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The origin of the "crack tip" mode of failure in boron filaments

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A study has been made of the "crack tip" mode of failure in boron filaments. Filaments produced by a technique that ensures a high percentage of this type of flaw were subjected to tensile testing and fracture characterization. These filaments were split longitudinally and etched to expose possible fracture-causing defects. A high density of voids was detected within the bulk boron coinciding with the residual stress neutral axis, which is also the location of the tip of the radial crack in the filament. A model is proposed for this type of failure that is consistent with experimental observations and theoretical predictions.

1. Introduction

Fracture characteristics in boron filaments have been described by several investigators [1–4]. Failure typically initiates within the core (original substrate) or at the core–boron interface, at bulk inclusions (for multistage filament only), at surface defects (including crystalline spots), and at the tip of a crack (oriented longitudinally along the filament) which extends radially outward from the core to approximately 75% of the radius of the filament. Stress levels associated with each flaw type are reasonably consistent and decrease in magnitude in the order presented above.

Causes for each of the flaw sites have been described and related to a process and/or growth phenomenon with the exception of the so-called "crack tip" or radial crack type of failure. Identification of the cause of this type of flaw has been elusive, since to this point there has been no direct evidence as to its origin or nature presented in the literature. The purpose of this paper is to propose a mechanism for this fracture nucleation site and present evidence of its validity.

Vega-Boggio and Vingsbo [5] suggest that this type of flaw and hence the radial crack is related to the residual stress pattern and the pre-existence of interfacial proximate voids (which serve as nucleation sites for the radial crack). Using the Griffith criterion they demonstrated that propagation of the proximate void crack with residual stress as a driving force is possible and likely. The radial crack propagates at a progressively slower rate as the tangential residual stress is relaxed. Upon reaching the neutral axis between tensile and compressive residual stresses the crack stops. The authors conclude that the radial crack is formed after the completion of fibre growth.

Layden [6] also suggested that the radial crack is related to the internal stress distribution; however, no firm evidence for its role in the fracture mechanism was given. Krukonis [7] has presented evidence that the radial crack is not pre-existing and forms only after a force is applied to a fibre causing it to fracture.

Several investigators have defined the residual stress distribution in the filament [8–11]. Fig. 1 is a schematic diagram depicting the stress distribution which results from several factors, namely (i) elongation of the boron during deposition [8, 9]; (ii) tungsten core expansion during boriding; and (iii) thermal quenching in a mercury electrode at the exit of the reactor [2]. With the stresses oriented as they are (tensile near the core and compressive on the surface) there is a neutral zone of zero stress at some point between the dilatation and compressive regions. This neutral zone occurs at approximately 75% of the radius of the filament. The radial crack generally terminates at this point because it is inhibited from propagating into the compressive region.

The residual stresses, while present in all boron filaments produced by chemical vapour deposition (CVD), vary in magnitude with changes in deposition conditions [10]. In filaments produced under optimum conditions, the crack is shorter and the "crack tip" mode of failure is seldom observed. However, if one attempts to speed up deposition and/or to alter the natural temperature profile of the filament being produced then the radial crack is longer, the filament becomes easily splittable longitudinally, and the "crack tip" flaw becomes dominant and is active at extremely low stress levels. Hence in order to obtain some direct evidence relating to this type of failure mode, the present study concentrated specifically on filaments produced which had a high probability of failure by this mode.

