The Korean Journal of Ceramics, **4** [4] 331 - 335 (1998)

R-Curve Behavior of Silicon Nitride at Elevated Temperatures

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(Received September 23, 1998)

R-curve, of three kinds of silicon nitride-based ceramics were measured, using single edge notched beam (SENB) method at room and at elevated temperatures, up to 1200°C. Stable fracture was seen on ceramic materials with SENB specimens if the machined notch is deep enough, even though the crack resistance did not increase with crack length. Hot pressed silicon nitride did not show the rising R-curve behavior at room temperature, but it showed some rising at 1000°C and above. Si_3N_4 reinforced with SiC whiskers showed no rising behavior at room and elevated temperatures, as it has smaller grain size, compare to the monolithic specimen. Gas pressure sintered silicon nitride had very large and elongated grains, and it showed rising R-curve even at room temperature. However, it showed some creep behavior at 1200°C and the calculated R-curve on this condition did not show a good result. We cannot apply this technique on this condition for obtaining the R-curve.

Key words : R-curve, Fracture toughness, Silicon nitride, Notched beam, Stable fracture

I. Introduction

It is important to investigate the dependence of the critical stress intensity factor on the crack extension, which is called as R-curve behavior, for understanding the fracture behavior of toughened engineering ceramics.[1-3] However, we have no established testing method of R-curve behavior of ceramics, especially for high temperature testing. Single edge notched beam (SENB) method is one of the test methods for measuring the fracture toughness of ceramics.[4,5] This method has a problem for the measurement in fracture toughness, as the measured value depends on notch width. However, this method is quite good for the analysis of stress intensity factor.[6,7] In usual SENB tests, the specimens break instantaneously. However, we found that the stable fracture occurred in SENB tests, if the machined notch is deep enough. This test method can be applied easily for high temperature test, so I tried to measure the R-curve behavior of three kinds of silicon nitride based ceramics, at elevated temperatures up to 1200°C.

II. Analysis

During the bending test, we can obtain the values of applied force and bending deflection of the specimen. The crack length can be calculated by the change of the compliance of the bending specimen. Then, the stress intensity factor and the crack length can be obtained from the values of the applied force and the deflection.[7,8]

The stress intensity factor on a straight-through notched four point bending bar is calculated by Equations 1 to 3,

$$K_I = \frac{P}{BW^{1/2}} \left(\frac{S_1 - S_2}{W} \right) Y_{(\alpha)} \tag{1}$$

$$Y_{(\alpha)} = \frac{3\Gamma_M \alpha^{1/2}}{2(1-\alpha)^{1/2}} \tag{2}$$

$$\Gamma_m = 1.9887 - 1.326\alpha - \frac{(3.49 - 0.68\alpha + 1.35\alpha^2)\,\alpha(1-\alpha)}{(1+\alpha)^2} \tag{3}$$

where K_I is the stress intensity factor and P is the applied force. The notations for the shape of the bending bar are shown in Fig. 1. Equation 3 is applicable for the range of $0<\alpha<1$.

The change of the compliance on the bending bar caused with the crack extension is calculated from Equations 4 and 5.

$$C = \frac{1}{E'B} \left(\frac{S_1 - S_2}{W} \right)^2 \int_0^{\alpha} 2Y_{(x)}^2\, dx + C_0 \tag{4}$$

$$C_0 = \frac{1}{E'B} \left(\frac{S_1 - S_2}{W} \right)^2 \left(\frac{S_1 + 2S_2}{4W} \right) \tag{5}$$

where $E' = E/(1-\nu^2)$ for plane strain (E: Young's modulus, n: Poisson's ratio).

