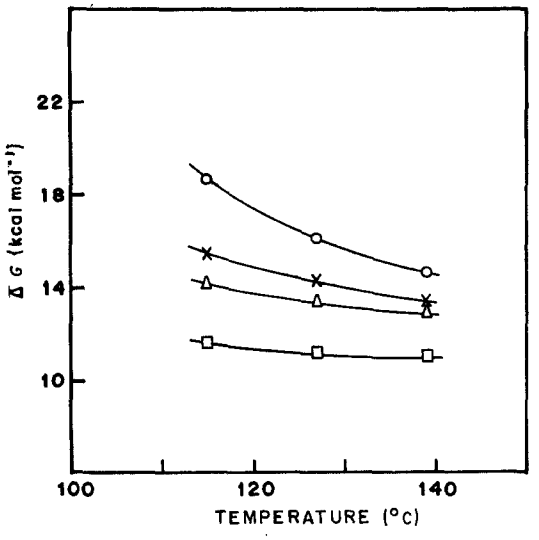

Figure 3 Variation of molar free energy of activation ΔG with temperature for PAN. (○) our experimental values, (×) data from [1], (△) data from [2] and (□) data from [15].

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On the use of small specimens in the measurement of the fracture toughness for brittle materials

The fracture toughness, K_{Ic}, has become a well-established parameter for the assessment of the fracture behaviour of brittle materials. Several methods of measuring this quantity have been advanced. The bend test, the double-cantilever beam test and the double torsion test are the best known of these methods. The bend test in 3-point or 4-point set-up is an especially popular method. The specimen length for this test ranges usually from 30 to 50 mm. Although this size is generally much smaller than the size necessary for other tests, it is nevertheless important that smaller specimens can be used. In order to see whether really small specimens give the same results as larger ones, some experiments using the 3-point bend method were carried out.

Large specimens (dimensions 3 mm × 9 mm × 45 mm) and small specimens (dimensions 1 mm × 3 mm × 15 mm) were machined of several brittle ceramics (see Table I). In each sample a notch

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TABLE I Comparison of fracture toughness data measured with large and small specimens

Materials description*	Large specimens				Small specimens				Atmosphere
	K_{Ic} (MPa $m^{1/2}$)	standard deviation†	N‡	v(μm sec^{-1})§	K_{Ic} (MPa $m^{1/2}$)	standard deviation†	N‡	v(μm sec^{-1})§	
Ni–Zn–ferrite (5.31, 6.1)	1.360	0.086	8	0.58	1.422	0.067	11	4.3	air, 50–60% r.h.**
Mn–Zn–ferrite (5.39, 18)	1.539	0.113	7	1.7	1.402	0.093	9	4.3	air, 50–60% r.h.
Mn–Zn–ferrite (5.40, 12)	1.523	0.127	7	1.7	1.495	0.143	10	4.3	air, 50–60% r.h.
Sr–hexaferrite (4.40, ~ 1)	1.796	0.064	4	1.7	1.710	0.163	10	0.58	water
OHAp¶ (2.32, 1.7)	0.599	0.034	3	1.7	0.655	0.051	6	1.7	dry N_2 gas
OHAp (2.47, 1.7)	0.636	0.076	8	1.7	0.642	0.137	5	1.7	dry N_2 gas
OHAp (2.62, 1.7)	0.668	0.112	3	1.7	0.814	0.118	7	1.7	dry N_2 gas
OHAp (2.85, 2.1)	0.991	0.103	10	1.7	0.888	0.091	8	1.7	dry N_2 gas
OHAp (2.97, 2.9)	0.978	0.083	9	1.7	0.928	0.059	8	1.7	dry N_2 gas
OHAp (3.07, 3.9)	1.074	0.091	6	1.7	1.010	0.079	8	1.7	see [1]

*The numbers in parentheses denote the density in g cm^{-3} and the grain size in μm respectively.
†The standard deviation is calculated according to $[\Sigma(x-\bar{x})^2/(n-1)]^{1/2}$ where x and $\bar{x}$ denote the individual and average fracture toughness values, respectively.
‡Number of specimens used.
§Cross-head speed of the testing machine.
¶$Ca_5(PO_4)_3OH$.
**r.h. = relative humidity.

of width about 100 μm and relative depth about 0.15 was sawn. Although deeper notches are not uncommon, this value is quite satisfactory since the value of the compliance factor in the calculation of the fracture toughness is relatively insensitive to the exact notch depth in this depth range. The span of the bending set-up was 36 mm for the large specimens and 12 mm for the small specimens. All dimension ratios as well as the calculation of the compliance factor were in accordance with [1]. Pre-cracking was performed with a Vickers hardness indentation (1 to 2 N load) just below the notch root on both sides of the specimen. Various values of cross-head speed of the testing machine* were used. The results are given in Table I.

Good agreement is observed between the results of the two series of measurements. For a more quantitative comparison, the two sets of data are plotted against each other; if no significant differences between the two sets exist, one would expect a plot showing a straight line through the origin. Since both data sets contain errors, a general linear least-squares fit $y = a + bx$ plot, making allowance for errors in the abscissa as well as in the ordinate quantities, would be appropriate. Here x and y denote the fracture toughness values for large and small specimens, respectively. A simple linear least-squares fit using equal weights, however, yields more conservative estimates for the standard deviations of the parameters a and b. Hence this procedure was used resulting in $a = 0.111$, with standard deviation of 0.071, and $b = 0.883$, with standard deviation of 0.060. At the 5% significance level the t-test [2] states that the slope b does not differ significantly from 1. Moreover, the quantity a does not differ significantly from zero at the same significance level. Hence, a preferable estimate [2] of b is made by a least-squares fit $y = bx$. This yields $b = 0.971$, with standard deviation of 0.022. The corresponding significance level is 22%. Hence, it is concluded that the small type of specimen can be used as safely for the measurement of the fracture toughness as the large one.

Two more remarks can be made. First, one may doubt the use of the normal distribution as used in the test. In fact, non-parametric tests among each sample pair and among the pooled sample pairs after normalization were also carried out. A comparison of the locations and variabilities in the results obtained by the two methods did not reveal any difference at the 5% significance level: they appear to have similar precisions and hence, the conclusion of the t-test remains. Second, the fractured area must be representative of the microstructure of the ceramic. Arbitrarily stating that at least 1000 grains should be present in the fracture surface to ensure that a representative area is examined, the grain size should not exceed about 30 μm when small specimens are used.

*Electomatic, Manufactured by Tinius Olsen.

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