2. Experimental procedure

Boron filaments were produced (at AVCO, Specialty Materials Division, Lowell, Massachusetts) under conditions known to generate a high frequency of "crack tip" failures. This was accomplished by altering the temperature profile assumed by the filament during production in order to produce filament at a faster rate. Fig. 2 is a schematic diagram comparing a normal and a "forced" temperature profile [7]. Filaments produced in this matter have higher internal stresses and

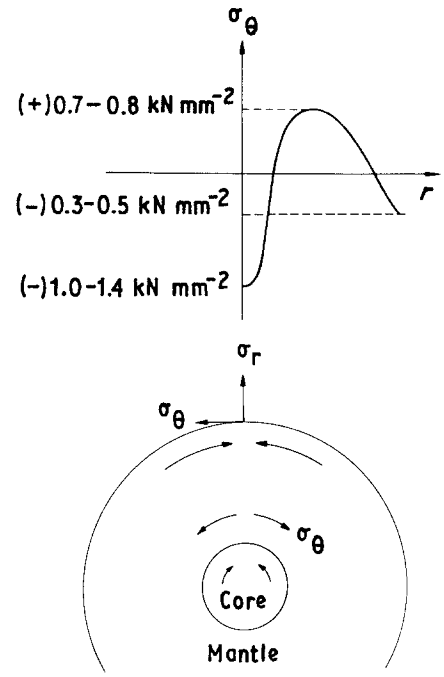

Figure 1 Residual stress distribution for forced temperature in boron filaments (after [5]).

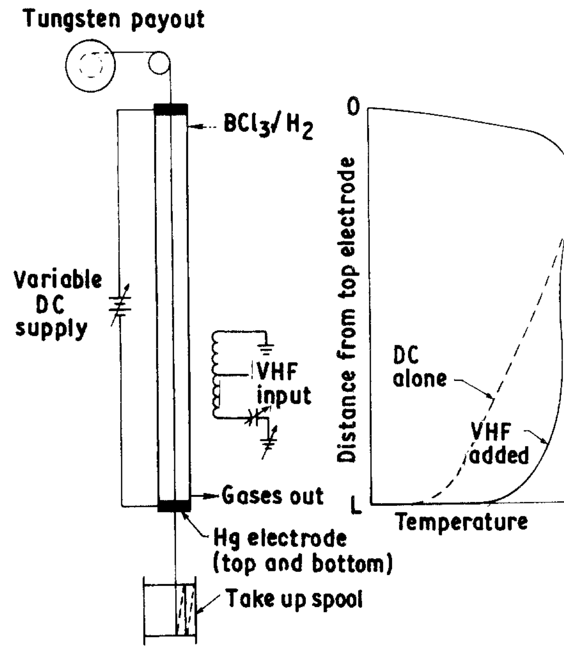

Figure 2 Schematic diagram of normal and forced temperature profiles in boron reactor (after [7]).

can be readily split in half longitudinally by applying a diametral force.

Sections of the split filaments were examined in a scanning electron microscope in the as-split condition and after oxidizing lightly in a gas flame (flame etching) or after etching in a fused salt of KOH : KNO_3. Both techniques generated similar surfaces. Etching times were on the order of a few seconds.

Tensile tests were performed on an Instron test machine using a gauge length of 2.5 cm and a crosshead speed of $2\,cm\,min^{-1}$. Broken ends of the tested filament were retrieved for examination of the fracture surfaces in the SEM.

3. Results

The tensile tests revealed that while sections of the filament had fracture stresses of $4.3\,GN\,m^{-2}$, most of the samples failed at approximately $1.7\,GN\,m^{-2}$ or less. Fig. 3a depicts a typical fracture surface for the low-strength filament showing the "crack tip" mode of failure. The high-strength filaments failed at the core–sheath interface. In the micrograph of the "crack tip" failure one can observe that the mirror area (denoting slow crack growth) surrounds the tip of the radial crack. Regions of this type normally surround the fracture nucleation site in brittle, glassy materials. Fig. 3b shows the extreme tip of a radial crack incorporating the fracture nucleation site. One can see that there is a step associated with the radial crack and at its tip is a differently appearing region (darker) approximately 5 μm in diameter. There is, however, no indication of an actual flaw in this region. The transverse slow crack has propagated at different levels from the tip of the crack inward and there are surface steps on the mirror area at the tip of the radial crack, oriented in the direction of the propagating transverse crack. Also obvious are some surface steps outside the dark fracture nucleation area [5].

