QUANTIFICATION OF THE CRACK AREAS OF STABLE AND UNSTABLE CRACK PROPAGATION DURING SINGLE-FIBER PUSH-OUT TESTS PERFORMED ON CERAMIC MATRIX COMPOSITE SAMPLES

Wolfgang M. Mueller¹, Judith Moosburger-Will¹, Michael Greisel¹ and Siegfried Horn¹

¹University of Augsburg, Institute of Physics, Experimental Physics II
86135 Augsburg, Germany
Email: wolfgang.mueller@physik.uni-augsburg.de,
web page: http://www.physik.uni-augsburg.de/exp2/

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ABSTRACT

It is well recognized that the mechanical properties of fiber-reinforced composites are closely related to the fiber-matrix interfacial properties. The interfacial fracture toughness is considered to be one of the most relevant quantities to characterize the material behavior at the fiber-matrix interface of a composite sample under mechanical load. Recently, a modification of the single-fiber push-out test was published by our group, enabling a quantification of the energy dissipated during stable crack propagation. The corresponding crack area, however, is simply estimated by the total fiber surface area of the pushed fiber in the established push-out evaluation methods described in literature. Since the occurrence of both, stable and unstable crack propagation is expected, the existing approximations represent an upper limit of the actual relevant area of stable crack growth. In the presented work, a new approach to quantify the specific crack areas is shown, which is based on a quantification of the crack energy dissipated during crack growth as a function of the total sample thickness. The procedure is applied to Siliconcarbide (SiC)-fiber reinforced SiC-matrix composites, produced via chemical vapor infiltration technique.

The new approach allows a quantification of the crack areas of stable and unstable crack propagation, which are created during push-out testing of ceramic matrix composite samples, for the first time by way of experiment. The length of unstable crack growth turns out to be in good agreement with expected values from stress-based models in literature.

The experimental determination of the specific crack areas leads to a more precise characterization of the interfacial fracture toughness compared to an approximation of the stable crack length by the total sample thickness. Therefore, the quantification of the crack areas presented here may contribute to a more reliable prediction of macromechanical properties from micromechanical single-fiber push-out tests.

1 INTRODUCTION

Ceramic matrix composites (CMC) are an attractive class of materials in lightweight, structural applications, especially under harsh environments (high temperatures, high stress levels, corrosive atmospheres). However, for widespread applications of CMC, a damage-tolerant fracture behavior is essential. A damage-tolerant, quasi-ductile behavior can be achieved by the occurrence of energy-dissipative mechanism at the fiber-matrix interface under mechanical load. These well-known micromechanical phenomena comprise fiber-matrix debonding, crack deflection at the fiber-matrix interface and fiber pull-out [1-3].

For mechanical characterization of interfacial properties on a microscopic scale, the single-fiber push-out test introduced by Marshall [4, 5] in 1984 plays a key role [6-14]. For this test, a composite sample is thinned to an appropriate thickness (typically below 150µm) and mounted on a sample holder.
with a groove located below the fibers to be tested. During the test, an individual fiber is loaded by a diamond indenter tip with an increasing load resulting in fiber-matrix debonding and fiber push-out. The interfacial parameters are extracted from load-displacement curves recorded during the experiment.

For the characterization of the fracture behaviour, the interfacial fracture toughness is considered to be one of the most relevant quantities to be determined [15]. Recently, a modification of the single-fiber push-out test was published by our group [16], enabling a quantification of the energy dissipated by stable crack propagation during debonding of fiber and matrix. However, the corresponding crack area is usually simply estimated by the total cylindrical fiber surface area of the pushed fiber. This approach disregards that both, stable and unstable crack propagation are expected to be involved in fiber-matrix debonding [15].

In the present study, a new approach to quantify the relevant crack area is presented, resulting in a more precise quantification of the interfacial fracture toughness by single-fiber push-out tests.

2 EXPERIMENTAL

2.1 Material and Sample Preparation

The push-out investigations were performed on Siliconcarbide (SiC)-fiber reinforced SiC-matrix (SiC/SiC) composites with a pyrocarbon (PyC) fiber coating. The samples were produced via chemical vapor infiltration technique from two-dimensional, plain-woven SiC-fiber fabrics (Tyranno Grade S, Ube Industries, Ltd). The PyC fiber coating has a thickness of 75 nm. The samples investigated in the current study are based on the same SiC/SiC composite material as described in Ref. [16] and were provided by MT Aerospace AG.

For the present investigation, the SiC/SiC composite material was thinned to obtain four samples with different thicknesses by a three-stage thinning process, inducing minimal damage to the samples. The samples were cut from the basic material by a precision low speed saw (Isomet, Buehler), perpendicular to the fiber axis orientation. These slices were thinned from both sides by a lapping process (Precision Lapping and Polishing System PM5, Logitech Ltd.), generating plane-parallel surfaces. For the lapping process, boron carbide particles with a grain size of 9µm were used. In the final step, a colloidal silica sol (mean particle size 32nm) was applied for surface polishing on both sides.

