multiaxial tests were measured over a distance of 50 mm by a sensitive extensometer (full range of displacement ±0.5 mm). The strain to failure of the specimen was calculated by subtraction of strains in aluminium rods. Strain rates for uniaxial tests and poker-chip tests were 1 % per minute and 0.2 % per minute respectively. All tests were performed at ambient conditions.

For the uniaxial tests, Young’s modulus E was determined as the secant modulus at 0.9 % strain. For the poker-chip tests, the apparent tensile modulus \( E_A \) was calculated as the secant modulus at 0.1 % strain. The poker-chip data in Table III are based on 30 specimens out of 65. Specimens with failure at the specimen/substrate interface were discarded. Data from a minimum of 7 specimens of each material are reported. For DGEBA/APTA, 3 out of 7 specimens failed at the interface. Since these specimens had higher strengths than those with true material failure, the data was used.

2.4. Fractography
Fracture surfaces were studied visually and in a scanning electronic microscope (SEM). The fracture surfaces were coated with carbon/gold
Asp; Paper 1

(C/Au) in a Blazers SCD 050 sputter coater. The SEM was a CAMSCAN SH-80D, operating at 30 kV accelerating voltage.

3. THEORETICAL ANALYSIS

3.1. FEM-analysis of transversely loaded composites
The Finite Element Method (FEM) was used to determine the stress state in a transversely loaded composite. The commercially available ABAQUS system was used for the FEM analysis. The generated cell contained a fiber surrounded by resin and was a unit element in a square array of fibers. Elements of general plain strain were used to generate the mesh. Periodicity was taken into account by coupling of node-displacements. For nodes on the sides perpendicular to the loading direction, the difference displacement of each opposite node-pair was equal to an initial displacement factor. The remaining two sides were kept straight and free to move.

The Young's modulus of the glass fiber in the model was $E_f = 76$ GPa, Poisson's ratio was $\nu_f = 0.2$. For the resin $E_m = 3.0$ GPa and $\nu_m = 0.34$. The composite material in the model had a fiber volume fraction of 50.2%. Calculations resulted in a composite Poisson's ratio of 0.31, close to the results presented by Adams and Doner\textsuperscript{12}. A triaxial stress state acts in the matrix of the composite. The ratio of the stress component magnitudes is approximately $1:1:2$ ($x:y:z$), where $z$, the largest stress component, is in the loading direction. This ratio varies with position in the composite. The presented results refer to a material volume in the vicinity of the fiber/matrix interface at the fiber center-line. Within this volume, the stress magnification is approximately $\frac{\sigma_z}{\sigma_z^{\text{ave}}}=1.8$, which is close to the results obtained by Adams and Doner\textsuperscript{12}. The local stress in the $z$-direction at the position $(x:y:z)$ is here denoted $\sigma_z$ and the global average stress in the $z$-direction is $\sigma_z^{\text{ave}}$.

3.2. Triaxial test
Ideally, we would like a triaxial test which mimics the matrix stress state determined in the previous section. An analysis of the poker-chip load-case in Figure 5 is therefore performed. The poker-chip test specimen is a circular disk with large diameter. It is bonded to rigid substrates and tested in monotonic tension. The test method has previously been used for rubbers\textsuperscript{18,19}. In the analysis of reference 18, the specimen was assumed to be infinitely thin. This implies vanishing in-plane strains and a non-zero...
homogeneous strain in the z-direction. With this configuration the stress field becomes

$$\sigma_z = \frac{E(1-\nu)}{(1-2\nu)(1+\nu)} \varepsilon_z$$  \hspace{1cm} (1)$$

$$\sigma_r = \sigma_\theta = \frac{\nu}{(1-\nu)} \sigma_z$$  \hspace{1cm} (2)$$

This analysis is not sufficient for a finite ratio between thickness and diameter. This problem was addressed by Lindsey, Schapery et al. for the purpose of polyurethane tests\textsuperscript{21}. For polyurethane\textsuperscript{19}, the stress state was almost purely hydrostatic, 1:1:1 (\(\sigma_x:\sigma_y:\sigma_z\)). This was due to the Poisson’s ratio of close to 0.5 and the aspect ratio (specimen diameter to thickness) which was larger than 10.

A more detailed analysis of the poker-chip method was also presented by Lindsey, Schapery et al\textsuperscript{21}. Figure 5 shows a circular disk of normalized radius (a) with its axis in the z-direction, and faces \(z=\pm1\). Normalization is with respect to the half thickness (\(t/2\)) of the disk. Notice that the half thickness is taken as unity, thus the aspect ratio is equal to the normalized disk radius (a). The disk is assumed to be loaded by increasing the thickness by \(2\epsilon\), see Figure 5. The following equations were assumed to describe the normalized radial and axial displacements \(u\) and \(w\) respectively:

$$u = -(1-z^2) \cdot g(r)$$

$$w = \varepsilon \cdot z$$  \hspace{1cm} (3)$$

where \(z\) is normalized thickness and \(g(r)\) is an unprescribed function of normalized radial position. Notice that both \(z\) and \(r\) are normalized with respect to the half thickness of the disk. The strains corresponding to these displacements are
Lindsey, Schapery et al. determined the function \( g(r) \), from the condition that the \( z \)-integrated equilibrium equation for the radial direction is to vanish, and used the equations (4a-d) to calculate the average stresses through the thickness of the specimen. The analytical solutions of the stress components are somewhat complicated. A simplified form of the analytical solutions was also derived. The stress components are in simplified form

\[
\begin{align*}
\sigma_r &= \frac{\partial u}{\partial r} = -(1-z^2) g'(r) \\
\sigma_\theta &= \frac{u}{r} = -(1-z^2) \frac{g(r)}{r} \\
\tau_{rz} &= \frac{\partial u}{\partial z} + \frac{\partial w}{\partial r} = 2g(r) \cdot z
\end{align*}
\] (4a-d)

\[
\begin{align*}
\varepsilon_r &= \frac{\partial u}{\partial r} = -(1-z^2) g'(r) \\
\varepsilon_\theta &= \frac{u}{r} = -(1-z^2) \frac{g(r)}{r} \\
\gamma_{rz} &= \frac{\partial u}{\partial z} + \frac{\partial w}{\partial r} = 2g(r) \cdot z
\end{align*}
\]

\[
\begin{align*}
\sigma_r &= \frac{\sigma_r}{E\varepsilon} = \frac{3v}{(1+v)} \cdot \frac{1}{3(1-2v)} \left[ \frac{1}{I_0} \left( \frac{rV3(1-2v)}{aV3(1-2v)} \right) \right] \\
\sigma_\theta &= \frac{\sigma_\theta}{E\varepsilon} = \frac{1}{(1+v)} \left[ \frac{1}{2I_0} \left( \frac{rV3(1-2v)}{aV3(1-2v)} \right) \right] \\
\tau_{rz} &= \frac{\tau_{rz}}{E\varepsilon} = \frac{3v}{(1+v)} \cdot \frac{1}{3(1-2v)} \left[ \frac{1}{I_0} \left( \frac{rV3(1-2v)}{aV3(1-2v)} \right) \right] \cdot z
\end{align*}
\] (5-7)

where \( a \), \( r \), and \( z \) are normalized disk radius, radial position, and position in thickness direction respectively. All parameters were normalized with respect to the half thickness of the disk. \( E \) is Young's modulus, \( v \) Poisson's ratio, \( I_0 \) and \( I_1 \) are modified Bessel functions, \( \sigma_r, \sigma_\theta, \sigma_z \) are normal stresses in \( r-, \theta-, \) and \( z \)-directions in a cylindrical coordinate system, \( \tau_{rz} \) is the shear stress in the \( r/z \)-direction. The stress distribution according to equations (5-6) at \( z=0 \) is presented in Figure 6.

Equations (5-7) are simplified. However, Lindsey, Schapery et al. concluded these approximations to be sufficiently accurate in the central region where \( r/a \leq 0.6 \). The analysis close to the edge is less accurate. FEM analyses by Adams et al. have shown stress concentrations to be present at the interface (\( z=\pm 1 \)) corners. Since adhesive failure is of no interest for our
purpose, we need to establish cohesive failure initiation in the central region, away from the interface.

The difference between the poker-chip and the related butt joint test is that the aspect ratio of the poker-chip specimen is much smaller. The purpose is to test the polymer material rather than the adhesive bond strength.

For each choice of aspect ratio of the poker-chip specimen, the stress distribution will be a function of the normalized radial position $r$. Figure 6 shows the stress distribution in the specimen to be fairly homogeneous from the center to approximately 60\% of the radial distance ($r/a=0.6$). The $z$-stress is almost twice as large as the other two. Note that $\sigma_z$ is normalized with respect to Young's modulus $E$. The average of the normalized $\sigma_z$ therefore becomes larger than one since $E$ is smaller than the apparent modulus of the specimen, $E_A$, see equation (8).

The shear stress is zero at $z=0$. It has its maximum value at the interface, $z=\pm1$, but it is significantly lower than the normal stress.

The aspect ratio of epoxy poker-chip specimens was chosen as 7.5 which induces a ratio of the stresses $x:y:z$ of close to 1:1:2 in the central region. Hence, the stress state is similar to that locally in the matrix of a transversely loaded GF/EP composite. Thermal stresses generated during cool-down of the epoxy after bonding to the aluminium substrates at elevated temperature, are not included in the analysis. Their magnitude is estimated to be similar in the poker-chip specimen as in the composite. However, if a quantitative failure criterion is sought, thermal stresses have to be included. They do not contribute to the apparent stress at failure, their effect is to increase the stresses in the plane and change the proportions between the stress components.

From the poker-chip test, the apparent modulus $E_A$ may be determined as

$$E_A = \frac{\sigma_{zA}}{\varepsilon_z} = \frac{2\pi \int_{r=0}^{a} \sigma_z r dr}{\pi a^2 \varepsilon_z}$$

where $\sigma_{zA}$ is the average stress over the bonded surface and $\varepsilon_z$ is the applied strain in the $z$-direction. Notice that $\sigma_z$ in equation (8) is in its unsimplified form expressed by Lindsey, Schapery et al\textsuperscript{21}. This can be used to write equation (8) differently, thus:
\[
\frac{E_A}{E} = \frac{3v}{1 + v} \left( \frac{1}{3(1 - 2v)} \right) \left[ 1 - \frac{2I_1(a\sqrt{M})}{a\sqrt{M}I_0(a\sqrt{M})} \right]
\]

\[
+ \frac{1}{1 + v} \left[ 1 + \frac{2I_1(a\sqrt{M})}{a\sqrt{M}I_0(a\sqrt{M})} \left[ 1 - \frac{I_1(a\sqrt{M})}{a\sqrt{M}I_0(a\sqrt{M})} \right] \right] \frac{1 + 1 - 2v}{v} \left( 1 - \frac{I_1(a\sqrt{M})}{a\sqrt{M}I_0(a\sqrt{M})} \right)
\]

(9)

where \( M = \frac{3}{2} \frac{(1 - 2v)}{(1 - v)} \)

The modulus \( E \) can be obtained experimentally from a uniaxial test and \( E_A \) from the poker-chip test. Equation (9) can then be used to calculate Poisson’s ratio.

4. RESULTS AND DISCUSSION

4.1. Uniaxial test

Although the primary interest is in results from triaxial tests, reference results from uniaxial tests are needed. Uniaxial tensile test data for four epoxies are presented in Table II and Figure 7. Strains to failure are in the range 1.8 to 7.0 %. The epoxies cured by aliphatic amines show 6 to 7 % strain to failure. Before failure, DGEBA/APTA showed significant localized yielding in the form of necking. For a few cases, our data differ from results previously reported in the literature, see Table II, probably due to differences in specimen geometry, strain rate and/or specimen preparation.

The behavior of the four epoxies is in agreement with current understanding of the effect of molecular structure on uniaxial tensile response. As expected from its densely cross-linked network, TGDDM/DDS has low strain to failure, 1.8 %, low \( G_{IC} \) and high \( T_g \), see Table II. The Young’s modulus is the highest (3.8 GPa) for this system. The high modulus of densely crosslinked aromatic thermosets is due to high density of secondary bonds from efficient packing of the aromatic segments. DGEBA/DETA has low crosslink density and low \( T_g \), see Table II. It also has the lowest modulus of the investigated systems, 2.1 GPa, indicating inefficient molecular packing and possibly some viscoelastic effects. The
fracture textures are in agreement with results by Morgan and O'Neal\textsuperscript{24}. DGEBA/DETA fails after significant deformation whereas TGDDM/DDS shows brittle fracture at low strain.

The observed differences in uniaxial behavior create interest in a comparison between the investigated epoxies in triaxial loading.

4.2. Triaxial test

The geometry of the poker-chip specimen was chosen in order to mimic the matrix stress state in a GF/EP composite subjected to transverse loading, as explained previously. The poker-chip test was used for natural rubber by Gent and Lindley\textsuperscript{18} and for polyurethane rubbers by Lindsey\textsuperscript{19}. However, compared with rubbers, polymers in the glassy state have significantly higher stiffness and strength. Higher stress-levels were therefore expected, accompanied by risk for bond failure at the interface between the specimen and the metal substrate. After some experimentation, the test procedure described in the experimental section was found to give satisfactory results.

The experimental data are presented in Table III (with uniaxial data) and Figure 8. All four epoxies exhibited a linear apparent stress-strain relation until failure, as is common with transverse composite data. It is important to notice that the apparent stress is different from the true stress, see the previous theoretical discussion. A dramatic reduction in strength and strain to failure is observed as compared with typical uniaxial tensile data. Lindsey found an increase in strength and a decrease in strain to failure of a rubber, as the number of stress-axes increases\textsuperscript{19}. The significant difference in Poisson's ratio between rubbers and epoxies (0.5-0.3) is an important factor. For this reason, quantitative comparisons between epoxy-rubber data are not very meaningful.

As an illustration of the stress state effect on neat epoxies, the apparent triaxial stress-strain curve and uniaxial test data are presented in Figure 9 for DGEBA/MHPA.

The epoxies can be divided into two groups with respect to their mechanical behavior. Epoxies cured with aliphatic curing agents, DGEBA/DETA and DGEBA/APTA, show similar stress-strain behavior with strains to failure around 0.8%. Epoxies cured with aromatic and cycloaliphatic curing agents, DGEBA/MHPA and TGDDM/DDS, also showed almost identical stress-strain behavior. These epoxies failed at strains of 0.5%. The lower strain to failure of more brittle epoxies is not unexpected for a composite-like stress state. Data on transverse strain to failure in GF/EP
generally show matrices with low fracture toughness, such as DGEBA/MHPA and TGDDM/DDS (see Table II), to have lower strain to failure than tougher matrices.

Typical data for transverse strain to failure in GF/EP fall in the range 0.3-0.8 % if the fiber volume fraction is 0.5-0.6. The similarity in stress states between the poker-chip specimen and the most severely stressed position in the matrix is a probable explanation for the fact that poker-chip data (0.5-0.8 %) correlate with composite data for this case.

Poker-chip data can be used in Equation (1) in order to estimate the Poisson's ratios of the four epoxies. Poisson's ratios between 0.28 and 0.36 result from such calculations if the apparent modulus is determined as the secant modulus at 0.1 % strain from the poker-chip test data. A more careful evaluation is not meaningful since the calculated Poisson's ratio is very sensitive to small variations in the measured moduli. It is still encouraging to find calculated values in agreement with epoxy data which were in the range 0.32-0.35.

4.3. Fracture mechanisms in triaxial test

In order to further verify the accuracy of the test method, crack initiation at the metal/polymer interface needs to be excluded. Fractographic studies may be used to confirm crack initiation in the interior rather than at either the edge or the substrate/specimen interface. Typical fracture surfaces of the four epoxies are presented in Figure 10.

Fracture surfaces of the two epoxies with the highest fracture toughnesses (see Table II) are presented on the left hand side, DGEBA/DETA and DGEBA/APTA (aliphatic curing agents). For these materials, all reported data are for failure initiation in the central, interior region of the specimens as concluded from fractography studies. Characteristic "river" markings on the fracture surfaces confirmed crack nucleation and growth within the specimen, see Figure 11. To be absolutely sure, for DGEBA/DETA a total of 23 specimens were tested where central, interior failure initiation was confirmed.

Two different types of fracture surfaces were found in DGEBA/DETA. One showed cracks to coalesce from several initiation points. The other showed the development of one single crack from one initiation point, as in DGEBA/APTA.

The related butt joint tests have much higher specimen aspect ratios and here failure commonly initiates at the edge of the specimen/substrate
interface. Adams et al. reported butt joints of brittle epoxy systems to show adhesive failure at the interface corners whereas butt joints made of plasticised epoxy failed away from the circumference. They observed failure initiation at or close to the interface for both materials. In a previous paper they showed that no stress concentrations are to be expected at the interface in the central parts of the butt joints. With a tough adhesive, the sensitivity to stress concentrations is lowered. In the present investigation, a tough adhesive film was therefore used to bond the poker-chip specimens to the substrate.