At this point it is not clear whether the tip of the

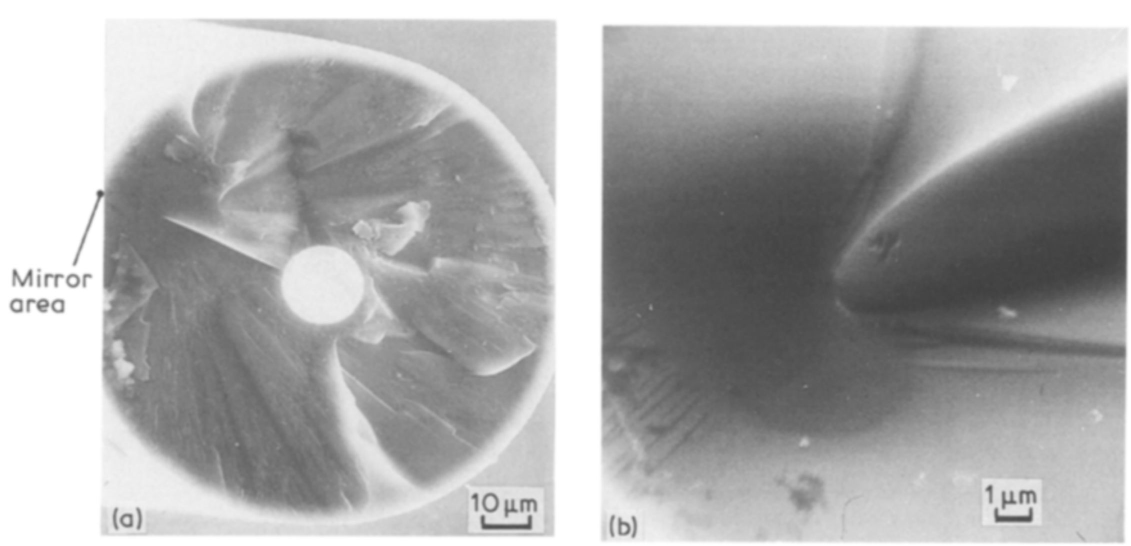

Figure 3 (a, b) Fracture surface showing "crack tip" mode of failure.

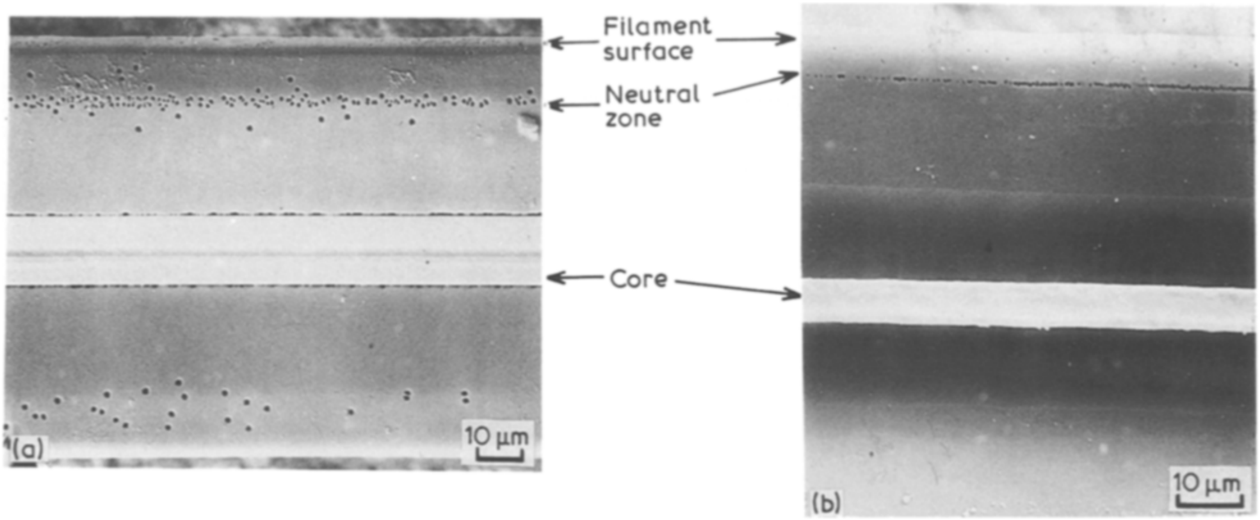

Figure 4 (a, b) Split filament showing voids, enhanced by etching, aligned along neutral stress zone.