The samples have a thickness of 68µm, 76µm, 94µm and 128µm. The thickness of the samples was determined independently by two methods: by height gauge and by optical microscopy. The deviation in measuring the thickness with both methods at several positions on each sample turned out to be less than 1µm. For the push-out tests, the samples were mounted on a sample holder by quartz wax with a groove located below the fibers to be tested.

2.2 Single-Fiber Push-out Test with Unloading-Reloading Cycle

Single-fiber push-out tests were performed using an Universal Nanomechanical Testing system (UNAT, Asmec GmbH) with a force measurement resolution of 0.01mN and normal indenter displacement resolution of 1nm. The lateral positioning accuracy of the indenter tip is 1µm. A SEM micrograph of the indenter tip used in this study is shown in Fig. 1.

The indenter tip has the shape of a truncated four-sided pyramid with an edge length of 4.0µm and a total included angle of 38.3° (Microstar Technologies).
Figure 1: Flat-end diamond indenter in geometry of a truncated, four-sided pyramid with a contact area of 4x4 µm².

The loading schedule was defined following the approach of an energy-based evaluation published recently by our group [16]. The experiment was performed in displacement-controlled mode. The loading schedule consists of four segments:

1. **initial loading**: constant loading rate of 50nm/s, up to an indenter displacement of 1.6µm
2. **unloading**: constant unloading rate of 100nm/s, back to zero indenter displacement
3. **reloading**: constant loading rate of 50nm/s, up to a final indenter displacement of 3.0µm
4. **final unloading**: constant unloading rate of 100nm/s, back to zero indenter displacement.

3 RESULTS AND DISCUSSION

3.1 Scanning Electron Microscopy

A SEM analysis was performed on the front and back surface of the samples subsequent to the push-out testing. On the front side of the sample (Fig. 2a), the fiber is displaced relative to the sample surface, resulting in a darkening of the fiber surface on the SEM image due to shadowing effects.

On the fiber surface, there is a plastic imprint from the indenter tip. No evidence of plastic deformation is found on SiC matrix. On the back side of the sample (Fig. 2b) the tested fibers are protruding above the sample surface, while no plastic deformation is found on fibers or matrix. A more detailed
microscopic analysis of that sample (including Atomic Force Microscopy) performed at different stages of the experiment is presented in Ref. [16].


The definition of the interfacial fracture toughness \( \langle G \rangle \) is based on the strain energy release rate \( G \). The strain energy release rate \( G \) is defined as the strain energy \( \partial U \) dissipated during an infinitesimal crack growth with a newly created crack area of \( \partial A \) [17]:

\[
G = -\frac{\partial U}{\partial A}
\]  

(1)

As the stable debonding process is dominated by interfacial properties and is much less affected by geometric effects than the unstable one, the characterization of the stable process is most relevant for the prediction of macroscopic failure behavior. Therefore, the interfacial fracture toughness is defined as the strain energy release rate \( G \) averaged over the area of stable crack growth \( A_{\text{crack, st}} \). For a push-out experiment, the interfacial fracture toughness \( \langle G \rangle \) is equal to the total energy dissipated in stable crack growth \( \Delta E_{\text{crack, st}} \) divided by the crack area \( A_{\text{crack, st}} \) created in this process [14, 16]:

\[
\langle G \rangle = -\langle \frac{\partial U}{\partial A} \rangle_{A_{\text{crack, st}}} = -\frac{\Delta E_{\text{crack, st}}}{A_{\text{crack, st}}}
\]  

(2)

The energy dissipated in stable crack propagation \( \Delta E_{\text{crack, st}} \) may be quantified according to the approach introduced in Ref. [16]. The main idea of this procedure is summarized by Fig. 3. In order to separate the different plastic and elastic energy contributions of the fiber debonding process during the push-out experiment, the loading schedule of the experiment is modified. Therefore, an unloading-reloading cycle is inserted into the schedule (Fig. 3a, red color). The start of the unloading-reloading cycle (Fig. 3a, point P) is located within that section of the experiment that is dominated by stable crack propagation at the fiber-matrix interface, reflected by an extended linear segment in the load-displacement curve [16].

![Figure 3: Schematic illustration of a typical load-displacement diagram with (a) unloading-reloading cycle at point P and (b) with unloading-reloading cycle extrapolated to the peak load position.](image)

In Fig. 3a, the gray shaded area located underneath the unloading segment, corresponds to the energy \( E_{\text{elast}} \) stored elastically in the sample at point P. The red shaded area which is enclosed by the unloading-reloading cycle corresponds to the work of friction \( E_{\text{frict}} \) which is dissipated by the movement of the debonded part of the fiber relative to the matrix. It can be shown that the green shaded area located between the initial loading and the reloading curve corresponds to the energy dissipated in stable crack
growth up to point P [16]. For a quantification of the total energy dissipated in stable crack growth \( \Delta E_{\text{crack,st}} \) during the fiber debonding, the unloading-reloading cycle measured at point P (red color) has to be shifted to the peak load position by extrapolation (blue color, Fig. 3b). In Fig. 3b, the green shaded area corresponds to the total energy \( \Delta E_{\text{crack,st}} \) dissipated in stable crack growth.