In his early poker-chip study, Lindsey presents polyurethane fracture surfaces much like those of DGEBA/DETA. In both materials, the central part of the surface has a large number of nucleation points and crack growth is presumably slow. Outside the coarser central region, no nucleation points are present, as expected for the final stages of crack growth. Parabolic marks indicate an initial crack growth direction from the center radially towards the edge. Gent and Lindley observed internal cavities at stresses as low as 15% of the final strength. The cavities grew until they filled the entire thickness of the specimen. Lindsey studied failure mechanisms through transparent substrates during loading of polyurethane rubbers. His results show cavity initiation in the vicinity of the central region. The cavities grow to fill the entire thickness of the specimen. At this stage two sharp cracks appear at the extremities of a bubble and propagate perpendicular to the direction of maximum principal load.

For the two epoxies on the right hand side in Figure 10, DGEBA/MHPA and TGDDM/DDS (aromatic and cycloaliphatic curing agents), the fracture surfaces are very rough. In several specimens, initiation points were identified close to, but not at the interface, see DGEBA/MHPA in Figure 10. The crack appeared to rapidly have grown at a small angle with respect to the z-direction and then to have followed the interface. However, for most DGEBA/MHPA and TGDDM/DDS specimens, initiation points could not be located at all. The evidence for crack initiation in the interior of these materials is therefore not conclusive. The difficulty to locate initiation points is related to the low fracture toughness of DGEBA/MHPA and TGDDM/DDS, see Table II. Small sizes of mirror- and smooth zones in glassy materials have been correlated with low fracture toughness. For cases where we could locate these zones, their size was much smaller than for the other epoxies.
Fractography demonstrates crack initiation to take place in the central, interior region of the specimens for DGEBA/DETA and DGEBA/APTA. For brittle epoxies, TGDDM/DDS and DGEBA/MHPA, several specimens show initiation points in the central, interior region but the evidence is not conclusive.

5. CONCLUSIONS

Previous studies suggest the triaxial matrix stress state to be important for the low values of transverse composite strains to failure\textsuperscript{13,14}. However, insufficient experimental data are available to verify this. A triaxial tensile test previously used for rubbers (poker-chip test) was therefore applied to four epoxies in the glassy state and proved successful. The specimen geometry mimics the most severe stress state in the matrix. This stress state is chosen on the basis of finite element analysis of a transversely loaded GF/EP composite (square array of fibers, fiber volume fraction 0.5). The triaxial strains to failure in the primary loading direction are 0.5 to 0.8 % whereas uniaxial strains to failure are in the range 1.8 to 7 %. The triaxial stress state in composite matrices may therefore by itself be a sufficient explanation for the low transverse composite strains to failure. Fiber/matrix debonding, pre-existing material flaws and non-uniform fiber distribution are likely to further reduce initiation values of transverse strain to failure. Although numerous attempts are available in the literature, uniaxial matrix strain to failure cannot be expected to generally correlate with initiation values of transverse strain to failure. Differences in stress state and the geometry of the problem will result in vastly different failure mechanisms.

Acknowledgements

Mr. Johnny Grahn is gratefully acknowledged for his SEM-work. The study was partially financed by the Swedish National Board for Technical Development (NUTEK) and The Swedish Institute of Composites (SICOMP).
REFERENCES


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### Table I. Cure schedule and material composition.

<table>
<thead>
<tr>
<th>Resin system</th>
<th>Cure</th>
<th>Post-cure</th>
<th>Material Composition (percent by weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>2 h/20 °C</td>
<td>24 h/102 °C</td>
<td>11.9%</td>
</tr>
<tr>
<td>DGEBA/MHPA</td>
<td>2 h/110 °C</td>
<td>6 h / 140 °C</td>
<td>95.4%, (0.5% MI)</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>16 h/60 °C</td>
<td></td>
<td>44.8%</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>4 h/150 °C</td>
<td>1 h / 200 °C</td>
<td>44%</td>
</tr>
</tbody>
</table>

### Table II. Experimental results and standard deviations from uniaxial tensile tests. $\sigma_u$ is the nominal ultimate stress. Literature data: DGEBA/DETA\textsuperscript{24,34}, DGEBA/MHPA\textsuperscript{27}, DGEBA/APTA\textsuperscript{28-30} and TGDDM/DDS\textsuperscript{24,31-33}.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>DGEBA/DETA</th>
<th>DGEBA/MHPA</th>
<th>DGEBA/APTA</th>
<th>TGDDM/DDS</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E$ (GPa)</td>
<td>present</td>
<td>2.07±0.15</td>
<td>2.92±0.12</td>
<td>2.93±0.13</td>
</tr>
<tr>
<td>$E$ (GPa)</td>
<td>[literature]</td>
<td>-</td>
<td>3.08</td>
<td>3.24</td>
</tr>
<tr>
<td>$\sigma_u$ (MPa)</td>
<td>present</td>
<td>69.0±5.4</td>
<td>85.9±3.8</td>
<td>73.1±1.2*</td>
</tr>
<tr>
<td>$\sigma_u$ (MPa)</td>
<td>[literature]</td>
<td>82</td>
<td>84</td>
<td>73</td>
</tr>
<tr>
<td>$\epsilon_u$ (%)</td>
<td>present</td>
<td>7.00±1.50</td>
<td>6.50±1.00</td>
<td>6.14±0.53</td>
</tr>
<tr>
<td>$\epsilon_u$ (%)</td>
<td>[literature]</td>
<td>14</td>
<td>3.1</td>
<td>4.8</td>
</tr>
<tr>
<td>$G_{IC}$ (J/m\textsuperscript{2})</td>
<td>present</td>
<td>130±20</td>
<td>110</td>
<td>300</td>
</tr>
<tr>
<td>$T_g$ (°C)</td>
<td>[literature]</td>
<td>107</td>
<td>150</td>
<td>93</td>
</tr>
</tbody>
</table>

*Yield stress

### Table III. Fracture data and standard deviations from triaxial and uniaxial tests.

<table>
<thead>
<tr>
<th>Resin system</th>
<th>Uniaxial strength (MPa)</th>
<th>Poker-chip strength (MPa)</th>
<th>Uniaxial strain to failure (%)</th>
<th>Poker-chip strain to failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(i) DGEBA/DETA</td>
<td>69.0±5.4</td>
<td>29.1±4.5</td>
<td>7.00±1.5</td>
<td>0.85±0.1</td>
</tr>
<tr>
<td>(ii) DGEBA/MHPA</td>
<td>85.9±3.8</td>
<td>26.9±5.7</td>
<td>6.50±1.0</td>
<td>0.57±0.2</td>
</tr>
<tr>
<td>(iii) DGEBA/APTA</td>
<td>73.1±1.2*</td>
<td>32.0±2.2</td>
<td>6.14±0.5</td>
<td>0.79±0.1</td>
</tr>
<tr>
<td>(iv) TGDDM/DDS</td>
<td>59.9±12</td>
<td>26.6±7.7</td>
<td>1.77±0.4</td>
<td>0.55±0.2</td>
</tr>
</tbody>
</table>

*Yield stress
FIGURE CAPTIONS

Figure 1. Schematic of initiation mechanisms for transverse cracks a) Transverse crack initiation in the matrix. b) Transverse crack initiation at the fiber/matrix interface.

Figure 2. SEM-micrograph of carbon fiber/PEEK cross-section in cross-ply laminate. Load applied in the horizontal direction. Micrograph obtained by Prof R. Talreja, Georgia Inst of Technology, USA and coworkers.

Figure 3. Chemical structures of the components in the three epoxies based on DGEBA. The epoxies are DGEBA/DETA, DGEBA/MHPA, and DGEBA/APTA. In (iii), X+Y+Z=5.3.

Figure 4. Chemical structures of the components in TGDDM/DDS.

Figure 5. Schematic of the poker-chip test set-up and the coordinate system for the stress analysis. The θ-direction is perpendicular to the r- and z-directions.

Figure 6. Calculated normal stress, σ_z, and in-plane stresses, σ_r and σ_θ for DGEBA/DETA as a function of radial position r/a, according to equations (5) and (6), aspect ratio (diameter/thickness) of 7.5.

Figure 7. Uniaxial stress-strain curves for four epoxies.

Figure 8. Apparent nominal stress versus strain in the z-direction for four poker-chip epoxies subjected to a composite-like triaxial stress state.

Figure 9. Stress-strain curves for DGEBA/MHPA in uniaxial and triaxial loading.

Figure 10. Photographs of typical fracture surfaces for four poker-chip specimens of different epoxies.

Figure 11. SEM-micrographs of fracture surfaces for a) DGEBA/DETA and b) DGEBA/APTA.
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A biaxial thermo-mechanical disk test for glassy polymers

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Abstract
Failure criteria for polymers need to include effects from the stress state. For this reason, biaxial test results are of interest. However, biaxial test methods usually require expensive equipment. In the presented test method, a disk of epoxy is bonded between a steel ring and a steel disk. The temperature is then lowered until fracture is observed. Experiments were performed on three different glassy epoxy polymers. The biaxial stress state was analyzed by finite element analysis and by an approximate analytical model. Experimental observations support the ability of the method to provide material property data. An approximate analytical model was found sufficiently accurate for stress analysis and determination of the stress state at failure.

1. INTRODUCTION

The development of improved polymer composite materials requires understanding of the role of material constituents during failure processes. Loading of the material in the weak direction transverse to the fiber direction is commonly associated with fracture at very low stresses and strains. For this loading case, the polymer matrix is subjected to a triaxial stress state at the microlevel\textsuperscript{1-5}. In recent work\textsuperscript{5}, the highly triaxial nature of the stress state in transversely loaded glass fiber/epoxy was evaluated by finite element analysis. Experimental results from triaxial tests on epoxies demonstrated significantly lowered strain-to-failure under these conditions as compared with uniaxial results\textsuperscript{5}. A criterion for failure initiation under arbitrary stress state therefore needs to be developed. In this context, the need for multiaxial tests with different stress states is apparent. Any proposed criterion could then be compared with experimental data from a variety of loading cases.

* To whom correspondence should be addressed.
Several biaxial tensile tests have been proposed in literature\textsuperscript{3,6-8}. Mönch et al\textsuperscript{6} developed the biaxial tension cruciform test method which has been successfully applied to metals and composites. However, the cruciform test is difficult to apply to brittle materials. The corners of the cruciform specimen act as stress raisers and are likely to initiate fracture. Sultan and McGarry\textsuperscript{8} performed biaxial tensile tests on pressurized epoxy tubes. In their study a pressurized silicone oil inside the cylinder provides the hoop stress while a tensile test machine applies the axial stress. Both in the cruciform and the pressurized tube test complicated experimental set-ups are needed. A simpler test method was suggested by Nicholls\textsuperscript{3}. He applied biaxial tensile load to investigate the effect of biaxial stress on the strain to failure of neat resins. Nicholls clamped a short and wide specimen in a tensile tester, claiming a biaxial stress state to be active. However, the stress state is difficult to analyze as clamping conditions are critical in this type of test.

In the present study a method for testing glassy polymers under biaxial tensile loading is developed. Finite element analysis is performed in order to determine the stress state at the microlevel and to locate sites of stress concentrations. Also, an approximate analytical model is investigated in order to evaluate if the stress state can be estimated by a simplified analysis.

2. EXPERIMENTAL

2.1. Materials

Three epoxy systems were tested. In two of the epoxy systems, the epoxy component is DGEBA, diglycidyl ether of bisphenol (DER 332, Dow Chem Co). The DGEBA is cured by two different curing agent: (i) DETA, diethylenetriamine (DEH 20, Dow Chem Co) and (ii) APTA, polyoxy propyleneamine, (Jeffamine T-403, Texaco Chem Co). The third system consists of, (iii) tetraglycidyl 4,4' diaminodiphenyl methane epoxy, TGDDM (MY 720, Ciba Geigy) cured by 4,4' diaminodiphenyl sulphone, DDS, (HT 976, Ciba-Geigy). DGEBA and TGDDM are aromatic epoxies. DETA and APTA are aliphatic amines whereas DDS is an aromatic amine.
2.2. Casting procedure
The three different systems were carefully mixed by hand, vacuum was applied to the mixtures ten minutes before casting. The mixtures were then poured into a fluoropolymer coated aluminium mold. Material compositions and cure schedules are presented in Table I.

2.3. Specimen fabrication and test methods
The cast plates were removed from the mold and machined to the specimen dimensions required for mechanical testing. The specimens for the thermal constraint tests were cut by a water jet cutter. The specimen diameter was 60 mm with a thickness of 2 mm. The epoxy disks were bonded to steel adherents in a guided fixture to ensure alignment of the adherents. The disks were bonded to the adherents by one of the epoxy resins, DGEBA/DETA. The epoxy adhesive was cured at 110 °C under a load of 10 kg. Prior to bonding, the steel adherents and epoxy plates were ground and then degreased with acetone. The two steel adherents were of different design. The upper adherent was a steel ring with outer and inner radius of 30 mm and 15 mm, respectively. The lower adherent was a disk with a radius of 30 mm. Both adherents were 7 mm thick. The use of a steel ring facilitates crack observation. A schematic of the test geometry and coordinate system is presented in Figure 1.

Specimens designed for uniaxial testing were milled to the dimensions suggested by ASTM D638M-81, type I, in a computer controlled milling machine. The strains in uniaxial tests were measured by strain gauges, type EP-08-125AD-120, manufactured by Measurement Group Inc. Poisson's ratio and Young's modulus at ambient conditions were measured in an Instron test machine.

Temperature dependencies of Young's modulus for the epoxies were measured by DMTA tests, in a Dynamical Mechanical Thermal Analyser MKIII from Rheometric Scientific Ltd. Tests were performed on 30 mm long cantilever beam specimens with cross sectional areas of 2x2 mm². The DMTA specimens were cut in a diamond wheel cutter.

Temperature dependencies of the thermal expansion coefficients for the epoxies were measured for a free expanding plate down to -160°C. The strains at free expansion were measured by strain gauges, type CEA-13-062UT-350 and CEA-06-240UZ-120, manufactured by Measurement Group Inc.

The temperature during cooling was measured with a digital thermometer, AΣΛ by Automatic Systems Laboratories, using a thermocouple, Pt 100, with an accuracy of ±0.1°C measuring down to -200°C.
The dimensions of the thermocouple were 10×2 mm. During the test the thermocouple was placed on the free epoxy surface of the specimen.

The specimen was placed on a perforated cardboard cylinder in an insulated box at room temperature, see Figure 2. Liquid nitrogen was poured into an insulated teflon funnel which ended at the bottom of the box. The cooling rate from room temperature was approximately 2°C per minute. However, the cooling rate in the beginning was higher than at the end, cooling rate at failure was approximately 1°C per minute. The specimen was observed through a window at the top of the test chamber, see Figure 2. Totally 18 specimens were tested, 6 of each material.

3. STRESS ANALYSIS

The polymer disks are bonded to steel adherents and thermally loaded as the temperature is lowered. This temperature decrease will cause both the polymer disk and the steel adherents to contract. Due to the difference in thermal expansion coefficients, the polymer disk is constrained by the steel adherents so that a biaxial tensile stress state is generated in the polymer.

The coordinate system is defined in Figure 1. The center of the epoxy specimen is the origin of the coordinate system.

3.1. FEM-analysis

The finite element method (FEM) was used to determine the stress state in a thermally loaded, constrained epoxy disk. The commercially available ANSYS® system was used for this analysis. In the epoxy disk and its adherent steel substrates a three-dimensional stress state is present. However, models of axisymmetric 3-D structures such as the present one can be represented in equivalent 2-D form. The ANSYS® program provides elements for this type of analysis. In the present investigation, the eight-node axisymmetric harmonic element, PLANE83, was used. The PLANE83 element assumes linear elastic material. The mesh is shown in Figure 3. A total number of 5000 elements were included in the mesh. The epoxy disk is assumed to be perfectly bonded to the steel adherents. Nodes on the symmetri axis, r=0 mm, were restrained in the radial direction. Nodes on the lower edge of the steel disk, z=-8 mm, were restrained in the z-direction. To avoid problems due to stress averaging for dissimilar materials, adherent and epoxy stresses were evaluated within selected elements.
Calculations were performed for one of the epoxies, DGEBA/DETA. Material data used in numerical as well as in analytical analyses are presented in Table II.

3.2. Analytical approximation

The approximate analytical analysis is based on the assumption that the steel adherents are rigid in comparison to the epoxy disks. The steel adherents are therefore free to contract or expand according to the change in temperature. This will simplify the analysis. Only the difference in thermal expansion coefficient between the epoxy and the steel multiplied by the change in temperature are required for calculation of the strain in the epoxy disk.

