Simple and accurate fracture toughness testing methods for pyrolytic carbon/graphite composites used in heart-valve prostheses

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Abstract: The fracture toughness is a critical material property for the pyrolytic carbon materials used in mechanical heart-valve prostheses; however, making accurate toughness measurements has traditionally been problematic due to difficulties in fatigue precracking specimens. In this work, a simple, effective, and reliable precracking method is presented where a sharp precrack is "popped in" from a razor micronotch, which allows significant savings of time and materials relative to fatigue precracking methods. It is further shown that equivalent results may be obtained using razor micronotched specimens directly without precracking, provided the notch is sufficiently sharp. Indeed, mean toughness values of 1.46 ± 0.13 and 1.35 ± 0.09 MPa$\sqrt{m}$ were obtained for the pyrolytic carbon-coated graphite materials, using precracked and razor micronotched specimens, respectively. The difference between these mean values proved to be statistically insignificant, and these values are in general agreement with published fracture toughness results obtained using fatigue precracked specimens. © 2005 Wiley Periodicals, Inc. J Biomed Mater Res 74A: 461–464, 2005

Key words: pyrolytic carbon; fracture; fatigue; micronotch; toughness

INTRODUCTION

Pyrolytic carbon (PyC), either as a monolithic material or as a coating on a graphite substrate, has become the biomaterial of choice for most new mechanical heart valves since its first use in 1969.[1] The widespread usage of PyC is due to its high resistance to contact-induced blood clotting (thromboresistance) combined with superior strength, fatigue, and wear resistance when compared to other thromboresistant carbonaceous materials such as graphite and amorphous carbon.[2–5] Indeed, PyC typically demonstrates strengths some three to four times that of graphite and amorphous carbon.[2,3,5] In fatigue, early results demonstrated 10^7-cycle fatigue endurance limits that fell within the scatter band for the fracture strength, indicating greater fatigue resistance than graphite, which typically demonstrates similar endurance limits at only 45–65% of the strength.[4] Although PyC is susceptible to fatigue-crack growth, its fatigue thresholds (below which cracks are essentially dormant) are typically a large (>95%) fraction of the fracture toughness.[5–7] Such high fatigue-crack growth thresholds are consistent with the high endurance limits observed. Most recently, it has been shown that the mean endurance limit for realistic heart valve lifetimes of $\sim 10^9$ cycles is essentially at the mean single-cycle strength, which is well above the actual service stresses, putting the statistical probability of fatigue failure much better than one in a million based on Weibull analysis.[8] Finally, PyC demonstrates superior wear resistance over forms of carbon with lower hardness,[2] particularly for situations of PyC on PyC or on Ti, which occur in heart valves.[2,9]

Although PyC has many desirable properties, it is a brittle material with a low fracture toughness ranging typically from 0.9 to 1.1 MPa$\sqrt{m}$,[5,6,10] i.e., it is only ~50% tougher than window-pane glass. Composites made from PyC-coated graphite, however, exhibit slightly higher toughness values, ranging from ~1.3 to 1.6 MPa$\sqrt{m}$.[5,7] Because the fracture resistance is a critical property of these materials, there is a often a

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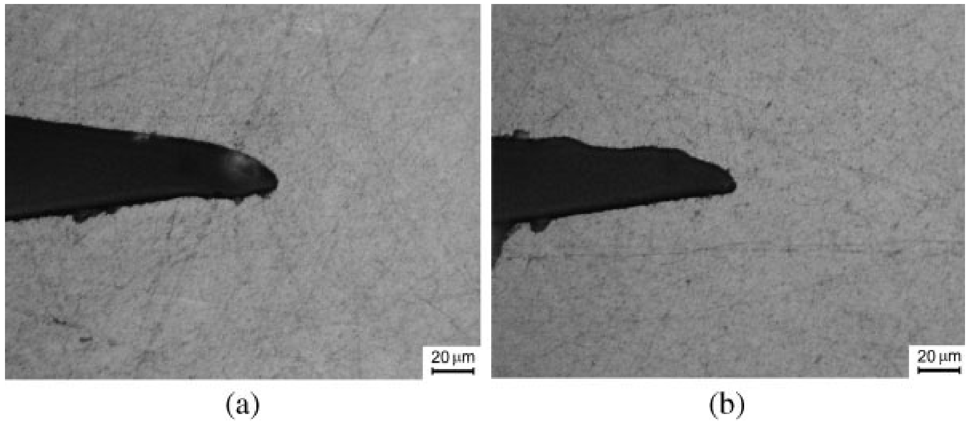

Figure 1. Optical micrographs showing examples of razor micronotches in PyC-coated graphite for (a) DC(T) sample 1 ($\rho \sim$ 5 µm) and (b) sample 6 ($\rho \sim$ 3 µm).