These equations suggest that the deflection of the bending bar with the notch can be calculated, if the parameters of the specimen, such as stress intensity factor at fracture, Young's modulus, notch depth, and the shape of the specimen are fixed. Fig. 2 shows the example of the relation between the load and deflection of the notched bending bar, by applying the parameters of typical silicon nitride (K_{IC}=5.0 MPa $m^{1/2}$, E=310 GPa) on 4-point bending of 30-10 mm spans. The height and

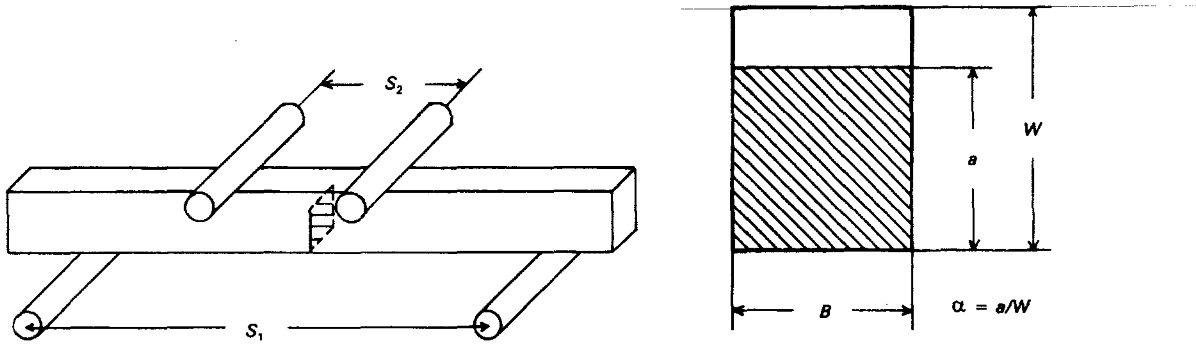

Fig. 1. The notations for the bending bar and the straight-through notch.

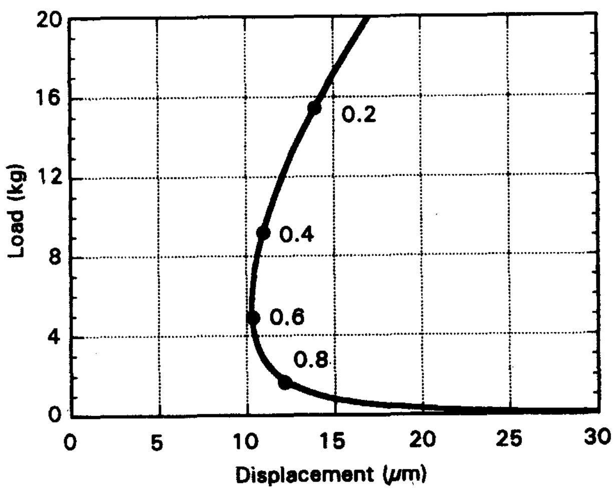

Fig. 2. Load-deflection curve obtained from calculation using the parameters of typical silicon nitride, on four-point bending. The numbers on the figure are dimensionless notch depth, α (=a/W).

width of the specimen were 4 and 3 mm, respectively.

This figure indicates that, if the dimensionless notch depth, a/W, is 0.2, about 14 μm deflection of the bending bar is required to start cracking. The deflection does not decrease in the constant cross-head speed test. Then, once the crack extends, the stress intensity factor increase with crack extension, in this case. On the other hand, if the test is started from a/W of 0.8, fracture starts at 12 μm of deflection, and stress intensity factor decreases with crack extension. That means, even if the specimen is brittle ceramics and it has no rising R-curve behavior, we can obtain the stable fracture if the machined straight-through notch is deep enough.

III. Experiments

Three kinds of silicon nitride based ceramics were tested; (1) hot pressed silicon nitride (HPSN), (2) hot pressed silicon nitride, containing 20 wt% of SiC whiskers (SCwSN), (3) gas pressure sintered silicon nitride (GPSN). All specimens contain yttria and alumina, as sintering additives. Fig. 3 shows the photographs of the fracture surface of the specimens by scanning electron microscope.