The analytical analysis does not consider any edge effects. The stresses in the epoxy disk will therefore be independent on the z-coordinate. Thus, the biharmonic equation

$$\nabla^4 \Phi = 0$$

is solved in polar coordinates for Airy's stress function $\Phi$, where $\Phi$ is a function of radial position, $\Phi=\Phi(r)$, only. Airy's stress function $\Phi$ is expressed as

$$\Phi = A \log r + B r^2 \log r + Cr^2 + D$$

where $A$, $B$, $C$, and $D$ are constants. The constants $A$ and $B$ equals zero as the solution must be finite on the symmetraxis. The stress components can be written as

$$\sigma_r = \frac{1}{r} \frac{\partial \Phi}{\partial r} + \frac{1}{r^2} \frac{\partial^2 \Phi}{\partial \theta^2} = \frac{1}{r} \frac{\partial \Phi}{\partial r}$$

$$\sigma_\theta = \frac{\partial^2 \Phi}{\partial r^2}$$

$$\tau_{r\theta} = -\frac{\partial}{\partial r} \left( \frac{1}{r} \frac{\partial \Phi}{\partial \theta} \right) = 0$$

where the indices $r$, $\theta$, and $z$ indicate radial, tangential, and normal directions, respectively. According to Equations (2) and (3), the two in-plane stresses $\sigma_r$ and $\sigma_\theta$ are identical and the in-plane shear stress $\tau_{r\theta}$ is zero throughout the disk. The stresses are calculated for the plane stress situation, according to
\[
\begin{align*}
\sigma_r &= \frac{E}{1-v^2} (\varepsilon_r + v \varepsilon_\theta) \\
\sigma_\theta &= \frac{E}{1-v^2} (\varepsilon_\theta + v \varepsilon_r) \\
\sigma_z &= 0
\end{align*}
\]  

(4)

\[\varepsilon_r = \varepsilon_\theta = (\alpha_{\text{steel}} - \alpha_{\text{epoxy}}) \Delta T \]  

(5)

where \(\alpha_{\text{steel}}\) and \(\alpha_{\text{epoxy}}\) are the thermal expansion coefficients for steel and epoxy, respectively, and \(\Delta T\) is the temperature change.

### 3.3. Temperature dependence of polymer properties

The elastic properties and the thermal expansion coefficient of epoxies in their glassy state depend on temperature\(^{10-12}\). In the described analyses, the dependencies, \(E(T)\), \(\alpha_{\text{epoxy}}(T)\), and \(\nu(T)\), are of direct importance, see Equations (4) and (5). The considered temperature dependence of strains to failure originates from the thermal expansion coefficients only. This is apparent from Equation (5) and the temperature dependence can be taken into account by integration of Equation (5).

\[
\varepsilon_r = \varepsilon_\theta = \int_{r_i}^{r_2} [(\alpha_{\text{steel}} - \alpha_{\text{epoxy}}(T))] dT.
\]

(6)

The effect on ultimate stresses caused by the temperature dependence of elastic properties and thermal expansion coefficient can be taken into account. The loading situation is such that the difference in thermal expansion coefficient between steel and the glassy polymer leads to thermally induced strains in the glassy polymer. For an elastic material, the stress state at failure can therefore be obtained from the strains expressed in Equation (6) through knowledge of only the moduli at the failure temperature. Measurement of Poisson’s ratio at the temperature of failure requires an additional separate experiment. However, according to Equation (4) a small change in Poisson’s ratio has a minor effect on the stresses in the epoxy disk. Poisson’s ratio data from the glassy polymer at room temperature could therefore be used.

As both numerical and analytical analyses consider linear elastic materials, the temperature dependencies of elastic polymer properties and thermal expansion coefficient have the same influence on the results. Since
the present paper merely investigates the test method itself, effects from temperature dependence of polymer properties are excluded unless specifically stated. This is in order to facilitate comparison of parameters calculated by different theoretical methods.

4. RESULTS AND DISCUSSION

Biaxial testing of epoxies was carried out by cooling specimens of the geometrical arrangement presented in Figure 1. Fracture was observed through the window on top of the test chamber, see Figure 2. Experimental results for three epoxies are presented in Table III. The ultimate stress $\sigma_u$ is the true stress at failure, taking the temperature dependence of Young's modulus and thermal expansion coefficient into account. The stress state is biaxial with equal magnitude of radial and tangential stress. $\sigma_u$ and $\varepsilon_u$ are in the ranges $54 - 73$ MPa and $0.6 - 1\%$ respectively for the three epoxies. These data seem physically reasonable since they are inbetween the values for uniaxial and triaxial loading of the same epoxies\(^5\). Measured temperature change, $\Delta T$, was between -219 and -269 °C. The scatter in $\sigma_u$ was low compared to the previous poker chip test results for the same materials subjected to a triaxial stress state\(^5\). All three epoxy systems were successfully tested by the thermo-mechanical disk method, 16 out of totally 18 specimens failed during cooling.

The fracture of a specimen was observed through the window on top of the test chamber, see Figure 2. In all cases, the cracks were observed to grow at high speed through the entire thickness, and in the central regions of the specimen. The cracks were always perpendicular to the disk plane. At the time of failure, a loud popping sound was heard. No initiation areas were detected in situ, neither could the position of the initiation region be determined conclusively. Typical features of fractured specimens are presented in Figure 4. All specimens showed highly branched cracks, either in the center or close to the inner edge of the steel ring, $r=15$ mm, see Figure 4. The branching points are interpreted as points where high speed cracks transform into two or more cracks with lower speed\(^\text{13}\). The stress analysis will be used to discuss the location of crack initiation.
4.1. FEM-analysis
A numerical stress analysis by FEM was performed for one of the epoxies, DGEBA/DETA, for the experimentally investigated temperature change ($\Delta T = -269 \degree C$). The element solutions show homogenous stresses in the central part of the epoxy disk, $r < 27 \text{ mm}$, for all stress components. This is clarified in Figures 5, 6 and 7, where the stresses at the lower interface ($z = -1 \text{ mm}$), the mid-plane ($z = 0 \text{ mm}$), and upper interface/free surface ($z = 1 \text{ mm}$) in the epoxy, respectively, are plotted against radial position. The coordinate system was defined above, see Figure 1. The stresses are shown to be almost constant through the thickness of the specimen. A small difference between stresses at the upper and lower interfaces is expected as the use of a steel ring brings in an edge at $r = 15 \text{ mm}$. In all three plots the radial and tangential stresses are almost identical. Also the normal and shear stresses are close to zero or zero, as expected from theoretical considerations already discussed. However, close to the edge the stress state is disturbed, and increases in tangential stress, normal, and shear stresses are observed at the steel/epoxy interface edge, see Figures 5 and 7. In the middle ($z = 0 \text{ mm}$) of the epoxy all the stresses are decreasing at the edge, see Figure 6. According to calculations, the shear stress is zero and the average radial and tangential stresses are 45.4 MPa at failure. The average normal and shear stresses are close to zero.

The results of these calculations were compared to results of a less refined mesh. The average stresses in the epoxy disk were not changed significantly (0.3 %). However, stresses at the edge show larger discrepancy as the mesh is changed. The more refined mesh gives higher stress concentrations at the interface/edge. The stress concentrations are difficult to determine due to the high stress gradients.

4.2. Approximate analytical stress analysis
For the purpose of comparing different theoretical methods, calculations of in-plane ultimate stresses were made disregarding the temperature dependence of Young's modulus and thermal expansion coefficient for the polymer. The in-plane ultimate stresses, $\sigma_{uu}$, for DGEBA/DETA are then 44.5 MPa, which differ from the FEM results by 2 % only. Therefore, the approximate analytical model is sufficient for estimates of the stress state in the test specimen provided stress concentrations at the polymer/metal interface edge do not cause crack initiation.

The analytically calculated true ultimate stresses for the three epoxies are presented in Table III. The true ultimate stresses were calculated taking the effect of temperature dependencies of Young's modulus and thermal
expansion coefficient into account. This effect was significant. For DGEBA/DETA, the ultimate stress increased by as much as 20 percent to a true ultimate stress of 54.1 MPa.

4.3. Crack initiation characterization

Premature crack initiation would lead to determination of lower ultimate stresses than the true material properties. As the in situ and post mortem studies did not give conclusive information about initiation sites we need to establish a plausible region of crack initiation.

According to the numerical analysis, stress concentrations are located at the epoxy/steel interface edge, \( r=30 \) mm. If crack initiation had taken place here, debonding at the polymer/steel interface would most likely have occurred due to the high shear and normal stresses. However, this was not the case for any of the 16 specimens. The ability of macroscopically brittle epoxies to undergo localized plastic yielding is well known\(^\text{14}\). For this reason, stress concentrations calculated assuming linear elastic material behavior do not necessarily lead to failure. This specimen design inflicts stress raise at the interfaces along the specimen circumference only, see Figures 5 and 7. The stresses a small distance away from the edge, \( 27 \text{ mm}< r< 30 \) mm, are lower than the average stresses and are therefore less likely to initiate cracking, or to provide the necessary crack driving force for a small defect.

Another possible cause of premature failure is radial crack initiation at the sample edge due to the tangential stress. A way to reduce the tangential stress is to use a spew fillet. Numerical stress analysis of a specimen with a spew fillet was therefore performed, see Figure 8. The stresses at the upper interface (\( z=1 \) mm) are plotted in Figure 9. The stress concentrations at the interface edge are significantly reduced, cf. Figure 7. This is in agreement with what was observed for butt joints by Adams et al.\(^\text{15}\). They showed a reduction in the normal stress, \( \sigma_z \), by almost 40 % by introduction of a spew fillet. Our results show a decrease in the tangential stress, \( \sigma_\theta \), of 43%. This is important as the tangential stress could initiate radial cracks at the edge. Two DGEBA/DETA specimens were therefore prepared with a spew fillet and tested. The results of these tests are encouraging. The average temperature change was measured to \( \Delta T=-258 ^\circ \text{C} \) and the crack appearance was similar to that in specimens with no spew fillet. This supports the hypothesis that the stress concentrations at the interface edge do not affect the measured ultimate stresses, and that true intrinsic material properties are measured by the test method. Crack initiation most likely occurred in the central regions of the
specimens, $r<27$ mm. The stresses at failure are then well approximated by the simplified analytical model.

5. CONCLUSIONS

A test method has been developed for biaxial tensile testing of glassy polymers. A disk of epoxy is bonded between a steel ring and a steel disk. As the temperature is lowered a state of biaxial plane stress is induced until fracture is observed. Experimental observations and finite element analysis suggest fracture to initiate away from the circumference of the specimen. Results support the ability of the method to provide material property data. An approximate analytical model was found sufficiently accurate for stress analysis and determination of the stress state at failure.

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REFERENCES


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Figure 1. Schematic of the sample design. Dimensions; disk diameter 30 mm, epoxy disk thickness 2 mm, steel ring inner diameter 15 mm, and thickness of steel adherents 7 mm.

Figure 2. Schematic of the insulated test chamber used for cooling of the ring specimen.

Figure 3. The element mesh used in the numerical analysis.

Figure 4. Photographs showing typical features of cracked specimens. Photographs a) and b) DGEBA/DETA specimens and c) DGEBA/APTA specimen.

Figure 5. Stresses as a function of radial position at the lower polymer/steel interface.

Figure 6. Stresses as a function of radial position at the specimen mid-plane.

Figure 7. Stresses as a function of radial position at the upper polymer/steel interface and free surface.

Figure 8. The element mesh for specimen with a spew fillet.

Figure 9. Stresses as a function of radial position at the upper polymer/steel interface and free surface with a spew fillet.
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Paper III
A criterion for crack initiation in glassy polymers subjected to a composite-like stress state

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Abstract
Three epoxy systems of interest as composite matrix materials are examined for their yielding and failure behavior under uniaxial, biaxial and triaxial stress states. Yield criteria applicable to glassy polymers, i.e., accounting for the hydrostatic stress effect on the deviatoric stress to yielding, are assessed. It is found that under stress states resembling those in matrix constrained between fibers, e.g., equibiaxial and equitriaxial tension, yielding is suppressed while brittle failure, presumably caused by crack growth from cavitation, occurs. A criterion for this mode of failure is proposed as the critical dilatational strain energy density. Experimental data are found to support this criterion.

1. INTRODUCTION

The development of polymers for use as matrix materials in composites has generally been driven by the desire to achieve high yield stress and toughness as well as good adhesion with fibers. Although these properties are desirable and generally lead to improved composite performance, it is important to realize that the local stress state in matrix within a composite can induce behavior which may not be reflected in these properties. An example of this was provided by Sternstein and Ongchin\textsuperscript{1} who showed that in glassy thermoplastics two modes of plastic deformation, shear yielding and crazing,

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can coexist and transition from one to the other is determined by the stress state. Interestingly, the hydrostatic stress component affects both yielding modes, albeit in different ways. In commercially produced composites with high fiber volume fractions it is inevitable that fibers are distributed unevenly and that clusters of fibers as well as resin-rich areas exist. In such conditions the local stress states will vary between nearly shear dominated to nearly hydrostatic. The same given matrix will thus be predisposed to different deformation and failure modes in different regions of the composite.

In most studies of glassy polymers, the shear driven yielding has been emphasised with attention being given to the associated influence of hydrostatic stress. A number of yield criteria for this purpose has been proposed\textsuperscript{2-5}. These criteria usually predict the effect of hydrostatic compression on the yield stress satisfactorily but do less well when hydrostatic tension is applied. Also, the consideration of the yield stress reduction alone cannot explain the observed fact that although the strain to failure in uniaxial tension for matrix materials ranges from 1.5\% to 70\%, the strain to failure in transverse tension of unidirectional fiber composites typically varies between 0.2\% and 0.9\%\textsuperscript{6-9}. A part of the explanation could lie in fiber/matrix debonding which may occur at low strains. However, as demonstrated in a previous study\textsuperscript{10}, the triaxial stress state can reduce the strain to failure of epoxies to within the observed failure strains in transverse tension of composites. The present study will therefore focus on yield and failure criteria for epoxies subjected to different stress states.

In the following a wide range of test data are reported under stress states of uniaxial tension, uniaxial compression, biaxial compression, biaxial tension with various combinations of normal stresses, and equitriaxial tension (hydrostatic tension). Three epoxy systems have been tested. Yield criteria modified to account for the effect of hydrostatic stress are examined for their validity against the test data. It is shown that the yield criteria do not predict the behavior in stress states approaching the purely hydrostatic tension. For these conditions the critical dilatation strain energy density is proposed as the criterion for failure. The failure mechanism associated with this criterion is proposed as cavitation followed by cracking.
2. EXPERIMENTAL PROCEDURE

2.1. Materials
Three epoxy systems were tested. In two of the epoxy systems, the epoxy component is DGEBA, diglycidyl ether of bisphenol A (DER 332, Dow Chem Co). The DGEBA is cured by two different curing agents: (i) DETA, diethylenetriamine (DEH 20, Dow Chem Co) and (ii) APTA, polyoxy propyleneamine, (Jeffamine T-403, Texaco Chem Co). The third system consists of, (iii) tetraglycidyl 4,4' diaminodiphenyl methane epoxy, TGDDM (MY 720, Ciba Geigy) cured by 4,4' diaminodiphenyl sulphone, DDS, (HT 976, Ciba-Geigy). DGEBA and TGDDM are aromatic epoxies. DETA and APTA are aliphatic amines whereas DDS is an aromatic amine.

2.2. Casting procedure
The three different systems were carefully mixed by hand, vacuum was applied to the mixtures ten minutes before casting. The mixtures were then poured into an aluminium mold coated by a fluoropolymer. Material compositions and cure schedules are presented in Table I.

2.3. Specimen fabrication and test methods
The cast plates were removed from the mold and machined to the specimen dimensions required for mechanical testing. The specimens designed for uniaxial testing were milled to the dimensions suggested by ASTM D638M-81, type I, in a computer controlled milling machine. The strains in uniaxial tests were measured by strain gauges, type EP-08-125AD-120, manufactured by Measurement Group Inc.

The specimens for compression tests were cut by a water jet cutter. The uniaxial compression specimens were cut into a rectangular shape, 4.9 x 30 mm for the two DGEBA-based resins and 4.9 x 10 mm for the TGDDM-based resin. The reduced size of the TGDDM/DDS specimen is due to the high yield stress of the material. Although square cross-sections are not ideal, the accuracy was considered sufficient for the purpose of the present investigation. The specimen designed for plane strain compression tests were square shaped, 30 x 30 mm. All compression specimens were 1.5-1.7 mm thick. Strains were measured by a COD extensometer mounted between the dies of the test equipment. The compression specimens were treated by a molybdenum-based lubricant in order to reduce the influence of friction11.
The dimensions of the steel dies of the test rig were, width=5 mm and length=50 mm.