requirement for a quality-control measure to ensure adequate fracture toughness is achieved. Traditional fracture mechanics testing, such as is used for metallic materials,[11] requires a sharp precrack, which achieved by fatigue cycling. Such precracking is generally essential; indeed, tests on PyC[12] and PyC-coated graphite[5] using saw-cut notches (notch root radii unreported), in place of precracks, yielded erroneously high toughness values of 2.5 and 3.9 MPa$\sqrt{m}$, respectively, a common trend seen in most ductile[13] and brittle[14] materials. Due to its high fatigue resistance, however, fatigue precracking is a difficult and costly procedure for PyC and its composites.[5–7] Furthermore, simple indentation toughness testing methods[15] are unsuitable for the PyC-coated graphite composites used in heart valves since the indenter stress field is affected by the underlying graphite layer, giving large variations in apparent toughness with changing indenter load.[16] In light of this, there is clearly a need for simple test methods that allow for accurate fracture toughness measurements for the PyC and PyC/graphite materials used in heart-valve prostheses. Accordingly, in the present work, we present a simple yet effective "pop-in" precracking method that utilizes an initial sharp razor micronotch; we further demonstrate that statistically equivalent fracture toughness results may be obtained, even without precracking, simply by initiating fracture from the razor micronotches themselves.

MATERIALS AND METHODS

Disk-shaped compact-tension, DC(T), specimens of (silicon-free) pyrolytic carbon coated graphite (~0.4 mm graphite core thickness) were provided by Medical CV, Inc. (Inver Grove Heights, MN) with nominal dimensions, $W = 18.8$ mm, $B = 0.9$ mm, and $a_o = 5.5$ mm, where W, B, and a_o are, respectively, the specimen width, thickness, and initial notch depth (measured from the load line). Samples were prepared using the same production parameters as the orifice for mechanical heart-valve components and were subject to the same basic processing and finishing operations as the components of the heart valve. After machining, the DC(T) samples were inspected to ensure that no cracks were present along the saw cut. Razor micronotches were then placed at the end of the saw cut notches by repeatedly sliding a razor blade over the notch under a light normal load (~1 N) using a custom made rig in the presence of a 1-µm diamond slurry. Using this method, notch root radii of ρ ~3–5 µm were readily achieved, as shown in Figure 1.

One set of six specimens was tested with only micronotches, while a second set of six samples was precracked after micronotching and prior to fracture toughness testing. Precracking was performed not by fatigue cycling but by loading the samples manually in displacement control using an electro servo-hydraulic load frame (Instron 1350 load frame, Instron Corporation, Canton, MA, with an MTS 458 controller, MTS Systems Corporation, Eden Prairie, MN) until a nonlinearity in the load-displacement curve was detected, indicating that a crack had "popped in" from the razor-sharp notch. The sample was then removed from the testing machine, and examined using optical microscopy. This process was repeated until a distinct, through-thickness precrack extending ~1–3.5 mm from the notch was clearly visible in the optical microscope, as seen in Figure 2. Precracks were then measured prior to fracture toughness testing using an optical measuring microscope with 0.5-µm resolution (STM-UM5 Measuring Microscope, Olympus America Inc., Melville, NY).

All samples were then tested in general accordance with ASTM Standard E-399[11] for fracture toughness measurement. Specifically, the DC(T) specimens were loaded under load control using the same electro servo-hydraulic load frame at a loading rate of 0.44 N/sec until unstable fracture occurred. The critical stress intensity, K_c, for each sample was then computed from the peak load P and sample dimensions, B, W, and a, using the standard stress-intensity solutions for the DC(T) geometry, namely:[11]

$$K = \frac{P}{B\sqrt{W}} f\left(\frac{a}{W}\right), \qquad (1)$$

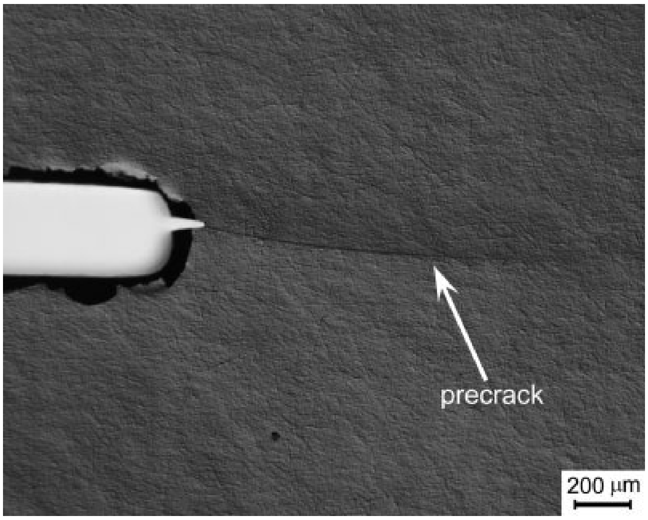

Figure 2. Optical micrograph showing a "pop-in" precrack in PyC-coated graphite extending from a razor micronotch in DC(T) sample 7.

where

$$f\left(\frac{a}{W}\right) = \frac{2 + \frac{a}{W}}{\left(1 - \frac{a}{W}\right)^{3/2}}\left[0.76 + 4.8\left(\frac{a}{W}\right) - 11.58\left(\frac{a}{W}\right)^2 + 11.43\left(\frac{a}{W}\right)^3 - 4.08\left(\frac{a}{W}\right)^4\right]. \quad (2)$$

RESULTS AND DISCUSSION

Fracture toughness results for the six tests using micronotched samples are listed in Table I, while the results for the precracked samples are listed in Table II. The mean K_c value for six micronotched specimens was 1.35 MPa$\sqrt{m}$ with a standard deviation of 0.09 MPa$\sqrt{m}$, while for the precracked experiments the mean K_c was 1.46 MPa$\sqrt{m}$ with a standard deviation of 0.13 MPa$\sqrt{m}$. Statistical analysis of the data in Tables I and II using an unpaired Student t test indicated that there was no statistical significance of the difference between the two mean values; the p value for the Student t test was 0.13. Thus, within random experimental scatter, the same results were achieved using either the micronotched or precracked testing methods.