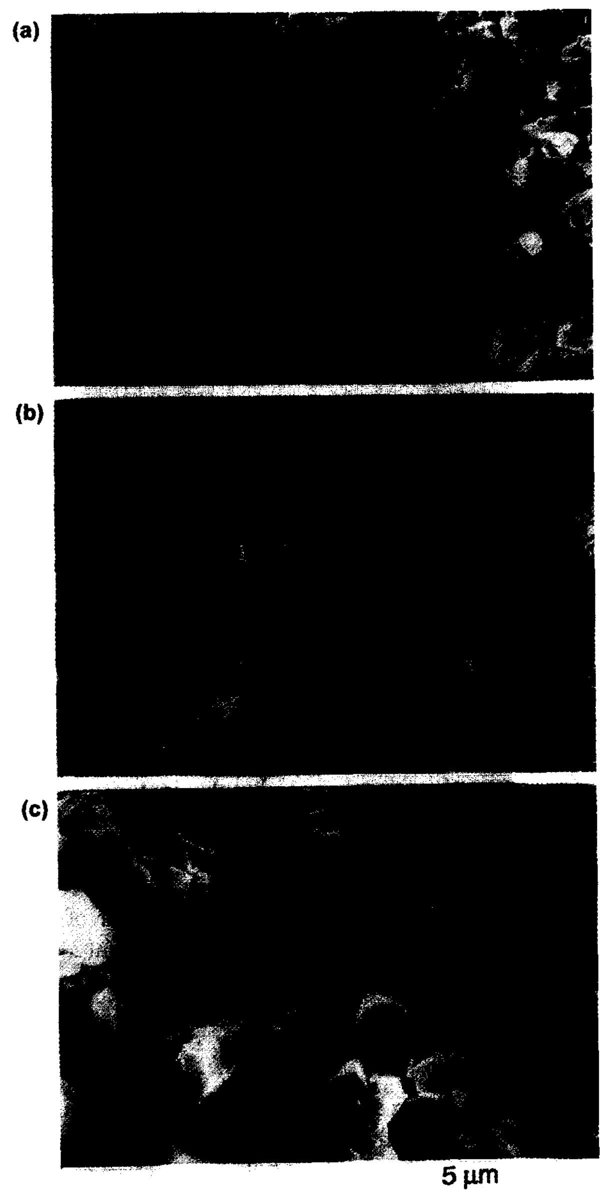

Fig. 3. Fracture surface of the tested three kinds of silicon nitride based ceramics. (a) HPSN, (b) SCwSN and (c) GPSN.

GPSN has quite coarse grain size. HPSN has intermediate grain size of three samples. SCwSN has the smallest grain size, and it is difficult to distinguish the silicon carbide whiskers from silicon nitride grains, in the fracture surface observation.

Test pieces for the R-curve were bending bars of 4×3 mm cross section. The depth of the machined notch was 3.5 mm for HPSN, 3.6 mm for SCwSN and 3.0 mm for

