

# Investigation of subcritical crack growth using load relaxation tests on fiber bundles

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## Abstract

Many inorganic fibers are sensitive to subcritical crack growth activated by the environment. Failure occurs even though the applied stress is much smaller than the fracture stress. This mechanism has been extensively investigated on ceramics, glasses and glass fibers, essentially at room temperature, and has recently been shown in SiC-based fibers at high temperatures. The present paper proposes a very powerful approach to static fatigue. It is based on tests performed on multifilament tows under deformation-controlled conditions (the load relaxation technique). This technique, which has not been used previously owing to practical difficulties of deformation control during long-term tests, permits the application of identical constant stresses on all the fibers during a single test. Thus it provides a statistically significant rupture time database containing just as many data as there are fibers in the tows. Bundles of ~2000 E-glass filaments were used in the present paper. The samples were immersed in water during the static fatigue tests. Crack velocity–stress intensity factor diagrams for single filaments were derived from experimental stress–rupture time data. A closed form expression for statistical distributions of fiber lifetimes was established, and was found to compare fairly well with the experimental results, which assessed the approach and validated new findings.

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## 1. Introduction

Fibers are fundamental materials which are used alone (such as optical fibers) or as reinforcing constituents in composites (glass or ceramic fibers). In both cases, characteristics such as high strength and resistance to fatigue in severe environments are important prerequisites for the reliable performance of systems or composite materials.

Many papers have addressed the delayed failure of glass and ceramic materials (including bulk material and fibers) under stress in environments containing humidity or oxygen at room or high temperatures [1–16]. The mechanism

of slow crack growth in glass and oxide ceramics is well known. In glass, it results from reaction between a strained Si–O–Si bond and water at the crack tip [2,3,7]. More recently, the mechanism of slow crack growth in SiC-based fibers at temperatures <1200 °C has been described [12–15]. It was attributed to reaction between strained carbon grain boundaries and oxygen at the crack tip [15]. In all cases, the power function  $V = AK_I^n$  has been shown to provide a sound description of subcritical crack propagation, although the exact relationship between crack velocity  $V$  and stress intensity factor  $K_I$  is not known [7,17]. Many experimental data on crack propagation have been produced on bulk glass or ceramics, using fracture mechanics samples. This technique was quite difficult for fibers until now. Indirect methods based on static fatigue under constant load or dynamic fatigue tests, and stress–rupture time diagrams have been used to determine the slow crack growth constants  $n$  and  $A$ . For glass fibers, most of tests

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were performed on single filaments. For ceramic fibers at high temperature, static fatigue tests were carried out on fiber bundles under constant load [12–14]. Multifilament tows represent a fundamental entity in textile composites [18]. Testing bundles offers several advantages, and one of the most important is that several hundred fiber data can be determined in a single test [19–22]. Furthermore, uncertainty problems in the determination of the slow crack growth constant  $n$  that are associated with sample size are avoided [23]. To the authors' knowledge, static fatigue tests on tows of E-glass fibers have been reported only in Ref. [24]. In any case, it seems that all the static fatigue tests on fibers and tows were carried out under constant load. Although this is an easy technique from a practical point of view, it presents important limitations. Since the stress on fibers increases as the fibers fail, the extraction of  $n$  from stress–rupture time data is not straightforward, and the results may be biased.

A wide scatter in  $n$  values has been reported in the literature. For glass fibers, values between 12 and 40 were collected in Ref. [7]. From the previously mentioned static fatigue tests on tows of E-glass using a complex model [24],  $n = 15$  was extracted. In both Refs. [6,8], it was argued that there should be a relationship between  $n$  and applied stress. Then it was suggested that a variety of flaw sizes and shapes as well as a variation in residual stresses and inert strength result in similar variability in fatigue behavior [7].

The intent of the present paper was to propose an approach to slow crack growth and lifetime prediction based on static fatigue tests under constant deformation on multifilament tows. There is a big advantage in such a deformation-controlled loading condition, since all the fibers are subjected to the same constant stress. The well-known E-glass fiber was selected for this work. It may be regarded as a model material as it has been extensively investigated, so it seemed appropriate to develop the proposed approach to the lifetime of fibers. The tows contained  $\sim 2000$  filaments, so that a statistically consistent database was obtained. This testing method permitted the production of original results on the static fatigue of fibers and an assessment of lifetime prediction models. These results and the models can be applied to other types of fibers.

## 2. Experimental procedure

### 2.1. Material and specimens

Bundles of E-glass fibers contained  $\sim 2000$  filaments, each  $14 \pm 2 \mu\text{m}$  in diameter. The precise number of fibers was determined from the initial slope of the load–strain curve. The length of specimens exceeded slightly by  $< 4 \text{ mm}$  the gauge length defined by the extensometer ( $\sim 60 \text{ mm}$ ). The preparation procedure was detailed in a previous paper [19]. The main fiber characteristics are given in Table 1.

Table 1

Main reference characteristics of single filaments of E-glass fibers obtained in an inert environment, including the statistical parameters of the strength distribution.

$m$	$\sigma_o$ (MPa)	$v_o$ ( $\text{m}^3$ )	$E_f$ (GPa)	$K_{IC}$ ( $\text{MPa}\sqrt{\text{m}}$ )	$r$ ( $\mu\text{m}$ )
4.0	2.7	1	72	0.75	7

### 2.2. Testing procedure

The tensile tests on bundles were carried out at room temperature: under monotonous loading (displacement rate  $2 \mu\text{m s}^{-1}$ ) and an inert environment for determination of reference fast fracture data; and in water under constant deformation (static fatigue).

A servo-pneumatic testing machine, designed and built in-house, was used. It was equipped with a 500 N load cell. Displacement control of the cross head for long-term tests was ensured through pneumatic cushions. Deformations were measured using a contact extensometer (with a  $\pm 2.5 \text{ mm}$  elongation displacement transducer) which was attached to the specimen.

Two 4-mm-long thermo-retractable rings were threaded on the bundle to allow the extensometer to be clamped. They were located close to the grips in order to avoid possible bending introduced by the extensometer. The inner distance between the rings defined the gauge length ( $\sim 60 \text{ mm}$ ) [19,20]. Thus, strain measurement was direct and unpolluted by load train deformations. The samples were first loaded up to 5% of the ultimate load so that the extensometer could be positioned and adjusted. A view of the experiment with a fiber bundle, the mechanical extensometer and the water drop impregnation device is shown in Fig. 1.

During the tensile tests in an inert environment, lubricant oil was used to avoid friction between the fibers, which could cause fiber interactions, leading to premature fracture. A typical stress–strain curve is shown in Fig. 2.

During static fatigue tests, the strain was controlled directly by the mechanical extensometer. Specimens were first impregnated with water and then loaded at a displacement rate to test deformation of  $5 \mu\text{m s}^{-1}$ . Then the strain was kept constant. Typical curves of strain and load vs. time are shown in Fig. 3.

## 3. Model of slow crack growth in single filaments

Models of slow crack growth have been proposed for engineering ceramics [2,4,5] and for multifilament tows and single filaments of ceramic or glass fibers [12,14,17,24]. These models do not incorporate the features of static fatigue behavior of bundles under constant deformation.

The subcritical crack growth model is based on the simple power form of crack velocity vs. stress intensity factor, which is usually employed to describe the slow propagation of cracks caused by the environment under load in ceramics and glass materials [1–16]:

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