TABLE I
Fracture Toughness Test Results for Micronotched Samples

Sample no.	K_c (MPa$\sqrt{m}$)
1	1.40
2	1.36
3	1.50
4	1.28
5	1.29
6	1.29
Mean ± Std. Dev.	1.35 ± 0.09

TABLE II
Fracture Toughness Test Results for "Pop-In" Precracked Samples

Sample no.	K_c (MPa$\sqrt{m}$)
7	1.56
8	1.57
9	1.37
10	1.37
11	1.58
12	1.29
Mean ± Std. Dev.	1.46 ± 0.13

Furthermore, the results presented here are in general agreement with previously published values for similar PyC-coated graphite composites using fatigue precracked samples, where mean fracture toughnesses of 1.3–1.6 MPa$\sqrt{m}$ were reported.[5,7] Note that the present values do not approach the erroneously high toughnesses (e.g., 3.9 MPa$\sqrt{m}$)[5] obtained using saw-cut notched samples. Thus, it appears that the only significant difference between the three types of toughness tests is the ease by which they can be performed. Testing using only the razor micronotch simply requires an extra machining step to prepare the specimen, which typically can be done in a matter of minutes with very little risk of inadvertent specimen breakage (unlike precracking by the current "pop-in" technique or by regular fatigue cycling). However, the sharpness of the notch is a concern since it is known that significantly blunter notches can give erroneous toughness values.[5,12] Notch sharpness may vary depending on the specific razor micronotching procedure; here a custom-made motor-driven rig was used to repeatedly slide the razor blade along the notch to obtain notch root radii of less than ~5 μm. This degree of sharpness proved to be adequate based on comparisons to results from precracked specimens, although it is not known what minimum notch radius is needed to get accurate results and whether this minimum value is the same for all PyC-based materials. Accordingly, initial comparisons to precracked fracture toughness data along with care in obtaining consistently sharp notches are necessary for obtaining accurate fracture toughness results using razor micronotched specimens.

In studies on this effect in metallic materials, the limiting notch root radius has been found to be comparable with some characteristic microstructural dimension associated with the local fracture mechanism, such as the grain size or inclusion spacing.[13] The successful use of micronotch fracture testing in brittle materials, however, most likely depends on several factors. One may suspect that during the machining process, microcracks may be introduced into the notch region, thus essentially providing a sharp crack analogue. If this is the case, a critical notch sharpness is essential to ensure that the stress field of the notch, which scales in size with the root radius, is not so large as to overwhelm the stress fields of any individual microcracks. Furthermore, while this method is easily extended to other brittle materials (e.g., as in ref.[17] for Si_3N_4), care should be exercised in the case of toughened ceramics that exhibit rising R-curve behavior since a single point on the R-curve will be assessed, which will be geometry and loading specific. Materials such as pyrolytic carbon, however, are ideal for such testing, as they tend to have fine-grained microstructures which are very brittle and exhibit minimal R-curve behavior.

If precracked fracture toughness tests are a requirement though, the presence of a sharp razor micronotch permits the "pop in" precracking method to be successful. Here the sharp micronotch allows crack initiation at relatively low loads, thereby preventing "runaway" cracking and potential catastrophic failure of specimens. The procedure can be performed in roughly 30 to 60 min, and in the present study was achieved without inadvertent specimen fracture. This is in sharp contrast to fatigue precracking methods, which for very brittle materials such as PyC can take several hours or days per sample, with a high risk of specimen loss.

Thus, a simple and effective precracking method has been presented, which may be used to verify fracture toughness results for samples that have only been razor micronotched, as well as provide a basis to determine the minimum notch radius necessary to get accurate results without precracking.

CONCLUSIONS

Based on an experimental study of measurement methods for obtaining the fracture toughness of pyrolytic carbon-coated graphite composites used for heart valve components, it is concluded that the razor micronotched compact-tension test provides an accurate and rapid procedure for assessing the fracture toughness of pyrocarbon materials, with the caveat that a critical notch sharpness must be maintained for all tests. Where a precracked fracture toughness test is required, the presence of the razor micronotch is further found to provide a rapid means of precracking, specifically by facilitating the formation of a "pop-in" crack under displacement-controlled loading. Tests on razor micronotched and "pop-in" precracked compact-tension samples gave fracture toughness values for the PyC-coated graphite that were not statistically different (K_c ~1.4–1.5 MPa$\sqrt{m}$), and that were in general agreement with previously reported K_c values for this material determined using fatigue precracked specimens.

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