Biaxial tension specimens were made by laminating 1.25 mm thick epoxy plates between glass fiber/epoxy (GF/EP) prepreg, Hy-E 9082F from ICI Fiberite, with fiber volume fractions ranging from 55 to 60%. The laminates were stacked in the following sequence, \([90_2, 0^\circ, \text{epoxy}]_s\), see Figure 1. The laminates were cured for 2 hours at 125°C at an external pressure of 0.7 MPa. GF/EP tabs were bonded to the cured laminate and 220 mm long and 20 mm wide specimens were cut from the plate by a water jet cutter. The specimens were prepared with a gauge length of 110 mm. Strains were measured by a 50 mm gauge length extensometer. Failure of the epoxy plates is defined to occur as the first crack or group of cracks appear in the epoxy. The thermal expansion coefficients of the three epoxies were measured in order to determine residual strains in the epoxy core of the biaxial tension test specimens.

All tension and compression tests were performed in an Instron test machine. Strain rates for uniaxial tests were 1% per minute. For the biaxial tension test and the compression tests, the strain rates are not constant. The stroke rates used for the biaxial tension test and the compression tests were 0.01 mm/s and 0.001 mm/s respectively. All tests were performed at ambient conditions. For the uniaxial tensile tests, yield stress was determined from the true-stress strain relation by the Considère construction\(^{12}\). For both types of compression tests, the yield stress was taken as the maximum stress value\(^4\) (the upper yield stress).

The specimens for the thermo-mechanical disk tests were cut by a water jet cutter. The specimen diameter was 60 mm with a thickness of 2 mm. The epoxy disks were bonded to steel adherents in a guided fixture to ensure alignment of the adherents. The disks were bonded to the adherents by one of the epoxy resins, DGEBA/DETA. The epoxy adhesive was cured at 110 °C under a load of 10 kg. Prior to bonding, the steel adherents and epoxy plates were ground and then degreased with acetone. The two steel adherents were of different design. The upper adherent was a steel ring with outer and inner radius of 30 mm and 15 mm, respectively. The lower adherent was a disk with a radius of 30 mm. Both adherents were 7 mm thick. The use of a steel ring facilitates crack observation. A schematic of the test geometry and coordinate system is presented in Figure 2. The center of the epoxy specimen is the origin of the coordinate system.
Temperature dependencies of the thermal expansion coefficients for the epoxies were measured for a free expanding plate down to -160°C. The strains at free expansion were measured by strain gauges, type CEA-13-062UT-350 and CEA-06-240UZ-120, manufactured by Measurement Group Inc.

The temperature in the cooling tests was measured by a digital thermometer, ΑΣΛ by Automatic Systems Laboratories, using a thermoelement, Pt 100, with an accuracy of ±0.1°C measuring down to -200°C. The dimensions of the thermoelement were 10x2 mm. The thermoelement was placed on the free epoxy surface of the specimen during the test.

The specimens were placed on a perforated cardboard cylinder in an insulated box at room temperature, see Figure 3. Liquid nitrogen was poured into an insulated teflon funnel which ended at the bottom of the box. The average cooling rate from room temperature was approximately 2°C per minute. However, the cooling rate in the beginning was higher than at the end, the cooling rate at failure was approximately 1°C per minute. The specimen was observed through a window at the top of the test chamber, see Figure 3.

Temperature dependencies of Young’s modulus for the epoxies were measured by DMTA tests, in a dynamical mechanical thermal analyser MKIII from Rheometric Scientific Ltd. Tests were performed on 30 mm long cantilever beam specimens with cross sectional areas of 2x2 mm². The DMTA specimens were cut in a diamond wheel cutter.

3. YIELDING AND FAILURE ANALYSIS

3.1. Yield criteria for polymers

It is known that in polymers the yield behavior is sensitive to hydrostatic pressure\(^5\). As a consequence, the yield stress in tension becomes different from that in compression. The classical yield criteria applicable to metals must therefore be modified for polymers. In the following the most common modifications to the von Mises and Tresca criteria are described since these will be used later to evaluate the test data.

3.1.1. Modified von Mises yield criteria

The von Mises yield criterion assumes the yielding material to be isotropic. It states that the yield function depends on the second invariant of the stress deviator, \(J_2\).
\[ J_2 = \frac{1}{2} S_{ij} S_{ij} = \text{const} = K^2 \]  

(1)

where $S_{ij}$ is the stress deviation tensor and $K$ is a constant. It can be shown that this is equivalent to

\[ (\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 = 6K^2 \]  

(2)

This is a more familiar form of the yield criterion, where $\sigma_1$, $\sigma_2$, and $\sigma_3$ are principal stresses. The von Mises yield criterion does not predict differences in yield stress between compression and tension. Modifications of the von Mises criterion have incorporated the effect of hydrostatic stress into Equation (2). The modified von Mises criterion can in general form be written as

\[ A(\sigma_1 + \sigma_2 + \sigma_3) + B\left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2\right] = 1 \]  

(3)

It is possible to define the constants $A$ and $B$ in terms of the simple uniaxial compressive and tensile yield stresses, $\sigma_{yc}$ and $\sigma_{yt}$ respectively. This will give the yield criterion suggested by Raghava et al.

\[ 2(\sigma_{yc} - \sigma_{yt})(\sigma_1 + \sigma_2 + \sigma_3) + \left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2\right] = 2\sigma_{yc} \sigma_{yt} \]  

(4)

Bauwens derived a yield criterion valid for an arbitrary state of stress based on the Eyring theory of non-Newtonian flow. They expressed the energy criterion as

\[ \tau + A(\sigma_1 + \sigma_2 + \sigma_3) = C \]  

(5)

where $\tau$ is the octahedral shearing stress, and $A$ and $C$ are constants. Bauwens determined the complete criterion at constant strain-rate and temperature to be

\[ \frac{\sqrt{2}(\sigma_{yc} - \sigma_{yt})}{(\sigma_{yc} + \sigma_{yt})}(\sigma_1 + \sigma_2 + \sigma_3) \]

\[ + \left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2\right] = \frac{2\sqrt{2}\sigma_{yc} \sigma_{yt}}{(\sigma_{yc} + \sigma_{yt})} \]  

(6)
Note that if $\sigma_{yc} = \sigma_{yl}$, both the modified von Mises criteria by Raghava and Bauwens, Equations (4) and (6), reduce to the von Mises criterion, Equation (2).

The strain energy in a strained material can be expressed as the sum of two terms, dilatational and distortional energies. The distortional energy density is expressed as

$$U_d = \frac{1}{4G} S_{ij} S_{ij}$$

where $G$ is the shear modulus. Comparison of the expressions in Equations (1) and (7) reveals that the distortional energy density is the physical basis of the von Mises criterion.

### 3.1.2. Modified Tresca yield criterion

Tresca proposed yielding to occur when a critical value of the maximum shear stress $\sigma_s$ is reached. If $\sigma_1 > \sigma_2 > \sigma_3$ the criterion is

$$\frac{1}{2}(\sigma_1 - \sigma_3) = \sigma_s$$

The Tresca yield criterion may also be modified to take the dependence of hydrostatic pressure into account. The simplest way is to make the critical shear stress a function of the hydrostatic pressure, so that $\sigma_s$ in Equation (8) is expressed as;

$$\sigma_s = \sigma_s^* - \mu (\sigma_1 + \sigma_2 + \sigma_3)$$

where $\sigma_s^*$ is the shear yield stress in the absence of any overall hydrostatic pressure and $\mu$ is a material constant. The hydrostatic pressure is taken to be positive for uniaxial tension loading and negative for uniaxial compression loading. The yield stress, $\sigma_s$, is therefore given by Equations (8) and (9). The material constants, $\sigma_s^*$ and $\mu$, are determined in uniaxial tension and compression.

$$\sigma_s^* = \frac{\sigma_{yc} \sigma_{yl}}{(\sigma_{yc} + \sigma_{yl})} \quad \text{and} \quad \mu = \frac{1}{2} \frac{(\sigma_{yc} - \sigma_{yl})}{(\sigma_{yc} + \sigma_{yl})}$$
If $\sigma_{yt}$ and $\sigma_{yc}$ are determined experimentally, the envelopes of the modified von Mises criterion by Raghava and the modified Tresca criterion may be drawn. The Raghava criterion is a distorted ellipse inscribed by the modified Tresca hexagon.

### 3.2. Dilatational energy density criterion

In thermoplastics, certain stress-field conditions may cause crazing. A craze is a narrow zone of highly deformed and voided polymer. The successful criterion for craze formation proposed by Sternstein and Ongchin\(^1\) can be interpreted as a critical volume increase related to a critical polymer chain mobility induced by a dilatational stress. Pressure related volume change effects have also been demonstrated by Sultan and McGarry\(^{13}\) who showed that microcavitation occurred in glassy epoxies around particles. Associated with this phenomenon was the finding that a larger effect of hydrostatic stress on yield stress existed when cavitation occurred than without it. It needs to be pointed out that for densely cross-linked glassy polymers such as epoxies, crazing does not occur\(^{14}\).

In stress states where the deviatoric component is small and much below that required for shear yielding, it would be expected that volume change related effects would dominate. Under the extreme condition of zero deviatoric stress, increasing the hydrostatic tension could lead to the critical condition of microcavitation within an elastic field. Such cavitation would be inherently unstable since the elastic field would offer little resistance to its growth. If a slight bias exists in the hydrostatic stress state, it would also be very likely that a crack initiates normal to the largest principal tensile stress. In such a case the initiated crack would grow unstably in the surrounding elastic, and thereby brittle, material. Figure 4 illustrates the proposed cavitation and cracking mechanisms. Fractographic studies of poker chip specimens of DGEBA/DETA support the cavity-induced brittle failure mechanism for triaxial tensile loading. A micrograph of an initiation point in a DGEBA/DETA poker chip specimen is presented in Figure 5. The micrograph shows a point of initiation from which cracks extend in multiple directions creating a star-like appearance. According to Figure 5, the diameter of any initiating cavity must have been smaller than 5 $\mu$m. The major crack propagated perpendicular to the direction in which the test machine grips were moving. Cracks extending in other directions were arrested.

Under the condition that the distortional energy density at a point is small, i.e., much below that required to cause yielding, we propose that
microcavitation will occur when the dilatational energy density at that point reaches a critical value. Assuming the material to be linearly elastic, the criterion can be written as

$$U_v = \frac{1 - 2v}{6E} (\sigma_1 + \sigma_2 + \sigma_3)^2 = U_v^{crit}$$  \hspace{1cm} (11)

where $\sigma_1$, $\sigma_2$, and $\sigma_3$ are the principal stresses, $v$ and $E$ are the Poisson's ratio and Young's modulus, respectively, and $U_v^{crit}$ is the critical dilatational energy density required for cavitation.

The proposed criterion can also be expressed in terms of the first invariant of the stress tensor (also known as the mean stress). However, in that case the critical value of the mean stress will become dependent on temperature, as the elastic constants appearing in Equation (11) are generally temperature dependent. It is possible that the critical value of the dilatational energy density required for microcavitation is a material constant independent of temperature. This contention appears to be supported by test data obtained at two different temperatures, which will be discussed in Section 4.

### 3.3. Analysis of test data

In order to examine the validity of several failure criteria for glassy polymers a number of tests were performed. The data reduction procedures for these test methods are presented below.

#### 3.3.1. Plane strain compression test

In the plane strain compression test, an epoxy plate is compressed between two metal dies, see Figure 6. The material under the dies is prevented from expanding in the direction perpendicular to $\sigma_1$ and $\sigma_2$, (3-direction in Figure 6), so that the strain rate during yield is zero in this direction. This creates a two dimensional stress state within the specimen, according to the Lévy-Mises\(^\text{15}\) equation for plastic flow:

$$\sigma_3 = \frac{1}{2} (\sigma_1 + \sigma_2)$$  \hspace{1cm} (12)

where $\sigma_2$ is zero as the influence of friction can be neglected. Therefore $\sigma_3 = 0.5\sigma_1$. Equation (12) is true only for a material that yields according to the von Mises yield criterion, but is used here to estimate the stress state in the compressed polymers.
3.3.2. **Uniaxial compression test**

In the uniaxial compression test, the restraining material of the plane strain compression test is removed. The compressed material is therefore free to expand in the plane, provided friction forces can be neglected. A uniaxial stress state is induced.

3.3.3. **Biaxial tension test**

A test was designed for subjecting the resins to biaxial tension. The GF/EP \([90^\circ, 0^\circ]\) part of the laminate restricts contraction of the epoxy sheet. The stiffer GF/EP laminates also suppresses brittle failure of the epoxy due to growth of surface defects. The latter facilitates plastic flow even for brittle epoxies. A similar test was proposed by Rezaifard et al.\(^{16}\) in which they bond a neat resin inbetween two 0°-layers of glass fiber reinforced plastic (GRP) laminates. However, the use of cross-ply laminates instead of 0°-laminates enhances the biaxial stress state in the epoxy core.

Classical laminate theory, CLT, was used to approximate the stress situation in the epoxy under loading. Residual strains in the epoxy due to curing of laminates were calculated and taken into account. The uniaxial stress-strain behavior in tension of the neat epoxies was used as input data for the epoxy lamellae. Figure 7 shows a typical nominal stress-strain curve for a biaxial tension specimen (DGEBA/APTA). There is a "knee" in the nominal stress-strain curve. This "knee" is due to multiple cracking of the transverse layers of the GF/EP. A variational approach\(^{17}\) was used to calculate reductions in stiffness and Poisson's ratio due to multiple cracking of the transverse layer of the GF/EP. The solutions were divided into two parts. Firstly, the situation where no cracks existed in the GF/EP, at strains less than about 1%. Secondly, the situation prior to failure of the epoxy layer, i.e. after transverse cracking of the GF/EP. The in-plane stresses in the epoxy were calculated for the corresponding strains using CLT.

3.3.4. **Thermo-mechanical test**

It was shown in a previous study that the stress state in the epoxy disk can be estimated by an approximate analytical method\(^{18}\). The analysis is based on the assumption that the steel adherents are rigid in comparison to the epoxy disk. Therefore, the strain in the epoxy equals the difference in free expansion between the steel and epoxy disks.
The analytical analysis does not consider any edge effects. The stresses will therefore be independent of the z-coordinate, see Figure 2. Thus, the biharmonic equation

$$\nabla^4 \Phi = 0$$  \hspace{1cm} (13)

is solved in polar coordinates for Airy's stress function $\Phi$, where $\Phi$ is a function of radial position, $\Phi = \Phi(r)$, only. As a consequence, the two in-plane stresses $\sigma_r$ and $\sigma_\theta$ are identical and the in-plane shear stress $\tau_{r\theta}$ is zero throughout the disk. The stresses are calculated for the plane stress situation, according to

$$\sigma_r = \frac{E}{1 - v^2} (\varepsilon_r + v \varepsilon_\theta)$$  
$$\sigma_\theta = \frac{E}{1 - v^2} (\varepsilon_\theta + v \varepsilon_r)$$  \hspace{1cm} (14)  
$$\sigma_z = 0$$

where the indices $r$, $\theta$, and $z$ indicate radial, tangential, and normal directions, respectively.

The mechanical properties of epoxies in their glassy state are known to depend on temperature\textsuperscript{19-21}. In the analysis described above these dependencies, $E(T)$, $\alpha_{\text{epoxy}}(T)$, and $v(T)$, are of great importance, see Equation (14). The influence of the moduli change is easily taken into account, as the moduli at the failure temperature corresponds to the ultimate stresses. Knowing the temperature dependence of $\alpha_{\text{epoxy}}$, the equibiaxial strains at failure are determined as

$$\varepsilon_r = \varepsilon_\theta = \int_{T_i}^{T_f} [\alpha_{\text{steel}} - \alpha_{\text{epoxy}}(T)] dT.$$  \hspace{1cm} (15)

where $\alpha_{\text{steel}}$ and $\alpha_{\text{epoxy}}(T)$ are the thermal expansion coefficients for steel and epoxy, respectively, and $T$ is the temperature. Poisson's ratio is assumed independent of temperature.
4. RESULTS AND DISCUSSION

4.1. Failure predictions by yield criteria

4.1.1. Uniaxial test results
Results from the uniaxial tests are presented in Table II. The uniaxial tensile test results were obtained in a previous study\(^{10}\). Figure 8 shows typical curves for materials tested in uniaxial compression. Uniaxial compressive yield stress was determined for all three epoxies. TGDDM/DDS has a very high yield stress due to aromatic groups in the molecular structure and a densely cross-linked network. The initial inflexion in the stress-strain curves are always present due to extrusion of excess lubricant and the general consolidation of the press and the test piece\(^{11}\).

For two of the materials, DGEBA/DETA and TGDDM/DDS, it was not possible to determine the yield stress in uniaxial tension, due to premature brittle failure. Morgan\(^{22}\) determined a yield stress of 83 MPa for DGEBA/DETA. The ratio of compressional to tensional yield stresses \((\sigma_{yc}/\sigma_{yt})\) is 1.4 and 1.2 for DGEBA/DETA and DGEBA/APTA respectively, see Table II. The relationships between compressive and tensile yield stresses are in the expected range for epoxies\(^{13,14}\). The results for DGEBA/APTA are in agreement with those presented by Lubin\(^{23}\). This in combination with the reasonable \(\sigma_{yc}/\sigma_{yt}\) ratios observed for DGEBA/DETA and DGEBA/APTA, are in support of the assumption of negligible friction in the compression test.