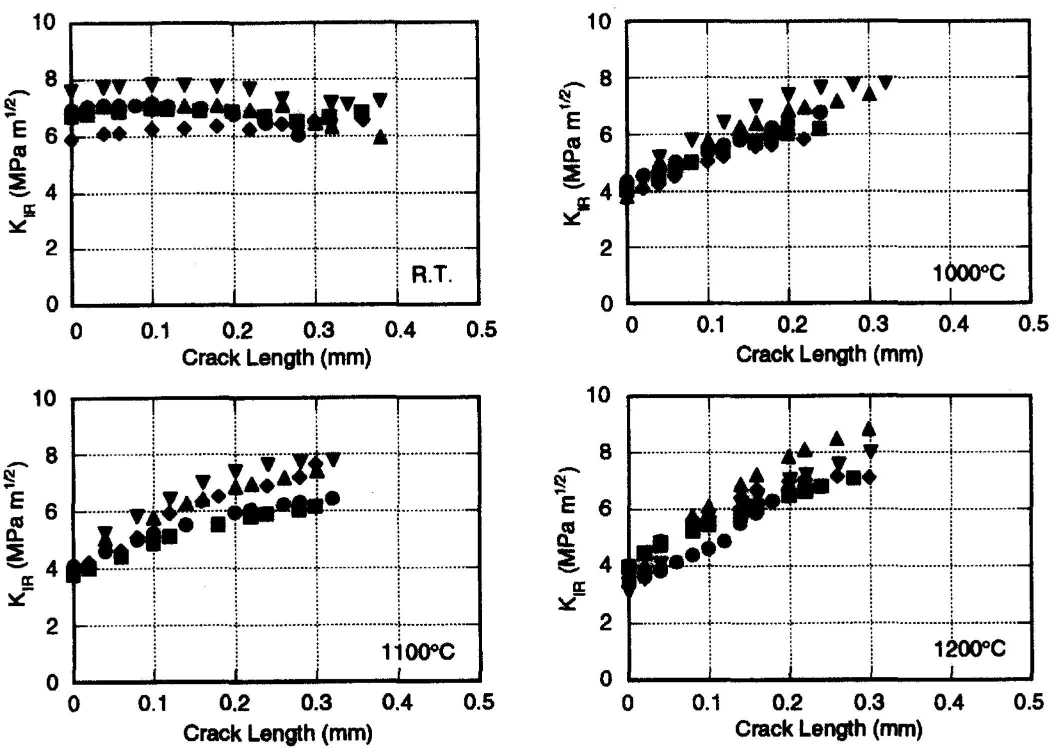

Fig. 4. R-curve behavior of hot-pressed silicon nitride (HPSN) at room temperature, 1000°C, 1100°C and 1200°C. Rising of the R-curve can be seen only at elevated temperatures.

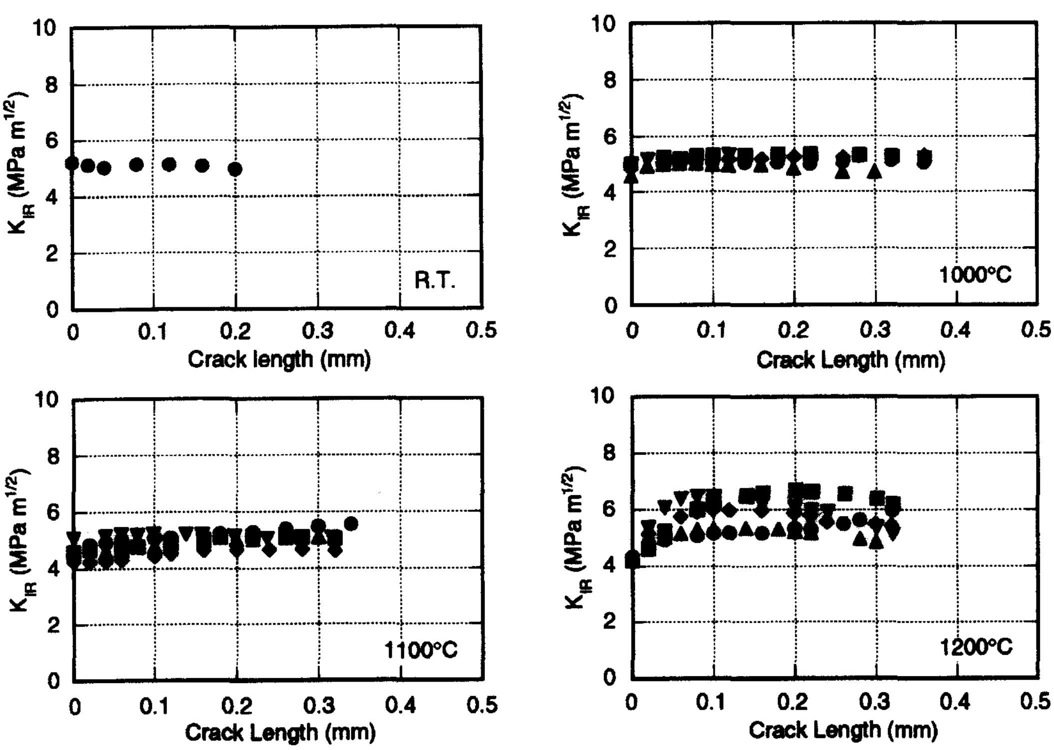

Fig. 5. R-curve behavior of SiC whisker reinforced silicon nitride (SCwSN). No rising R-curve is observed either at room or elevated temperature.