radial crack is the actual fracture nucleation site or whether the fracture first nucleated at some flaw in the bulk boron and the radial crack initiated at a proximate void [6] and propagated to this region as a result of stored elastic energy in the highly strained filament (Krukonis' concept [7]). In an attempt to generate information, etched split filament was examined in the SEM. Figs 4a and b show two representative micrographs. Holes or pits that have been enhanced by the etching can be seen in the bulk boron. Whereas on occasion the holes were randomly distributed, in the majority they were aligned in a row parallel to the fibre axis and located in the region corresponding to the neutral axis between tensile and compressive residual stress regions in the filament. All split and etched filament exhibited a similar phenomenon.

3.1. Discussion

During chemical vapour deposition of boron the very simplified chemical equation $2BCl_3 + 3H_2 \rightarrow 2B + 6HCl$ describes the deposition process. In reality there are other intermediate species plus hydrogen that exist in the residual gases. If one examines the kinetics of the reaction there are two rate-limiting steps: the desorption of HCl from the surface and the diffusion of HCl through the reactant gases away from the filament. Hence there is a high probability that some of the gas-phase species are trapped in the deposited boron. Mass spectrometer measurements have shown that normally produced filament contains detectable and consistent HCl concentrations [12].

Filament produced at a faster deposition rate can be expected to trap a greater amount of the reactant gas products. If these trapped molecules were to coalesce into bubbles then they could be the source of the fracture-producing flaw.

Trapped gaseous species in CVD materials have been previously observed [13–15]. It was suggested in these studies that not only can bubbles grow by stress-induced diffusion of vacancies and precipitation of gas atoms but they can also link up to form stable crack nuclei [14]. A further theoretical study predicted that the bubbles, once formed in a material containing stress gradients, will be driven to regions of lower stress regardless of whether the solid is in tension or compression [16]. Each of these studies is directly pertinent to the present observations in boron filaments.

Boron filaments, in addition to containing trapped gases, have a high density of vacancies which are quite mobile as was demonstrated in interfacial void formation studies [17]. These factors combined with the severe internal stress distribution allow one to propose a model for the "crack tip" flaw which is consistent with the studies mentioned above and with present experimental evidence.

4. Fracture model

It is proposed that as the rate of deposition in CVD boron filament is increased due to forcing the temperature profile, greater amounts of reactant gases are trapped within the bulk boron. These gases migrate to voids formed by the coalescence of vacancies which subsequently grow to bubbles due to stress-induced mechanisms described by Stiegler *et al.* [14]. The bubbles then migrate to a region of lower stress in the manner suggested by Martin [16]. This region of lower stress coincides with the neutral zone between tensile and compressive residual stresses in the boron deposit. The bubbles align themselves along this region and link up to form stable crack nuclei [14]. Scanning electron micrographs shown in Figs 4a and b demonstrate that this does occur.

Upon loading, the radial crack is initiated at some stress level lower than the ultimate axial fracture stress and propagates to the neutral zone with internal stress as the driving force. Transverse crack formation is then initiated at the voids along the neutral axis (which coincides with the tip of the radial crack) and slowly grows with increasing applied stress during the tensile test. The fact that the transverse slow growth crack propagates at different levels on opposite sides of the crack (Fig. 3b) suggests that the radial crack is present before the transverse crack is initiated.

The foregoing offers a plausible explanation for the "crack tip" mode of failure in boron filaments that is consistent with experimental observations and theoretical predictions in this system as well as in others.

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