4.1.2. Biaxial test results
Figure 9 shows the stress-strain curves for the plane strain compression tests of DGEBA/DETA and DGEBA/APTA, for which yield stresses were found. No yield could be detected for TGDDM/DDS before fracture. The yield stresses are presented along with uniaxial test data in Table II. The yield stresses of the plane strain compression specimen are close to those measured in uniaxial compression. The ratio between the yield stresses in plane strain compression and uniaxial compression is 1.08 and 1.05 for DGEBA/DETA and DGEBA/APTA, respectively. This is in agreement with results for PS (1.04) by Bowden et al\(^4\) but less than their results for PMMA (1.28).

The results from the biaxial tension tests are presented in Table III. These results are calculated based on data for Poisson's ratios and thermal expansion coefficients presented in Table IV. The results in Table III are stresses at failure of the epoxy layers. The results for TGDDM/DDS are not of
interest for the evaluation of yield criteria as it did not yield in uniaxial tension.

4.1.3. Yield predictions

Experimental data and theoretical predictions of yielding are presented in Figures 10 (DGEBA/DETA) and 11 (DGEBA/APTA). Raghava’s\(^2\) and Bauwens’s\(^3\) yield criteria are almost identical for materials with \(\sigma_{y0}/\sigma_{yt} = 1.4\) or less. Bauwens’s yield criterion is therefore excluded in Figures 10 and 11.

The biaxial tension data, in the first quadrant of Figure 11 agree with the von Mises and Raghava criteria. This indicates that the DGEBA/APTA specimen yielded prior to brittle fracture. This does not appear to be the case for DGEBA/DETA. Observations of the specimens after failure showed a number of cracks to appear simultaneously in all materials.

Comparison with experimental data show the modified Tresca criterion to predict yield on the compression side better than the modified von Mises criteria. The modified von Mises criteria show better agreement on the tension side. It can therefore be concluded that the examined yield criteria are found to predict yielding of glassy epoxies subjected to uniaxial and biaxial stress states.

Since the examined yield criteria are three-dimensional, it is possible to predict yielding for the composite-like stress state of the poker-chip specimen\(^{10}\). The actual stress states (including residual stresses) prior to fracture of the poker-chip specimen are presented in Table V. The thermal stresses are calculated under the assumptions that the aluminium substrates are infinitely stiff compared to the epoxy disks and that no creep is taking place in the adhesive nor in the epoxy disks. The calculated effective and yield stresses of the poker chip specimens are presented in Table VI. It is obvious from these results that the effective stress is always lower than the predicted yield stress limit, visualized in Figures 10 and 11. Therefore, according to the criteria, yielding has not occurred in the poker-chip specimens before or during failure. Poker-chip strain to failure was previously found to agree with typical transverse data for GF/EP\(^{10}\). According to the criteria, initiation of failure in a transversely loaded GF/EP composite will not take place by matrix yielding in regions with equitriaxial stress states.
4.2. Failure predictions by dilatational energy density criterion
The dilatational energy density criterion is applicable only when the level of distortional energy density is low. For this reason, only methods that subject the glassy epoxies to a multiaxial tensile stress state are considered. These are the thermo-mechanical disk test\textsuperscript{18}, the biaxial tensile test presented above, and the poker-chip test\textsuperscript{10}.

The thermo-mechanical disk test method was thoroughly investigated in a previous study\textsuperscript{18}. A failure behavior analysis is presented in that study and therefore will not be repeated here. The ultimate in-plane stresses, $\sigma_{uu}$, and strains, $\varepsilon_{uu}$, are calculated according to Equations (14) and (15). For this analysis the temperature dependencies of the mechanical properties are required. The Young's modulus is known to increase with a decrease in temperature\textsuperscript{19,20}. The moduli of tested epoxies were found to increase by 55 to 85 percent for a temperature interval between room temperature and -160 °C, see Table VII. The strains at failure for the thermo-mechanical tests were calculated from the free expansions of the epoxies at the corresponding temperatures. The results from the thermo-mechanical disk tests are presented in Table VIII. The in-plane stresses are found to be in the interval 54-73 MPa for the three epoxies. Notice that the stress state is equibiaxial, meaning that radial and tangential stress components are equal.

The calculated dilatational energy density at failure for the different test methods, according to Equation (11), are presented in Table IX. Table IX also includes the distortional energy density, for comparison. As seen in Table IX the critical value of the dilatational energy density is roughly the same for each material in poker-chip and thermally loaded disk tests, while it is slightly higher in the biaxial tension test. This is probably because the stress state in the biaxial tension test has unequal principal stresses leading to a significant deviatoric stress component and a corresponding increase in distortional energy density, see Table IX. Furthermore, the high critical dilatational energy density of DGEBA/APTA is explained by that it is likely to have failed by yielding in the biaxial tension test, see Figure 11. The low dilatational energy for the uniaxial tensile test of TGDDM/DDS is due to crack initiation at a surface flaw. Therefore, comparison between critical dilatational energy densities for different tests is only valid for specimens that fail by the same mechanism, i.e. cavity formation.

Sternstein and Ongchin\textsuperscript{1} found the craze formation in PMMA to be dependent on temperature. Similarly, for the present data a temperature dependence is found for the dilatational stress component ($\sigma_1+\sigma_2+\sigma_3$) and hence the average stress. This was observed as data from poker-chip and
biaxial tension tests at ambient temperature were compared with those from thermo-mechanical disk tests at temperatures between -110 and -160 °C. However for dilatational energy density, no temperature dependence was observed for our results. Therefore, we suggest that the use of dilatational energy density is preferred to that of dilatational or average stress. Nevertheless, for failure prediction of a transversely loaded composite at ambient temperature, a critical average stress of the resin measured at the same temperature will apply. No correlation is found between the distortional energies at different load cases for the three epoxies, see Table IX. This is expected as yielding was not the mechanism of failure.

The results imply that brittle failure in glassy epoxies subjected to multiaxial tensile loads can be predicted by the suggested dilatational energy density criterion.

5. CONCLUSIONS

Examination of test results on three epoxy systems under different stress states reveals that their yield stress can be described satisfactorily by yield criteria only when the stress state results in sufficiently high energy density of distortion. When the energy density of distortion is low such that yielding is not imminent, the energy density of dilatation causes failure by microcavitation and subsequent crack initiation. This behavior suggests that in evaluating matrix behavior in fiber composites loaded transversely to fibers, both yielding and crack initiation from microcavitation must be considered.

Acknowledgements
Ms Kristiina Oksman and Mr. Philippe Genty are gratefully acknowledged for measurements of temperature dependence of Young's modulus and the biaxial tension test results.
REFERENCES


### Tables

#### Table I. Cure schedule and material composition.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>Cure</th>
<th>Post-cure</th>
<th>Material Composition (curing agent by weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>2 h/20 °C</td>
<td>24 h/102 °C</td>
<td>11.9 %</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>16 h/60 °C</td>
<td>24 h/102 °C</td>
<td>44.8%</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>4 h/150 °C</td>
<td>1 h/200 °C</td>
<td>44%</td>
</tr>
</tbody>
</table>

#### Table II. Yield stresses for epoxy systems under different load conditions.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>Uniaxial tension</th>
<th>Uniaxial compression</th>
<th>Plain strain compression</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\sigma_{yt}$</td>
<td>$\sigma_{yc}$</td>
<td></td>
</tr>
<tr>
<td>DGEBA/DETA</td>
<td>83 MPa*</td>
<td>113±5 MPa</td>
<td>122±3 MPa</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>78±1.2 MPa</td>
<td>91.4±2.0 MPa</td>
<td>96.2±1.6 MPa</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>no yield</td>
<td>207 MPa</td>
<td>no yield</td>
</tr>
</tbody>
</table>

*reference 22

#### Table III. Biaxial tension data, stresses at failure.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>$\sigma_x$ (MPa)</th>
<th>$\sigma_y$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>65</td>
<td>26</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>94</td>
<td>34</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>83</td>
<td>34</td>
</tr>
</tbody>
</table>

#### Table IV. Experimental data for the three epoxies. Thermal expansion coefficient, $\alpha$, measured in the interval 10-70°C.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>Poisson’s ratio $v$</th>
<th>Thermal expansion coefficient, $\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>0.345</td>
<td>$66 \cdot 10^{-6}$/°C</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>0.318</td>
<td>$66 \cdot 10^{-6}$/°C</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>0.328</td>
<td>$52 \cdot 10^{-6}$/°C</td>
</tr>
</tbody>
</table>

#### Table V. Active stresses at failure in the center of the poker-chip specimen (ref. Asp94), including thermal stresses from specimen preparation.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>normal stress</th>
<th>in-plane stresses</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>$\sigma_z$=27.6 MPa</td>
<td>$\sigma_z=\sigma_y=27.5$ MPa</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>$\sigma_z$=32.9 MPa</td>
<td>$\sigma_z=\sigma_y=33.8$ MPa</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>$\sigma_z$=30.5 MPa</td>
<td>$\sigma_z=\sigma_y=31.1$ MPa</td>
</tr>
</tbody>
</table>
### Table VI. Table of yield criteria. Left side gives value of yield criteria at poker-chip failure, including thermal stresses from specimen preparation. Right side gives predicted values at yielding. All units in MPa.

<table>
<thead>
<tr>
<th>Epoxy System</th>
<th>von Mises</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>$\sqrt{3J_2} \leq \sigma_y$</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>$0.1 &lt; 85$</td>
</tr>
<tr>
<td></td>
<td>$0.9 &lt; 78$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Epoxy System</th>
<th>Raghava</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>$\sqrt{6J_2 + 2(\sigma_{yc} - \sigma_{yr})I_1} \leq \sqrt{2\sigma_{yc} \sigma_{yr}}$</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>$70 &lt; 137$</td>
</tr>
<tr>
<td></td>
<td>$52 &lt; 119$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Epoxy System</th>
<th>Bauwens</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>$\sqrt{6J_2 + \sqrt{2}} \left(\frac{\sigma_{yc} - \sigma_{yr}}{\sigma_{yc} + \sigma_{yr}}\right)I_1 \leq \frac{2\sigma_{yc} \sigma_{yr}}{\sigma_{yc} + \sigma_{yr}}$</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>$18 &lt; 135$</td>
</tr>
<tr>
<td></td>
<td>$12 &lt; 119$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Epoxy System</th>
<th>Modified Tresca</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>$</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>$0.1 &lt; 83$</td>
</tr>
<tr>
<td></td>
<td>$0.9 &lt; 76$</td>
</tr>
</tbody>
</table>
Table VII. Young’s modulus of the tested epoxies at room temperature and at the corresponding failure temperature in the thermo-mechanical test.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>E (at room temperature)</th>
<th>E (at failure temp.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>2.07 GPa</td>
<td>3.85 GPa</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>2.93 GPa</td>
<td>4.81 GPa</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>3.77 GPa</td>
<td>5.84 GPa</td>
</tr>
</tbody>
</table>

Table VIII. Results from thermo-mechanical disk test, temperature change at failure, ultimate stress and strain.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>ΔT (°C)</th>
<th>σu (MPa)</th>
<th>εu (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>-269±17</td>
<td>54.1</td>
<td>0.92</td>
</tr>
<tr>
<td>DGEBA/APTA</td>
<td>-243±38</td>
<td>72.6</td>
<td>1.02</td>
</tr>
<tr>
<td>TGDDM/DDS</td>
<td>-219±30</td>
<td>54.8</td>
<td>0.63</td>
</tr>
</tbody>
</table>

Table IX. Test results, dilatational (U_v) and distortional (U_d) energy densities (MPa) at fracture for different test methods.

<table>
<thead>
<tr>
<th>Epoxy system</th>
<th>DGEBA/DETA</th>
<th>DGEBA/APTA</th>
<th>TGDDM/DDS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test method</td>
<td>U_v</td>
<td>U_d</td>
<td>U_v</td>
</tr>
<tr>
<td>Poker chip (3-D)</td>
<td>0.17</td>
<td>0.00</td>
<td>0.20</td>
</tr>
<tr>
<td>Thermally loaded epoxy disk (2-D)</td>
<td>0.16</td>
<td>0.33</td>
<td>0.27</td>
</tr>
<tr>
<td>Biaxial tension test (2-D)</td>
<td>0.21</td>
<td>0.70</td>
<td>0.34</td>
</tr>
<tr>
<td>Uniaxial tension</td>
<td>*</td>
<td>*</td>
<td>0.055</td>
</tr>
</tbody>
</table>

*Non-linear elastic behavior. U_v and U_d are defined from linear elasticity.
FIGURE CAPTIONS

Figure 1. Schematic of the sample design. A hybrid laminate consisting of an epoxy core inbetween two cross-ply laminates, causing a biaxial tensile stress state.

Figure 2. Schematic of specimen for the thermo-mechanical disk test.

Figure 3. Schematic of the insulated test chamber used for cooling of the ring specimen.

Figure 4. a) Nucleation and equiaxial growth of a cavity under equitriaxial tension. b) Crack formation from cavity and growth in a preferred direction normal to the maximum principle stress.

Figure 5. Scanning electron micrograph of the initiation point in a poker chip specimen of DGEBA/DETA.

Figure 6. Plane strain compression test set-up.

Figure 7. Stress-strain behavior of the DGEBA/APTA biaxial tension specimen.

Figure 8 Experimental data from the uniaxial compression test for the three epoxies.

Figure 9. Stress-strain curves from the plane strain compression test of DGEBA/DETA and DGEBA/APTA.

Figure 10. Comparison between experimental data for DGEBA/DETA and the different yield criteria predictions.

Figure 11. Comparison between experimental data for DGEBA/APTA and the different yield criteria predictions.
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Figure 9. Stress-strain curves from the plane strain compression test of DGEBA/DETA and DGEBA/APTA.
Figure 10. Comparison between experimental data for DGEBA/DETA and the different yield criteria predictions.
Figure 11. Comparison between experimental data for DGEBA/APTA and the different yield criteria predictions.
Prediction of matrix initiated transverse failure in polymer composites

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Abstract
A study is conducted of failure in unidirectionally reinforced fiber composites loaded in tension normal to fibers. The case considered is when this failure is governed by failure of the matrix rather than fiber/matrix debonding. Yielding as well as cavitation-induced brittle failure of matrix are considered. The latter mode of failure was suggested by a previous study\(^1\) as the likely mode to occur in epoxies under stress states that are purely or nearly hydrostatic tension. Three fiber packing arrangements (square, hexagonal and square-diagonal) with different fiber volume fraction are studied numerically by a finite element method to determine the local stress states. It is found that cavitation-induced brittle failure occurs much before yielding in all cases. Experimental data taken from the literature support this finding.

1. INTRODUCTION

When a glassy polymer such as epoxy is loaded uniaxially it displays different yield stress in tension and compression. This is attributed to the effect of hydrostatic stress on shear-driven yielding. The classical yield criteria, e.g. von Mises and Tresca, which are insensitive to the hydrostatic stress, have thus been modified to account for this effect\(^2-4\). Although the physical basis of the role of hydrostatic stress on the yield behavior is not fully understood, there is no doubt that in glassy polymers this stress

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component has a special significance. For instance, Sternstein and Ongchin\textsuperscript{5} found that craze formation in PMMA occurred in a biaxial stress field when the first invariant of the stress tensor was positive. They used this stress invariant in a criterion for dilatational yielding. Sultan and McGarry\textsuperscript{6} found that cavitation in glassy polymers was facilitated by particles and that the yield stress showed more sensitivity to the hydrostatic tensile stress when cavitation occurred than when it did not occur. This apparent coupling of dilatational and distortional effects in glassy polymers manifests in behavior not observed in crystalline materials such as metals.

Most studies reported in the literature have considered the dilatational effects on yield in polymers. One may ask the question: what if the distortional effects (deviatoric stress) are small and the dilatational effects (hydrostatic stress) are dominant? In other words, if yield is suppressed, what would the effect of the hydrostatic stress be? This question was considered in a previous study\textsuperscript{7} where the so-called poker chip test was performed on epoxies to introduce an equitriaxial tensile stress state. The apparent strain to failure was found to reduce dramatically. Based on the linear stress-strain behavior and appearance of failure surfaces, cavitation without yielding may be proposed as the cause of failure. In a later study\textsuperscript{1} it was postulated that cavitation within an elastic field in a glassy polymer will grow unstably and that this will occur when the stored dilatational energy density reaches a critical value. This criterion was evaluated by conducting tests on three epoxy systems in stress states ranging from uniaxial to biaxial with different principal stress ratios and finally to equitriaxial tension. As the stress state approached equibiaxial plane stress (where the distortional energy density is low) and equitriaxial tension (where the distortional energy density is zero), the modified yield criteria accounting for hydrostatic stress failed to predict the critical states. Instead, the dilatational energy density approached a constant value for these states. The critical value of the dilatational energy density was found also to be nearly the same at two different temperatures.