GPSN. The width of the notch was 0.1 mm. Bending was conducted with 4-point bending of 10-30 mm spans. Bending test fixture was made of SiC. Cross-head speed of the testing was 1 μm/min. Test temperatures were room temperature, 1000°C, 1100°C and 1200°C. Heating atmosphere was in air.

IV. Results

Fig. 4 shows the R-curves on HPSN at room temperature, 1000°C, 1100°C and 1200°C. Fig. 5 shows the R-curve of SCwSN at each temperature. Fig. 6 shows the R-curve of GPSN at each temperature. The scattering of the data became a little larger on high temperature tests, but this test method is well applied for high temperature measurement. However, on GPSN, creep deformation was observed at 1200°C. The obtained load-displacement data was analyzed, but the data was not meaningful. This test method cannot be applied to the condition with deformation.

All the samples show the initial fracture toughness values at room temperature around 5 to 6 MPa $m^{1/2}$. The rising of R-curve was found only on GPSN, at room temperature. At elevated temperatures, rising of R-curve was found on HPSN and GPSN samples, but SCwSN sample showed no rising even at elevated temperatures.

V. Discussion

In the results of present work, the rising of R-curve became larger with ascending temperature. In high temperature fracture, grain boundary fracture is observed, and it results in the crack deflection and the grain bridging. It brought about the rising of R-curve. In SCwSN sample, we cannot see the rising of R-curve even at high temperature. As shown in Fig. 3, we could find that the SCwSN sample has the finest microstructure among these three samples. The SiC whisker inhibited the grain growth of silicon nitride matrix, and it is the reason of the fine microstructure on SCwSN. The crack deflection on the SCwSN sample was less than that on HPSN and GPSN samples, because of this fine microstructure. This result means that the rising of R-curve is not influenced by the existence of secondary phase, but the size of the microstructure. In the results of SEM observation, HPSN has finer microstructure than GPSN. The order of the average grain size corresponds to the tendency of R-curve behavior. The rising of R-curve was easily observed in samples with large grain size.

The initial fracture toughness of each sample decreased with ascending the temperature, especially on HPSN and GPSN. In the case of fracture at elevated temperature, grain boundary fracture is observed. Then, it can be considered that the degradation of the initial fracture toughness with ascending the temperature is related to the subcritical crack growth. As in this test, the cross-head speed of 1 μm/min was used, and the total time from the initial loading to the final fracture became one hour in some test conditions. Then, the effect of subcritical crack

