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Quantification of crack area in ceramic matrix composites at single-fiber push-out testing and influence of pyrocarbon fiber coating thickness on interfacial fracture toughness

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Abstract

For mechanical characterization of interfacial properties in fiber-reinforced ceramic matrix composites by single-fiber push-out tests, a determination of the relevant crack area is required. In established evaluation methods, the relevant crack area is approximated by the total cylindrical fiber surface of the pushed fiber. This concept disregards that stable crack propagation, which is relevant for prediction of macromechanical behavior, may occur on just part of the fiber-matrix interface area. In the present publication, a new approach to quantify the relevant crack area is presented, enabling a more reliable determination of the interfacial fracture toughness of ceramic matrix composites.

The new concept is applied to SiC-fiber reinforced SiC-matrix composites with pyrocarbon fiber coatings (SiC/PyC/SiC) produced via chemical vapor infiltration technique. The occurrence of stable and unstable crack growth, as predicted in literature, can be verified experimentally. A strong correlation between PyC fiber coating thickness and interfacial fracture toughness is found.

Keywords: Single-fiber push-out test; Crack area; Interfacial fracture toughness; Pyrocarbon fiber coating; Composites

1. Introduction

Ceramic matrix composites (CMC) have found their way as structural materials into a wide field of applications in harsh environments, involving high temperatures, high stress levels and corrosive atmospheres. The fields of application include the areas of aerospace, ground transportation, power generation and chemical industries. It is widely accepted that the mechanical properties of fiber-reinforced ceramic matrix composites are closely related to the fiber-matrix interfacial properties [1,2]. In particular, 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 [3]. In silicon carbide fiber-reinforced silicon carbide matrix composites (SiC/SiC), fiber-matrix interfacial properties are modified by fiber coatings which facilitate fiber-matrix debonding and thus micro-crack deflection at the fiber-matrix interface. Today, pyrolytic carbon (PyC) coatings applied by a chemical vapor deposition process are predominantly used in technical applications.

The single-fiber push-out test plays a major role in the micromechanical characterization of the interfacial properties since its introduction by Marshall [4,5] in 1984 [6–14]. Recently, a modification of the single-fiber push-out test was published by our group [15], 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. Since the occurrence of both, stable and unstable crack propagation is expected [3], the existing approximations represent an upper limit of the actual relevant area of stable crack growth.

In the present publication, a new approach to quantify the relevant crack area is presented. It is based on a quantification

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of the crack energy dissipated during crack growth as a function of the total sample thickness. The enhanced quantification of the crack area leads to a more reliable determination of the interfacial fracture toughness of the samples.

In previous push-out studies [8,10], it has been shown that the PyC coating thickness is a parameter to influence fiber-matrix interfacial properties, e.g. the interfacial shear strength. In the current publication, a correlation between the PyC fiber coating thickness and the interfacial fracture toughness of SiC/PyC/SiC composites is investigated for the first time. To this end, the new push-out evaluation method presented here is applied to SiC/PyC/SiC composites with PyC fiber coatings of three different thicknesses and the effect on the interfacial fracture toughness is discussed.

2. Experimental

2.1. Material and sample preparation

The samples investigated in the present study are SiC-fiber (Tyranno Grade S, Ube Industries, Ltd.) reinforced SiC-matrix composites with PyC fiber coatings. The samples were produced from plain-woven SiC-fiber fabrics via chemical vapor infiltration (CVI) method by a two-step procedure to deposit a PyC coating on the fibers and, subsequently, to form the SiC-matrix. In the present study, three types of CMC samples are investigated, which differ from each other in their fiber coating thickness. The coating thickness of the three samples was measured via scanning electron microscopy (SEM) on polished cross sections. The measurement was performed on 15 fibers per sample type with 2 positions per fiber. The coating thickness was taken as the distance of two parallel straight lines, which were aligned tangentially to the fiber-coating and coating-matrix interface, respectively. The thickness and standard deviation turned out to be (75 ± 5) nm, (40 ± 3) nm and (20 ± 5) nm, respectively (see Fig. 1). In the following sections, they are referred to as PyC-A, PyC-B and PyC-C. The samples were provided by MT Aerospace AG.