In composites loaded in tension normal to fibers, three competing initiation mechanisms can be expected to occur: fiber/matrix debonding, yield in matrix and cavitation-induced brittle matrix failure. In a real composite, which may have an irregular distribution of fibers, it is expected that the local stress states would vary such that the mix of deviatoric and dilatational stress components could vary from purely deviatoric to purely dilatational. Thus the matrix could yield in some regions while other regions could fail by cavitation-induced cracking. The fiber/matrix debonding
depends on not only the stress state at the fiber/matrix interface but also on a range of other factors such as the adhesion and toughness of the interface. Numerous studies of interfaces in composites have been conducted and this field is still very active. The present study is focused on stress field induced effects on the yielding and failure of matrix constrained within stiff fibers, assuming that the fiber/matrix bond remains intact. Three fiber packing arrangements - square, hexagonal and square-diagonal - are considered in a fiber composite loaded transversely in tension. A numerical stress analysis by the finite element method is conducted. The von Mises yield criterion and the dilatational energy density criterion are applied to locate the zones of yielding and cavitation-induced brittle failure, respectively. The results are obtained for different fiber volume fractions and against these the experimental data available in the literature are examined.

2. METHOD OF ANALYSIS

2.1. Materials
The analyzed composite consists of an epoxy system, DGEBA/DETA, reinforced by glass fibers. The mechanical properties of the epoxy system have been experimentally investigated in previous studies\(^1,7,8\). The material properties used in the analysis are presented in Table I. Recently, de Kok\(^10\) reported experimental transverse strength and modulus values for a glass fiber reinforced epoxy at different fiber volume fractions. Those data will be used here for comparison with our analysis. The material properties of the constituents in the composite used by de Kok are shown in Table II.

2.2. Material model and packing arrangements
Analysis of the local stresses in a fiber composite loaded in transverse tension was conducted using a commercial finite element code ANSYS\(^\circ\). Three fiber packing arrangements - square, hexagonal and square-diagonal - were analyzed, and for each case a unit cell was constructed. These are shown in Figure 1. Due to the uniformity and symmetry of the fiber packing arrangements, all quantities averaged over a unit cell are also averages over a representative volume element (RVE) of the composite. The matrix is assumed to be perfectly bonded to the fibers throughout the analysis. Both matrix and fibers are assumed to be linearly elastic. The volume fraction of fibers was varied between 20 and 70 or 80 percent in the RVEs depending on
the fiber distribution. Eight node "PLANE82" quadrilateral-triangular elements were used in the finite element code in all unit cells. The element has two degrees of freedom at each node. In this analysis the "PLANE82" element assumes a unit depth and was configured to model plane strain. The "PLANE82" element does not admit the assumption of generalized plane strain, i.e. that the average stress in the z-direction is zero and the strain in the z-direction at any point is nonzero but constant throughout the region. However, the differences in local stresses caused by the use of plane strain rather than generalized plane strain are expected to be small\textsuperscript{11}.

The unit cells were subjected to loading and boundary conditions representative for a state of transverse tensile loading, see Figure 2. For all fiber distributions, unit cell displacements in the x-direction were prohibited for all nodes on the left edge. Similarly, displacements in the y-direction were prohibited for all nodes on the lower edge. External stress was applied to the unit cell on the right edge by means of negative pressure, $\sigma_T$. To fulfil compatibility with neighboring unit cells, the upper and right edges were constrained to remain straight after deformation. Thermal stresses were computed under the assumption that the temperature is spatially uniform throughout the unit cell. Based on the maximum cure temperature, a temperature change of -82 °C was used in the analyses.

Radial, tangential (hoop) and z-directional stresses ($\sigma_r$, $\sigma_\theta$, $\sigma_z$), were calculated for the transverse tensile stress ($\sigma_T$) applied on the unit cells. To avoid problems due to stress averaging for dissimilar materials, matrix stresses were evaluated within selected elements.

FE-meshes of the three different unit cells are shown in Figures 3-5. To analyze a square array of fibers, the modelled unit cell is a quarter of a periodic element, see Figures 1 and 3. Past experience suggests that this model yields reasonable results\textsuperscript{12-14}. The unit cell of the hexagonal fiber array is modelled by one sixth (1/6) of the periodic element, see Figures 1 and 4. Also this unit cell is constrained to straight edges. A similar mesh was analyzed for metal matrix composites by Böhm et al.\textsuperscript{15}. As for the square fiber array, the RVE of a square-diagonal array is modelled by one quarter of the unit cell, see Figures 1 and 5. Similar meshing has been used in the analyses of metal matrix composites by Brockenbrough et al.\textsuperscript{12}. 
2.3. Failure criteria

Two different criteria are used to predict failure initiation in the matrix within the composite. The criterion first to reach its critical value in any point of the matrix is assumed to initiate failure in the GF/EP composite.

First, the dilatational energy density criterion assumes failure to initiate in the matrix material of a composite due to the induced triaxial stress state. The dilatational energy density criterion was proposed for cavitation-induced failure in a previous study\(^1\). The dilatational (volumetric) energy density for a linear elastic material is given by

\[
U_v = \frac{1}{6E} \left( \sigma_1 + \sigma_2 + \sigma_3 \right)^2
\]  

(1)

where \(\sigma_1, \sigma_2,\) and \(\sigma_3\) are the principal stresses, \(v\) and \(E\) are the Poisson's ratio and Young's modulus, respectively. Cavitation-induced brittle failure is assumed to occur at a point when this quantity attains a critical value \(U_v^c\). This material parameter is obtained by an equitriaxial test such as the poker chip test. \(U_v^c\) for DGEBA/DETA epoxy was obtained in a previous study\(^1\). The critical dilatational energy density appears not to depend on temperature, while the critical hydrostatic stress is temperature dependent due to the temperature dependency of the elastic properties.

The second criterion used here is the von Mises yield criterion. Yield stress of glassy polymers is known to be sensitive to hydrostatic pressure. The von Mises yield criterion does not take the dependence of hydrostatic pressure into account. However, the influence of the hydrostatic pressure will result in the von Mises effective stress becoming an underestimate of the true yield stress in the first quadrant and an overestimate elsewhere\(^1\). Also, it was shown in the referred study that failure in multiaxially tensile loaded (first quadrant) DGEBA/DETA epoxy was not caused by yielding. Therefore, the von Mises yield criterion will give a conservative estimation of yield initiated failure.

3. RESULTS AND DISCUSSION

3.1. Local stresses and failure initiation sites

We examine two modes of failure: cavitation-induced cracking and yielding, and assume that each occurs when the local stress state becomes critical for that mode of failure. The failure initiation sites in a unit cell are determined
by applying the dilatational energy density criterion for cavitation-induced cracking and the von Mises criterion for yielding. The location of these sites are presented in Figure 6. The sites of cavitation-induced failure in all three fiber packing configurations lie at the fiber poles, i.e., at intersections between the fiber center-line parallel to the loading axis and the fiber surface. The sites of yielding, on the other hand, depend on the fiber packing configuration. Thus, for the square distribution the sites are at the fiber equators, $\theta=90^\circ$, while for the hexagonal distribution these are at $\theta=60^\circ$ and for the square-diagonal the sites are at $\theta=45^\circ$ or $\theta=0^\circ$. In the last case of $\theta=0^\circ$, the location of yielding is not at the fiber pole but at the edge of the unit cell.

The failure initiation sites for the two modes of failure do not coincide since the underlying failure criteria are based on two different energy densities. Indeed, the three principal stresses at the fiber poles are nearly equal when the thermal stresses are included. Thus, the deviatoric energy density is almost zero at these points. At a point in a unit cell where yielding occurs the dilatational energy density reaches its minimum.

### 3.2. Critical failure mode and transverse failure predictions

The critical dilatational energy density and yield stress of DGEBA/DETA were investigated in a previous study\(^1\) and found to be 0.2 MPa and 83 MPa, respectively. The calculated dilatational energy density and von Mises effective stress in the matrix are compared to its critical dilatational energy density and yield stress for each considered fiber volume fraction and fiber distribution. In no case does the von Mises effective stress reach the yield stress before the dilatational energy density reaches its critical value. In fact, out of all the considered cases the largest value of the von Mises effective stress at initiation of cavitation-induced brittle failure was only 52 MPa. This was for a square-diagonal fiber distribution at a volume fraction of 0.7. Thus, the cavitation-induced brittle failure is the more critical one out of the two modes of failure considered. Also, quite importantly, this failure mode becomes critical when the stress state in the surrounding matrix material is still much below that required for yielding. The conditions for brittle failure are therefore present.

Although the cavitation-induced cracks will initiate at the fiber poles, the growth of these cracks into the surrounding matrix may not occur instantaneously. As a crack grows it will enter zones where the stress field is less intensive than at the fiber poles. Also, the crack tip will induce high stresses locally causing yielding. Thus the crack tip can slow down or even be arrested. An increased stress would then have to be imposed to cause further
crack growth, crack link-up and failure. Since the experimental data available is for transverse strength at total failure (separation) we shall compare our numerical predictions of failure initiation with this value.

Predicted transverse strength (transverse failure initiation) of fiber composites is found to be strongly dependent on the fiber volume fraction and fiber distribution, see Figure 7. The effect of fiber volume fraction on transverse strain to failure is strong, as seen in Figure 8. The most severe of the analyzed fiber distributions for GF/EP composites are hexagonal and square arrays, as shown in Figures 7 and 8. Transverse strength and strain to failure of square and hexagonal fiber distributions are significantly lower than those for the square-diagonal array. The square-diagonal array is a square array tilted $45^\circ$ to the load direction. The altered load direction results in an increase in strength except for high volume fractions of fiber ($V_f=0.7$) and a general increase in strain to failure, as seen in Figures 7 and 8, due to the lowered transverse modulus. These results imply that the presence of regions with square or hexagonal fiber distributions will control failure initiation in a transversely loaded GF/EP composite. For these fiber distributions it is important to observe the dependence of transverse strength on fiber volume fraction, see Figure 7. According to the dilatational energy density criterion, minima in transverse strength for intermediate fiber volume fractions are predicted. As a consequence, the transverse strength of a GF/EP composite with an average fiber volume fraction of 0.4-0.6 will not be weakened by fiber agglomerates as they are of high local fiber volume fractions and therefore can sustain higher loads than their surroundings.

The observation that failure will initiate in regions of square and hexagonal fiber distributions provides a possibility to extract information about composite microstructure at the failure initiation point from experimental transverse tensile strength data. However, as the fibers are more or less randomly distributed in the matrix, such low strength regions in which failure will initiate are always present. Measured strain to failure of transversely loaded composites will not provide any information about the fiber distribution in the composite. The reason for this is simply that the measured strain is an average value over the whole gauge length rather than that at the initiation point.

The strong effect of fiber volume fraction on transverse moduli for the analyzed fiber distributions is demonstrated in Figure 9. The transverse modulus is higher for the square fiber array than for the hexagonal. This result is confirmed in the literature, cf. the study presented by Adams and
Tsai\textsuperscript{16} and de Kok\textsuperscript{10}. The square-diagonal fiber array has the lowest stiffness of the analyzed fiber distributions. Brockenbrough et al.,\textsuperscript{12} analyzed a square-diagonal fiber distribution for a MMC material and compared the results to those of square and hexagonal fiber distributions. Their result showed on differences under yielding of MMC materials. However, no significant differences in modulus were reported between hexagonal, square and diagonal fiber arrays in the linear elastic region.

3.3. Effect of thermal stresses on transverse failure

According to the dilatational energy density criterion, presence of thermal residual stresses causes a general decrease in transverse strength of the GF/EP composite, as shown in Figures 10-12. However, for high fiber volume fractions ($V_f=0.6-0.8$) of square as well as hexagonal fiber distributions, no such decrease in strength is predicted. On the contrary, the presence of thermal stresses in a densely packed square fiber array causes an increase in the composite transverse strength as seen in Figure 10. The thermal residual stresses in the square and square-diagonal arrays are identical. Still the influence of thermal residual stresses is beneficiary on composite strength (high $V_f$) in the case of square and disadvantageous for the square-diagonal fiber distribution, see Figures 10 and 12. This is explained by that the local stresses due to mechanical loading are different as the load angle is changed. In the strength analysis, the residual thermal stresses are superimposed on the local mechanical stresses. Therefore, the influence of thermal stresses on transverse composite strength is dependent on the fiber distributions present and the direction of the applied load. Residual thermal stresses as well as mechanical stresses in the matrix depend strongly on position and fiber distribution. General features of the residual thermal stresses are therefore difficult to describe.

As stated above, failure initiation in all analyzed cases takes place at the fiber poles. In the cases of square and hexagonal fiber arrays, failure always initiates at the poles regardless of thermal residual stresses. For higher fiber volume fractions of the square-diagonal array ($V_f$ above 0.5), thermal residual stresses constrain the failure initiation location in the matrix to the fiber poles, as seen in Figure 13. Figure 13 shows the effect of thermal residual stresses on the failure initiation position in a square-diagonal fiber array. Disregarding thermal stresses, for a fiber volume fraction of 0.6 (Figure 13a) failure initiates at the poles as well as inbetween fibers simultaneously. Further increase in fiber volume fraction to 0.7 reduces the failure initiation volume as the
initiation site moves to a position inbetween fibers. As a consequence, localization of stresses to a smaller volume in the matrix causes a drop in the transverse strength. In Figure 13b, transition of failure initiation position is shown to be prohibited by the presence of thermal residual stresses.

3.4. Comparison with composite data
Failure initiation predictions by the dilatational energy density criterion are compared to experimental data reported by de Kok\textsuperscript{10}. The procedure is the following. Stress analysis by the finite element method is performed on a square fiber array ($V_f=0.46$) using the material properties of the composite tested by de Kok\textsuperscript{10}, see Table II. The stress at transverse failure measured by de Kok is applied to the square array. The dilatational energy density in the matrix at failure is then computed and found to be $U_f^{\text{crit}} = 0.40$ MPa. The analysis includes thermal residual stresses for a thermal cool-down of 120°C. Predictions of transverse strength and strain to failure are then made for other fiber contents on the basis of $U_f^{\text{crit}} = 0.40$ MPa. Furthermore, failure initiation due to matrix yielding was predicted by the von Mises yield criterion. The yield stress at room temperature was 94.2 MPa\textsuperscript{10}.

Comparison with experimental data reported by de Kok\textsuperscript{10} is made based on the described fitting procedure, see Figures 14 and 15. The predictions based on the dilatational energy density criterion are close to experimental data. Also, the predicted increase in strength for high fiber volume fractions is supported by the experimental data. Strength and strain to failure predictions by the von Mises yield criterion are also presented in Figures 14 and 15. The results imply that failure initiation due to yielding is always preceded by cavitation-induced brittle failure.

The critical dilatational energy density, $U_f^{\text{crit}}$, is a material property and is expected to vary between different epoxy systems. For epoxies previously studied this variation was fairly small\textsuperscript{1}. $U_f^{\text{crit}}$ extracted from the experimental data of de Kok is significantly higher (50 percent) than that of DGEBA/DETA. Our interpretation is therefore that the experimental transverse strength will generally be higher than that predicted by the dilatational energy density criterion. We may discuss the difference between transverse stress at failure as predicted by $U_f^{\text{crit}}$ and the experimental transverse strength. Let us call this difference "additional stress". The additional stress reflects crack growth processes and will among other factors depend on the matrix yield stress and the fiber packing configuration. Assuming that the additional stress is the same at all fiber volume fractions, transverse strength predicted using $U_f^{\text{crit}}$
obtained from one fiber volume fraction, will hold for all fiber volume fractions. This is supported by comparisons with de Kok's data (see Figures 14 and 15). The predicted and somewhat surprising increase in transverse strength at high fiber contents is also supported by experimental data. This verifies the effect of thermal residual stresses on transverse strength observed in the finite element analysis.

The trends of transverse composite strength for RVEs of square and hexagonal fiber distributions follow the trend of the experimental data by de Kok (see Figures 7 and 8). In addition, predictions based on one data point (Vf=0.46) agree with the experimental data for other fiber volume fractions. Although this may be interpreted as being in support of the dilatational energy density criterion we need to keep in mind that interfacial debonding is a competing mechanism which cannot be excluded.

4. CONCLUSIONS

The study reported here shows that in unidirectional fiber composites of glass fiber/epoxy loaded in transverse tension, failure in the matrix initiates by cavitation-induced cracks. This failure is found to occur earlier than yielding at all fiber volume fractions. The location of the maximum dilatation energy density in the matrix is found to lie in regions close to the fiber/matrix interface. These regions lie along the tensile stress axis passing through the centers of fiber cross sections. The calculated trends in the composite failure stress and strain with fiber volume fraction agree with the experimental data for one glass fiber/epoxy material.
REFERENCES


Table I. Material properties of composite constituents.