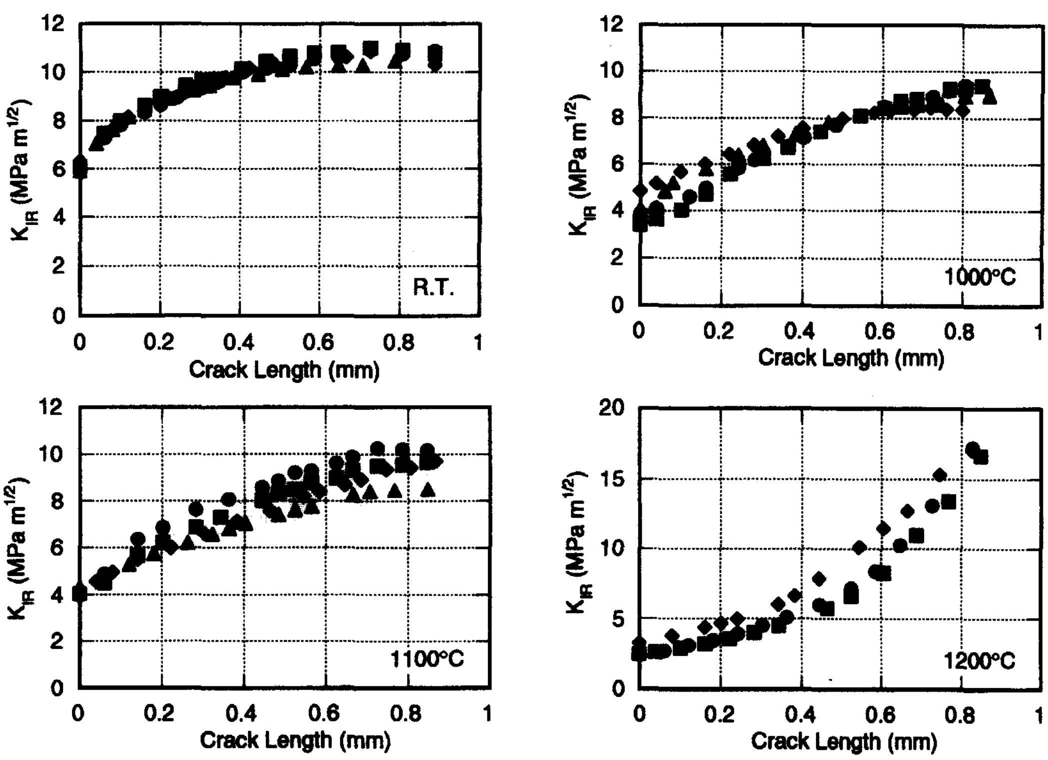

Fig. 6. R-curve behavior of gas pressure sintered silicon nitride (GPSN). Note the difference of vertical axes from Fig. 4 and 5. Rising R-curve can be found at room temperature, 1000°C and 1100°C. The influence of the creep deformation was found at 1200°C.

growth to this fracture test appeared. On the other hand, the values of fracture resistance at long enough crack length were not so different in changing the temperature. If the amount of fracture surface interaction, such as crack deflection and grain bridging, determine this value, it can be less dependent on the temperatures, because the fracture surface interaction shall be fixed by the geometrical condition, and it is independent of the temperature.

The creep behavior was found only on the GPSN sample. Normally, creep deformation is easily to occur in fine grain samples, in the case of ceramic materials. However, the creep was found only in the coarse grain sample. It may be caused by the difference of crystallization of the grain boundary phase, because of the difference of sintering procedure, but it is not yet investigated.

VI. Conclusions

R-curve behavior of three kinds of silicon nitride based ceramics were measured at elevated temperatures up to 1200°C, using SENB specimens. In HPSN, the rising R-curve could be seen only at elevated temperatures. In SCwSN, the rising R-curve did not appear even at elevated temperatures. In GPSN, creep deformation was found at 1200°C, and this test method cannot be applied on this test condition. The initial fracture toughness value became smaller with ascending temperature on all the samples. However the final rising values were not so different at each temperature on each sample. This test method can be well applied for the measurement of R-curve on ceramics at high temperatures.

References

1. J. Nakayama, H. Abe and R. C. Bradt, *J. Am. Ceram. Soc.*, **64**, 671 (1981).
2. N. Ramachandran and D. K. Shetty, *J. Am. Ceram. Soc.*, **74**, 2634 (1991).
3. T. Fett and D. Munz, *J. Am. Ceram. Soc.*, **75**, 958 (1992).
4. R. F. Pabst, J. Steeb and N. Clausen, pp. 821-33, in "Fracture Mechanics of Ceramics," vol. 4, (R. G. Bradt, D. P. H. Hasselman and F. F. Lange Eds., Plenum Press, New York, 1977).
5. Y. Matsuno, F. Wakai, M. Okada and H. Okuda, *J. Ceram. Soc. Jpn.*, **90**, 227 (1982).
6. J. E. Srawley, *Int. J. Fract.*, **12**, 475 (1976).
7. D. Munz, R. T. Bubsey and J. L. Shannon Jr., *J. Am. Ceram. Soc.*, **63**, 300 (1980).
8. S. Sakaguchi, N. Murayama, Y. Kodama and F. Wakai, *J. Mater. Sci.*, **29**, 5183 (1994).