For the push-out tests, the samples need to be thinned to an appropriate thickness (typical below 150 μm) with plane-parallel surfaces orientated perpendicular to the fiber axis direction. This was done by a multi-stage thinning process, including a precision low speed cutting process (Isomet, Buehler), a lapping process (Precision Lapping and Polishing System PM5, Logitech Ltd.) with boron carbide particles (grain size 9 μm) and a final polishing step with a colloidal silica sol.

From each sample type (PyC-A, PyC-B, PyC-C) push-out samples of different thicknesses were prepared: Sample PyC-A was thinned to obtain four push-out samples having a thickness of 68 μm, 76 μm, 94 μm and 128 μm, respectively. From sample type PyC-B, push-out samples with a thickness of 60 μm and 85 μm were prepared. Sample PyC-C was thinned to obtain an 86 μm and an 102 μm thick push-out sample. The thickness of the samples has been measured by a height gauge and by optical microscopy. The deviations in thickness resulting from multiple measurements with both methods turned out to be less than 1 μm. An overview of the samples is given in Table 1. The samples cover a maximum range of feasible sample thicknesses for the push-out tests, being limited by the mechanical stability of the composite samples on the one hand and the fiber compressive strength on the other hand.

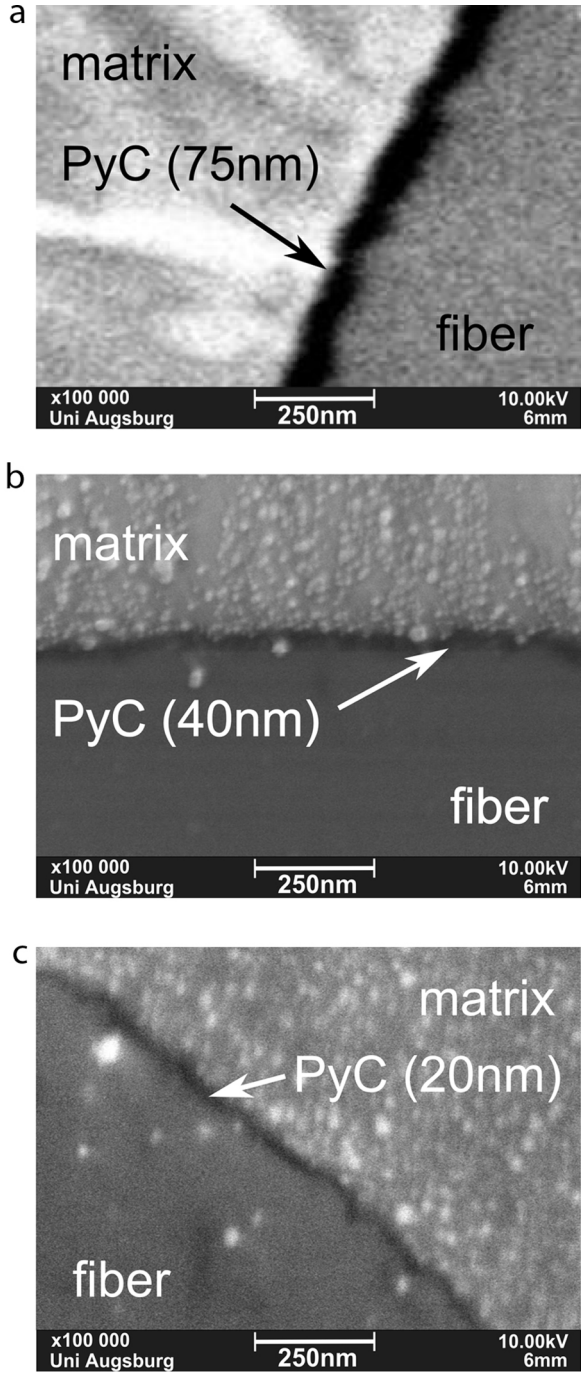

Fig. 1. SEM analysis of the PyC fiber coating on polished cross sections of the SiC/PyC/SiC samples (magnification 100,000×). (a) Sample PyC-A, (b) sample PyC-B and (c) sample PyC-C.

For carrying out the push-out tests, the specimens were then placed on a glass substrate with a groove of 60 μm in width, and fixed by quartz wax ensuring close and stiff contact to the substrate.