3.3 Model for Stable and Unstable Crack Propagation

According to considerations of Kerans and Parthasarathy [15], both stable and unstable crack propagation are expected to contribute to fiber-matrix debonding during single-fiber push-out testing. However, the area of unstable crack propagation \( A_{\text{crack,unst}} \) has not been quantified by microscopic methods to date and, therefore, this contribution was neglected in the evaluation of single-fiber push-out tests so far.

In the current study, a new model is introduced accounting for both stable and unstable crack propagation. Fig. 4 represents a schematic illustration of that model assuming cylindrical symmetry with respect to the fiber axis.

![Figure 4: Schematic illustration of the lengths of stable and unstable crack propagation assuming cylindrical symmetry with respect to the fiber axis.](image)

After crack initiation at the top surface of the sample, the fiber-matrix debonding is dominated by stable crack growth, resulting in a gradual progression of the crack in an early stage of the experiment. With increasing indenter displacement, there is an increasing amount of energy stored elastically in the fiber. At the moment when the energy stored elastically exceeds the energy required to debond the unaffected part of the fiber-matrix interface, the crack growth turns from stable to unstable propagation, which almost instantly results in complete fiber-matrix debonding.

According to that model, the total cylindrical fiber surface (fiber radius \( r_f \), fiber length \( L \)) is equal to the sum of stable and unstable crack area.

\[
A_{\text{total}} = 2\pi r_f L = A_{\text{crack,st}} + A_{\text{crack,unst}}
\]

Due to the cylindrical symmetry of the configuration, the crack areas are cylindrical shaped surfaces with

\[
A_{\text{crack,st}} = 2\pi r_f L_{st}
\]

and

\[
A_{\text{crack,unst}} = 2\pi r_f L_{unst}
\]

Here, \( L_{st} \) denotes the length of stable crack propagation and \( L_{unst} \) the length of unstable crack propagation.
Together with equations (3), (4) and (5), equation (2) modifies to

$$\langle G \rangle = - \frac{\Delta E_{\text{crack, st}}}{A_{\text{crack, st}}} = - \frac{\Delta E_{\text{crack, st}}}{2\pi f (L - L_{\text{unst}})}$$

(6)

Hence, there is a linear relation expected between the sample thickness $L$ and the energy dissipated in stable crack growth $\Delta E_{\text{crack, st}}$, assuming $L_{\text{unst}}$ to be independent of sample thickness:

$$- \frac{\Delta E_{\text{crack, st}}}{2\pi f} = \langle G \rangle (L - L_{\text{unst}})$$

(7)

### 3.4 Evaluation of Crack Area and Interfacial Fracture Toughness

Based on equation (7) a quantification of the length of unstable crack growth and the interfacial fracture toughness can be performed. Therefore, single-fiber push-out tests on several push-out samples of the same sample type thinned to different sample thicknesses are carried out, and individual crack energies $\Delta E_{\text{crack, st}}$ are evaluated for each sample thickness.

In the present study, a minimum of 20 single-fiber push-out tests has been performed on each sample. In Fig. 5, $\Delta E_{\text{crack, st}}$ (normalized to the individual fiber circumference) is plotted for the four push-out samples having a thickness of 68$\mu$m, 76$\mu$m, 94$\mu$m and 128$\mu$m, respectively.

![Figure 5: Diagram of the total energy dissipated by stable crack growth during single-fiber push-out testing versus the sample thickness. The measured data are fitted by a linear regression according to equation (7).](image)

The standard deviation resulting from the evaluation of push-out tests on individual fibers is indicated by error bars. The data points are approximated by a linear regression using the functional relationship of equation (7). The model presented in section 3.3 is confirmed by the good agreement between the data points and the linear regression.

The linear regression yields an interfacial fracture toughness of 85±12 J/m² and the length of unstable crack growth to be 51$\mu$m. The length of unstable crack growth turns out to be in good agreement with the consideration of Kerans and Parthasarathy [15] which expected a length of several fiber diameters (average fiber diameter of Tyranno Grade S: 11$\mu$m). The large percentage of unstable crack area in the total crack area of 40 – 75% for the samples investigated underlines that a consideration of the specific crack areas is of major importance for a reliable quantification of interfacial properties by single-fiber push-out tests.
4 CONCLUSIONS

In summary, a new approach to quantify the stable and unstable crack areas was shown, based on a quantification of the crack energy dissipated during crack growth as a function of the total sample thickness. In this study, the procedure was applied to Silicon carbide (SiC)-fiber reinforced SiC-matrix composites, produced via chemical vapor infiltration technique.

The new approach allows a quantification of stable and unstable crack areas, which are created during push-out testing of ceramic matrix composite samples, by way of experiment. The length of unstable crack growth turns out to be in good agreement with expected values from stress-based models in literature and are in the range of 40 – 75% of the total sample thickness for the samples investigated.

The experimental determination of the specific crack areas leads to a more precise characterization of the interfacial fracture toughness compared to an approximation of the stable crack length by the total sample thickness. Therefore, the quantification of the crack areas presented here may contribute to a more reliable prediction of macromechanical properties from micromechanical single-fiber push-out tests.

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REFERENCES