<table>
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<tr>
<th>Material</th>
<th>Young's modulus [GPa]</th>
<th>Poisson's ratio [-]</th>
<th>Thermal expansion coefficient $[10^{-6}/^\circ\text{C}]$</th>
<th>Tg $[^\circ\text{C}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>E-glass (ref. 9)</td>
<td>72</td>
<td>0.200</td>
<td>5</td>
<td>-</td>
</tr>
<tr>
<td>DGEBA/DETA</td>
<td>2.07</td>
<td>0.345</td>
<td>66</td>
<td>107</td>
</tr>
</tbody>
</table>

Table II. Material properties of the constituents of the composite tested by de Kok (ref. 10).

<table>
<thead>
<tr>
<th>Material</th>
<th>Young's modulus [GPa]</th>
<th>Poisson's ratio [-]</th>
<th>Thermal expansion coefficient $[10^{-6}/^\circ\text{C}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>E-glass</td>
<td>70</td>
<td>0.22</td>
<td>7</td>
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<tr>
<td>Epoxy</td>
<td>3.2</td>
<td>0.37</td>
<td>67.5</td>
</tr>
</tbody>
</table>
Figure Captions

Figure 1. Schematic of a loaded RVE for a square array of fibers and the corresponding coordinate system, z-direction out of the paper.

Figure 2. Schematics of fiber configurations; a) Square fiber array, b) Hexagonal fiber array and c) Square-diagonal fiber array.

Figure 3. FEM-mesh of a periodic element in a square fiber array.

Figure 4. FEM-mesh of a periodic element in a hexagonal fiber array.

Figure 5. FEM-mesh of a periodic element in a square-diagonal fiber array.

Figure 6. Schematic of the loaded periodic element of a square fiber array. The zone locations of maximum dilatational and von Mises effective stresses are indicated. The coordinate system is the same as in Figure 1.

Figure 7. Predicted transverse strength (transverse failure initiation, $\sigma_{ult}$) as a function of fiber volume fraction ($V_f$) for three different unit cells.

Figure 8. Predicted strain to failure (transverse failure initiation, $\varepsilon_{ult}$) as a function of fiber volume fraction ($V_f$) for three different unit cells.

Figure 9. Predicted transverse modulus ($E_T$) as a function of fiber volume fraction ($V_f$) for three different unit cells.

Figure 10. Transverse strength (transverse failure initiation, $\sigma_{ult}$) of a square array as a function of fiber volume fraction, with and without thermal stresses ($\Delta T=-82^\circ C$).

Figure 11. Transverse strength (transverse failure initiation, $\sigma_{ult}$) of a hexagonal array as a function of fiber volume fraction, with and without thermal stresses ($\Delta T=-82^\circ C$).

Figure 12. Transverse strength (transverse failure initiation, $\sigma_{ult}$) of a square-diagonal array as a function of fiber volume fraction, with and without thermal stresses ($\Delta T=-82^\circ C$).

Figure 13. Initiation volumes (marked black) in the matrix material of a transversely loaded square-diagonal array ($\Delta T=-82^\circ C$). a) No thermal stresses included in the analysis and b) Thermal stresses included in the analysis.

Figure 14. Experimental data$^{10}$ for transverse strength of GF/EP versus fiber volume fraction. Predictions based on von Mises yield criterion and the dilatational energy density criterion.
Figure 15. Experimental data\textsuperscript{10} for transverse strain to failure of GF/EP versus fiber volume fraction. Predictions based on von Mises yield criterion and the dilatational energy density criterion.
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Figure 11. Transverse strength (transverse failure initiation, $\sigma_{ult}$) of a hexagonal array as a function of fiber volume fraction, with and without thermal stresses ($\Delta T=-82^\circ C$).
Figure 12. Transverse strength (transverse failure initiation, $\sigma_{\text{ult}}$) of a square-diagonal array as a function of fiber volume fraction, with and without included thermal stresses ($\Delta T=-82^\circ C$).
a) No thermal stresses

\[ V_f \leq 0.5 \quad V_f = 0.6 \quad V_f = 0.7 \]

b) With thermal stresses

\[ V_f \leq 0.5 \quad V_f = 0.6 \quad V_f = 0.7 \]

Figure 13. Initiation volumes (marked black) in the matrix material of a transversely loaded square-diagonal array (\(\Delta T = -82^\circ\text{C}\)). a) No thermal stresses included in the analysis and b) Thermal stresses included in the analysis.
Figure 14. Experimental data\textsuperscript{10} for transverse strength of GF/EP versus fiber volume fraction. Predictions based on von Mises yield criterion and the dilatational energy density criterion.
Figure 15. Experimental data\textsuperscript{10} for transverse strain to failure of GF/EP versus fiber volume fraction. Predictions based on von Mises yield criterion and the dilatational energy density criterion.
Effects of fiber and interphase on matrix initiated transverse failure in polymer composites

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Abstract
Failure initiation in polymer matrix composites loaded transverse to fibers is investigated by a numerical parametric study where the effects of constituent properties as well as interphase properties and thickness are examined. Failure initiation in matrix only is studied; interfacial debonding is not considered. Two modes of failure - yielding and cavitation-induced brittle failure - are examined. A criterion for the cavitation-induced brittle failure was proposed in a previous study\(^1\) and failure prediction based on this criterion was found to agree with experimental data for a glass fiber reinforced epoxy\(^2\). The present study shows that the elastic modulus of fibers has a large effect on the stress and strain to failure initiation. A rubbery interphase material is found in most cases to have a beneficial effect. The site at which failure initiates and the governing mode of failure initiation are also affected by the fiber modulus and the interphase properties.

1. INTRODUCTION
Transverse failure is one of the most important failure modes in polymer composites. This phenomenon often causes the first deviation from linear laminate behavior. Also, in pressure vessels and pipes, fluid leakage through a path of transverse cracks is often the limiting design condition. Since the fibers are usually much stronger than the matrix, one may postulate two

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primary mechanisms of failure initiation. One is fiber/matrix debonding and the other is matrix failure. In this study we focus on failure initiation in the matrix. The motivation for this lies in the observation that regardless of the value of the matrix failure strain, the composite failure strain in transverse loading falls in the range 0.2-0.9 percent. In the literature, explanations for low composite failure strain such as presence of voids, non-uniform fiber distribution, the multiaxial stress-state in the matrix and fiber/matrix debonding have been suggested\textsuperscript{3-6}. An earlier study by Nicholls\textsuperscript{5} demonstrated a significant reduction in strain to failure in biaxial loading as compared with uniaxial tensile loading. In the present and previous studies\textsuperscript{1,7,8} we have therefore focused on the matrix failure behavior due to the multiaxial stress state.

A convenient way to study the effects of triaxial stress states is by the poker-chip test which has earlier been used for rubbers\textsuperscript{9,10}. In a previous study this test was used for glassy epoxies\textsuperscript{7} where an equitriaxial stress state was generated. Compared to the uniaxial failure strains of neat resins, significant reductions in the failure strains were found in this composite-like stress state. The mechanism proposed for this failure mode was cavitation-induced brittle failure and the critical dilatational energy density was proposed as a criterion\textsuperscript{1}. Experimental data for three epoxies provided support for the proposed criterion. A numerical study was then conducted to investigate failure initiation in transversely loaded composites with different fiber packing arrangements and fiber volume fractions\textsuperscript{2}. Yielding as well as cavitation-induced brittle failure were examined and it was found that in all cases the critical equitriaxial stress state was reached before the critical stress state for yielding. Experimental data for a glass fiber/epoxy composite of different fiber volume fractions\textsuperscript{4} supported the calculated trends in the applied stress at initiation of local matrix failure.

Motivated by the studies described above we set out to seek ways of improving the failure behavior of transversely loaded composites. To examine the effects of constituents and interphase materials we conduct a parametric numerical study using the finite element method (FEM). The interphase thickness is also included as a parameter. Fibers are placed in a square array at a fixed fiber volume fraction. The local stress states are computed and the criteria for yielding and cavitation-induced brittle failure are applied. In order to locate the position at which debonding is most likely to occur, the points of maximum radial tensile stress at the interfaces are also calculated.
2. METHOD OF ANALYSIS

2.1. Materials
The analyzed composites are unidirectional glass fiber reinforced epoxy (GF/EP) and carbon fiber reinforced epoxy (CF/EP). The epoxy is based on DGEBA, which is diglycidyl ether of bisphenol A (DER 332, Dow Chem Co). The DGEBA is cured by DETA, diethylenetriamine (DEH 20, Dow Chem Co). The mechanical properties of the epoxy system have been determined in previous studies\textsuperscript{1,7,8} and are presented in Table I. Glass fiber properties are given in the same table. Carbon fiber properties are presented in Table II. The analyzed composites have a fiber volume fraction of 0.5. The effect of thin interphases between fiber and matrix on composite properties was studied by FEM. Interphase properties are given in Table III.

2.2. Material modeling and packing arrangements
Analysis of the local stresses in a fiber composite loaded in transverse tension was conducted using a commercial finite element code ANSYS\textsuperscript{®}. A square fiber packing arrangement was analyzed, for which a unit cell was constructed. Due to the uniformity and symmetry of the fiber packing arrangement, all quantities averaged over a unit cell are also averages over a representative volume element (RVE) of the composite. Two different unit cells were modelled, a first consisting of two phases (fiber and matrix) and a second in which a third phase interphase is introduced. Throughout the analysis the matrix is assumed to be perfectly bonded to the fibers and interphase. Both matrix, fibers, and interphase are assumed to be linearly elastic. The volume fraction of fibers was constant at 50 percent in the RVE. Analysis of three phase composites was conducted for three thicknesses of the interphase. The analyzed interphase thicknesses were 1, 5, and 10 percent of the fiber radius. Eight node "PLANE82" quadrilateral-triangular elements were used in the finite element code in all unit cells. The element has two degrees of freedom at each node. In this analysis the "PLANE82" element assumes a unit depth and was configured to model plane strain. The "PLANE82" element does not admit the assumption of generalized plane strain, i.e. that the strain in the z-direction at any point is non-zero but constant throughout the region. However, the differences in local stresses caused by the use of plane strain rather than generalized plane strain are expected to be small\textsuperscript{11}.
The unit cells were subjected to loading and boundary conditions representative of a state of transverse tensile loading. Unit cell displacements in the $x$-direction were prohibited for all nodes on the left edge. Similarly, displacements in the $y$-direction were inhibited for all nodes on the lower edge. External stress was applied to the unit cell on the right edge by means of negative pressure, $\sigma_T$. To fulfil compatibility with neighboring unit cells, the upper and right edges were constrained to remain straight after deformation. Thermal stresses caused by different coefficients of thermal expansion for matrix and fiber were computed under the assumption that the temperature is spatially uniform throughout the unit cell. A temperature change of $-82^\circ C$ based on the difference between cure temperature and room temperature was used in the analyses.

Radial, tangential (hoop) and $z$-direction stresses ($\sigma_r$, $\sigma_\theta$, $\sigma_z$), were calculated as a transverse tensile stress ($\sigma_T$) was applied to the unit cells. To avoid erroneous interpretations of the stress state in the matrix due to stress averaging for dissimilar materials, matrix stresses were evaluated within selected elements. FE-meshes of two different unit cells are shown in Figure 1. To analyze a square array of fibers, the modelled unit cell is a quarter of a periodic element. Past experience suggests that this model yields reasonable results.

### 2.3. Failure criteria

Two different criteria are used to predict failure initiation in the matrix within the composite. The criterion which first reaches its critical value at any point in the matrix is assumed to initiate failure in the GF/EP composite.

The first criterion considered is the dilatational energy density criterion which assumes failure to initiate in the matrix material of a composite due to the induced triaxial stress state. In a previous study, the dilatational energy density criterion was proposed for cavitation-induced failure based on the critical dilatational energy density of the matrix. A second study showed that in unidirectional GF/EP composites loaded in transverse tension, matrix failure initiates by cavitation-induced cracks. The dilatational (volumetric) energy density for a linear elastic material is given by

$$U_v = \frac{1-2v}{6E}(\sigma_1 + \sigma_2 + \sigma_3)^2$$

where $\sigma_1$, $\sigma_2$, and $\sigma_3$ are the principal stresses, $v$ and $E$ are the Poisson's ratio and Young's modulus, respectively. Cavitation-induced brittle failure is
assumed to occur at a point when this quantity attains a critical value \( (U_{\text{crit}}) \).

This material parameter is obtained by an equitriaxial test such as the poker-chip test. For DGEBA/DETA epoxy this value was obtained in a previous study\(^1\). As discussed there the critical dilatational energy density appears not to depend on temperature, while the critical hydrostatic stress is temperature dependent due to the temperature dependency of the elastic properties.

The second criterion used is the von Mises yield criterion. The yield stress, \( \sigma_Y \), of glassy polymers is known to be sensitive to hydrostatic pressure\(^{15} \). The von Mises yield criterion does not take the dependence of hydrostatic pressure into account. As a consequence, von Mises yield criterion will overestimate the yield stress in the first quadrant of the stress plane, while in the other quadrants the yield stress will be underestimated.

The critical values of the dilatational energy density and the yield stress of the DGEBA/DETA matrix material were determined experimentally in a previous study\(^1\) and are presented in Table I.

3. RESULTS AND DISCUSSION

Failure initiation was predicted by the dilatational energy density criterion as well as the von Mises yield criterion. The criterion first to reach its critical value in any region of the matrix is assumed to predict the failure initiation mode. Also, calculations of maximum radial stress at fiber/matrix and interphase/matrix interfaces were made. However, predictions of debonding were not performed as interfacial failure properties for the analyzed materials are not available.

3.1. Fiber reinforced epoxy

A parametric study was performed to determine the influence of fiber properties on the transverse stress at and strain to failure initiation of epoxy based composites with fiber volume fractions of 0.5. The fiber properties varied are: Modulus, Poisson's ratio and thermal expansion coefficient. The values of the fixed properties in the parametric study are those of glass fiber with exception of Poisson's ratio which is set to 0.4. This value is a compromise between \( v_f = 0.2 \) for glass and \( v_f = 0.5 \) for rubber fibers. The choice of \( v_f \) is not critical since the present study shows that transverse failure predictions are not so sensitive to \( v_f \).
3.1.1. Transverse failure initiation predictions

The results of transverse stress at failure initiation versus fiber modulus are presented in Figure 2. The graph shows transverse failure initiation stress, \( \sigma_{ult} \), to depend strongly on the modulus of the reinforcement. A peak in the transverse stress at failure initiation is shown for a fiber modulus equal to that of the matrix. The strain to failure initiation, \( \varepsilon_{ult} \), as a function of fiber modulus is plotted in Figure 3. These results imply a fiber modulus equal to that of the matrix to be the most beneficial (Note, however, that the Poisson's ratio of the two constituents are different.). For stiff fibers (stiffer than the matrix) a large decrease in strain to failure initiation was observed. However, for fibers softer than the matrix the drop in strain to failure initiation is small due to a decrease in transverse composite modulus.

There was no dependence of transverse stress at failure initiation on Poisson's ratio for a fiber modulus of 72 GPa. A softer fiber (Ef=1 GPa) was therefore investigated. For Ef=1 GPa, the effect of Poisson's ratio of the fiber on transverse strength was small. Stress at failure initiation varied only between 26 and 27.5 MPa.

The effect of thermal expansion coefficient of the fiber and the resulting residual thermal stresses on transverse stress at failure initiation was found to be small. The transverse stress at failure initiation increased with an increase in fiber thermal expansion coefficient. As Ef increased from 0 to 100\(\times10^{-6}/^\circ\mathrm{C}\) the transverse stress at failure initiation increased from 23.5 MPa to 27.8 MPa. The transverse composite modulus was independent of residual thermal stresses.

As a conclusion, the effects of fiber Poisson's ratio and thermal expansion coefficient on the stress at failure initiation are small compared to the effect of fiber Young's modulus.

3.1.2. Failure mode and failure initiation site

For all values of the fiber modulus considered here failure initiation is caused by the dilatational energy density attaining its critical value. Even for the case of equal fiber and matrix moduli, a triaxial stress state is present due to differences in Poisson's ratio and thermal expansion coefficient. However, a transition in position of the initiation region occurs for equal fiber/matrix modulus. For stiff fibers failure was predicted to initiate at the fiber poles, whereas it was predicted to initiate at the fiber equators of soft fibers. This transition, in addition to lowered transverse modulus, results in a decrease in composite failure initiation stress for fibers softer than the matrix. Nevertheless, failure initiation due to high dilatational energy density is likely
to be suppressed for soft fiber composites since the distortional energy density has its maximum in the same region. If fiber/matrix debonding occurs, it is likely to initiate at the site of maximum radial stress. The position of the maximum radial stress at the interface was found to depend on fiber modulus. For fibers of the same stiffness or stiffer than the matrix, the maximum radial stress is located at the fiber poles. This coincides with the position of maximum dilatational energy density. However, for soft fibers the positions of maximum radial stress and dilatational energy density are separated. The maximum radial stress on the interface is located at an angle of 45° to the load direction, whereas the maximum dilatational energy density is at the fiber equators. For the case of glass fiber/epoxy, the fiber is significantly stiffer than the matrix. The two major failure mechanisms, debonding and cavitation-induced brittle failure, will therefore both occur at the fiber poles. Since a matrix-initiated crack may quickly grow to the interface, it may be difficult to experimentally distinguish between the two mechanisms.

Failure initiation was unaffected by variations in Poisson's ratio. For a fiber Young's modulus of 72 GPa, failure initiated at the fiber poles due to the high dilatational stresses. For a fiber modulus of 1 GPa, failure was also due to high dilatational stresses, but the location of failure was at the fiber equators.

For each choice of \( \alpha_f \), failure was found to initiate due to high dilatational stresses. For a thermal expansion coefficient of the fiber which was lower or equal to that of the matrix, failure initiated at the fiber poles. However, for a high thermal expansion coefficient of the fiber (\( \alpha_f=100\times10^{-6}/°C \)), the maximum von Mises effective stress moved to the same region as the maximum dilatational energy density. Thus, the region of maximum von Mises effective stress was observed to move from the fiber equators as \( \alpha_f \) was set to a value significantly higher than that of the matrix. This change in position of maximum von Mises effective stress is due to a change in the residual thermal stress state, since tensile residual stresses in the matrix become compressive and vice versa.

3.1.3. Special cases: CF/EP and GF/EP

Analyses of two carbon fiber/epoxy composites have been performed. Material data for the Type I and Type II carbon fibers are presented in Table II. The transverse stresses at failure initiation as a function of fiber volume fraction for the two carbon fiber composites are depicted in Figure 4. For
comparison, the results for a glass fiber composite are presented in the same graph. The predictions imply that failure in carbon fiber composites initiates roughly at the same transverse stress level as in glass fiber composites. This has been experimentally verified. Baron et al.\textsuperscript{16} reported transverse strengths for carbon fiber/epoxy composites in the range between 35 and 50 MPa. These data correspond well with those for glass fiber/epoxy composites by de Kok\textsuperscript{4}. The curves in Figure 4 all show the same trends. Minima in transverse stresses at failure initiation are found at a fiber volume fraction of 0.5 in all three cases. Transverse modulus of carbon fiber composites is lower than that of glass fiber composites. Thus, failure initiation at the same stress levels results in higher strains to failure for carbon fiber composites. The results for transverse moduli and strain to failure initiation are depicted in Figures 5 and 6.

Failure predictions suggest transverse failure to initiate at the fiber poles due to high dilatational stresses in all three composites. The lower predicted transverse strength as compared with experimental data may be explained by either interfacial debonding or that the dilatational energy density criterion predicts failure initiation rather than final composite failure. As the stress state within the matrix varies, a growing crack may arrest in regions of high shear stresses\textsuperscript{2}.

3.2. Fiber reinforced epoxy with interphase
A parametric study was performed in order to demonstrate how mechanical property variations of a third phase interphase affect transverse properties. A glass fiber reinforced epoxy with a fiber volume fraction of 0.5 was studied. Mechanical properties of epoxy and glass fiber are presented in Table I. The mechanical properties varied in the interphase are: Modulus, Poisson's ratio, and thermal expansion coefficient. In addition, interphase thickness (t) was varied between 1 and 10 percent of the fiber radius (R). In the presented figures the normalized interphase thickness is expressed as (t/R). The values of the fixed properties in the parametric study are those of the thermoplastic interphase in Table III. In the graphs presented, curves for each set of data for $E_i/E_m$, $v_i/v_m$ and $\alpha_i/\alpha_m$ are fitted by interpolation. $E_i/E_m$, $v_i/v_m$ and $\alpha_i/\alpha_m$ are interphase modulus, Poisson's ratio, and thermal expansion coefficients normalized with respect to the corresponding matrix property.
3.2.1. Transverse failure initiation predictions

The results of variation in interphase modulus for the three different interphase thicknesses are presented in Figures 7 and 8. The stress at failure initiation strongly depends on the interface thickness for soft interphases, see Figure 7. The stress at failure initiation decreases with increased low modulus interphase thickness. However, for interphase moduli close to or stiffer than that of the matrix, stress at failure initiation is independent of interphase thickness. It is interesting to note that the strain to failure is significantly higher for soft interphases, as shown in Figure 8.

The results of variations in interphase Poisson's ratio are presented in Figures 9 and 10. A general increase in transverse strain to failure with increased interphase thickness is apparent. Strain to failure initiation is significantly affected by the interphase Poisson's ratio. For an interphase thickness of 10 percent of the fiber radius, a difference of 40 percent in strain to failure is observed between \( v_i/v_m \) of 0.58 and 1.45. Low strain to failure in Figure 9 is a consequence of increased transverse composite modulus for high Poisson's ratios, see Figure 10. The increase in transverse composite modulus with increased interphase Poisson's ratio is of interest. For Poisson's ratios lower than 0.333 the bulk modulus, \( K \), will be smaller than the Young's modulus of the interphase since

\[
K = \frac{E}{3(1-2\nu)}
\]

In Equation (2), \( E \) and \( \nu \) are the Young's modulus and Poisson's ratio, respectively. Thus, for Poisson's ratios above 0.333 the modulus of the constrained interphase will approach the value of its bulk modulus. According to Equation (2), the modulus of a constrained interphase approaches the bulk modulus as the Poisson's ratio is larger than 0.333. Ellis et al.\(^{17} \) pointed out the possibility of this effect on thin rubbery interphases in glass fiber/vinyl ester composites. Although strain to failure is strongly influenced by \( v_i \), the effect on stress at failure initiation, \( \sigma_{ult} \), is small. A general increase in \( \sigma_{ult} \) of 4 to 10 percent is observed.

Positive effects of increased interphase thermal expansion coefficient and thickness on \( \sigma_{ult} \) are demonstrated in Figure 11. This is because of favorable changes in thermal stresses. No change in transverse composite modulus was observed. Hence, a general increase in strain to failure initiation followed with the increase in stress at failure initiation.
3.2.2. Failure mode and failure initiation site
Failure initiation is always predicted to be due to high dilatational energy density rather than distortional stresses. However, the position of initiation site is affected by interphase modulus. For all interphase thicknesses with $E_i/E_m=0.0048$, initiation occurs at the fiber equators. For the other examined $E_i/E_m$ ratios, failure initiated at the fiber poles.

Failure initiation is unaffected by $v_i$ and $\alpha_i$. Failure initiates due to high dilatational energy densities at the fiber poles for each investigated interphase Poisson's ratio, thermal expansion coefficient and thickness. The regions of maximum von Mises effective stress are always located at the fiber equators.

Maximum radial stress at fiber/interphase and interphase/matrix interfaces were found at the fiber poles. Hence, in the case debonding occurs it will initiate in the same region as cavitation-initiated brittle failure of the matrix.

3.2.3. Special case interphases
Analyses of three different interphase GF/EP composites were performed. The interphases consisted of: A rubber, a thermoplastic polymer and an intermediately stiff interphase material. The mechanical properties of the three interphases are presented in Table III.

The transverse stress and strain to failure for different interphase thicknesses are presented in Figures 12 and 13. The results show positive effects from thin rubbery interphases. The strain at failure initiation is increased by 100 to 300 percent when compared to composites with thermoplastic or intermediate modulus interphases. Also an increase in stress at failure initiation is observed. However, for an interphase thickness of 10 percent of the fiber radius, the stress drops to the same level as those for the stiffer interphases, see Figure 12. The high failure initiation stresses of rubber interphase (thin interphases) GF/EP composites are due the low modulus and high Poisson's ratio of the interphase. The predicted increase in strength by use of very thin rubbery interphases is supported by experimental data from Tryson and Kardos\textsuperscript{18}. They presented data for thin low modulus ductile interphases in a GF/EP composite. The increase in transverse strength was 67% as compared with the same material without interphase.

The results of the analysis of a rubber interphase composite are of special interest. Not only are the transverse failure initiation stresses and strains high, a change in failure mode is also observed as the interphase thickness is altered. For the thinnest interphase (1% of the fiber radius) the composite fails by cavity-initiation at the fiber poles. As the interphase thickness increases,
the position of the maximum dilatational energy density moves along the interphase/matrix interface and instead failure initiates by yielding at the fiber equators. As the interphase becomes sufficiently thick, the maxima of the dilatational energy density and the effective von Mises stress are both located at the fiber equators. For both criteria, failure in a composite with a relative interphase thickness of 10% is predicted to initiate at the fiber equators. However, cavitation-induced brittle failure may be suppressed by the high distortional stresses and failure may occur at a lower transverse stress than predicted by the von Mises criterion. This is because of the influence of hydrostatic pressure on the yielding of glassy polymers. Furthermore, high dilatational energy densities are observed in the rubber interphase. Triaxial tensile test data by Gent and Lindley\textsuperscript{9} show natural rubber to fail at dilatational energy densities of 0.3 MPa. The calculated dilatational energy densities in the rubber interphase are as high as 1.1 MPa for a relative interphase thickness of 5%. This suggests cavitation initiation in the rubber interphase to precede any failure initiation in the matrix. Such interphase initiated cracks may arrest in the rubber. In composites with thermoplastic and intermediate modulus interphases, failure initiates at the fiber poles due to high dilatational energy densities. For each interphase material and thickness, the maximum radial stress is located at the fiber poles.

4. CONCLUSIONS

A parametric study of fiber and interphase properties and interphase thickness on failure initiation in matrix of transversely loaded composites has been made. The study shows that fiber modulus has a significant effect on the stress and strain to composite failure caused by cavitation-induced brittle failure of the matrix. A thin interphase of a rubbery material provides improvement in transverse failure properties. The mode of matrix failure and the corresponding location around fibers are also influenced by the fiber and interphase properties.
REFERENCES


Table I. Mechanical properties of epoxy (DGEBA/DETA) and glass fiber. $U_v^{\text{crit}}$ is the critical dilatational energy density.

<table>
<thead>
<tr>
<th>Material</th>
<th>$E$ (GPa)</th>
<th>$\nu$</th>
<th>$\alpha$ $(10^{-6}/^\circ\text{C})$</th>
<th>$U_v^{\text{crit}}$ (MPa)</th>
<th>$\sigma_y$ (MPa)</th>
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</thead>
<tbody>
<tr>
<td>DGEBA/DETA</td>
<td>2.07</td>
<td>0.345</td>
<td>66</td>
<td>0.2</td>
<td>83</td>
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<tr>
<td>Glass fiber</td>
<td>72</td>
<td>0.2</td>
<td>5</td>
<td>-</td>
<td>-</td>
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</table>

Table II. Mechanical properties of Type I and Type II carbon fibers$^{4,6}$.

<table>
<thead>
<tr>
<th>Mechanical property</th>
<th>Type I</th>
<th>Type II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Long. modulus $(E_L)$</td>
<td>390 (GPa)</td>
<td>250 (GPa)</td>
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<tr>
<td>Transv. modulus $(E_T)$</td>
<td>12 (GPa)</td>
<td>20 (GPa)</td>
</tr>
<tr>
<td>Long. therm. exp. coeff. $(\alpha_L)$</td>
<td>-1 $(10^{-6}/^\circ\text{C})$</td>
<td>-0.3 $(10^{-6}/^\circ\text{C})$</td>
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<tr>
<td>Transv. therm. exp. coeff. $(\alpha_T)$</td>
<td>10 $(10^{-6}/^\circ\text{C})$</td>
<td>10 $(10^{-6}/^\circ\text{C})$</td>
</tr>
<tr>
<td>Minor Poisson’s ratio $(\nu_{TL})$</td>
<td>0.013</td>
<td>0.013</td>
</tr>
<tr>
<td>In-plane Poisson’s ratio $(\nu_{TT})$</td>
<td>0.25</td>
<td>0.25</td>
</tr>
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</table>
### Table III. Mechanical properties of three interphases.

<table>
<thead>
<tr>
<th>Material</th>
<th>Young's modulus $E$ (GPa)</th>
<th>Poisson's ratio $v$</th>
<th>Therm. exp. coeff. $\alpha$ ($10^{-6}/°C$)</th>
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</thead>
<tbody>
<tr>
<td>Rubber</td>
<td>0.004</td>
<td>0.4998</td>
<td>66</td>
</tr>
<tr>
<td>Thermoplastic</td>
<td>1</td>
<td>0.4</td>
<td>66</td>
</tr>
<tr>
<td>Intermediate modulus</td>
<td>34</td>
<td>0.2</td>
<td>5</td>
</tr>
</tbody>
</table>
Figure captions

Figure 1. FEM-mesh of a periodic element a) in a square fiber array, and b) a square fiber array with an interphase of a relative thickness (t/R) of 10%. Fiber volume fraction, V_f=0.5.

Figure 2. Transverse stress at failure initiation as a function of fiber modulus in a two phase composite with epoxy matrix and V_f=0.5.

Figure 3. Transverse strain at failure initiation as a function of fiber modulus in a two phase composite with epoxy matrix and V_f=0.5.

Figure 4. Transverse stress at failure initiation as a function of fiber volume fraction for epoxy composites with different fibers.

Figure 5. Transverse strain to failure initiation as a function of fiber volume fraction for epoxy composites with different fibers.

Figure 6. Transverse modulus as a function of fiber volume fraction for epoxy composites with different fibers.

Figure 7. Transverse stress at failure initiation for three interphase stiffnesses (E_i/E_m) as a function of relative interphase thickness (t/R) in a three phase composite based on GF/EP of V_f=0.5.

Figure 8. Transverse strain at failure initiation for three interphase stiffnesses (E_i/E_m) as a function of relative interphase thickness (t/R) in a three phase composite based on GF/EP of V_f=0.5.

Figure 9. Transverse strain to failure initiation for three interphase Poisson’s ratios (v_i/v_m) as a function of relative interphase thickness (t/R) in a three phase composite based on GF/EP of V_f=0.5.

Figure 10. Transverse composite stiffness for three interphase Poisson’s ratios (v_i/v_m) as a function of relative interphase thickness (t/R) in a three phase composite based on GF/EP of V_f=0.5.

Figure 11. Transverse stress at failure initiation for three interphase thermal expansion coefficients (α_i/α_m) as a function of relative interphase thickness (t/R) in a three phase composite based on GF/EP of V_f=0.5.

Figure 12. Transverse stress at failure initiation for rubbery, thermoplastic, and intermediate modulus interphases in GF/EP of V_f=0.5.

Figure 13. Transverse strain at failure initiation for rubbery, thermoplastic, and intermediate modulus interphases in GF/EP of V_f=0.5.
Figure 1. FEM-mesh of a periodic element a) in a square fiber array, and b) a square fiber array with an interphase of a relative thickness ($t/R$) of 10%. Fiber volume fraction, $V_f=0.5$. 
Figure 2. Transverse stress at failure initiation as a function of fiber modulus in a two phase composite with epoxy matrix and $V_f=0.5$. 
Figure 3. Transverse strain at failure initiation as a function of fiber modulus in a two phase composite with epoxy matrix and $V_f=0.5$. 

\begin{figure}
\centering
\includegraphics[width=0.8\textwidth]{figure3.png}
\caption{Transverse strain at failure initiation as a function of fiber modulus in a two phase composite with epoxy matrix and $V_f=0.5$.}
\end{figure}
Figure 4. Transverse stress at failure initiation as a function of fiber volume fraction for epoxy composites with different fibers.
Figure 5. Transverse strain to failure initiation as a function of fiber volume fraction for epoxy composites with different fibers.
Figure 6. Transverse modulus as a function of fiber volume fraction for epoxy composites with different fibers.
Figure 7. Transverse stress at failure initiation for three interphase stiffnesses \((E_i/E_m)\) as a function of relative interphase thickness \((t/R)\) in a three phase composite based on GF/EP of \(V_f=0.5\).
Figure 8. Transverse strain at failure initiation for three interphase stiffnesses ($E_i/E_m$) as a function of relative interphase thickness ($t/R$) in a three phase composite based on GF/EP of $V_f=0.5$. 
Figure 9. Transverse strain to failure initiation for three interphase Poisson's ratios ($v_i/v_m$) as a function of relative interphase thickness ($t/R$) in a three phase composite based on GF/EP of $V_f=0.5$. 
Figure 10. Transverse composite stiffness for three interphase Poisson’s ratio ($v_i/v_m$) as a function of relative interphase thickness ($t/R$) in a three phase composite based on GF/EP of $V_f=0.5$. 
Figure 11. Transverse stress at failure initiation for three interphase thermal expansion coefficients ($\alpha_i/\alpha_m$) as a function of relative interphase thickness ($t/R$) in a three phase composite based on GF/EP of $V_f=0.5$. 
Figure 12. Transverse stress at failure initiation for rubbery, thermoplastic, and intermediate modulus interphases in GF/EP of $V_f=0.5$. 
Figure 13. Transverse strain at failure initiation for rubbery, thermoplastic, and intermediate modulus interphases in GF/EP of Vf=0.5.