**CONCENTRIC RING ON RING TEST FOR UNIRRADIATED AND IRRADIATED MINIATURE SIC SPECIMENS -** S. Kondo, Y. Katoh, and L.L. Snead (Oak Ridge National Laboratory)

## OBJECTIVES

The objectives of this paper are to report on the development and qualification of a equibiaxial flexural strength test for miniature thin SiC samples, and obtain strength data of SiC irradiated at very high temperatures (>1100˚C).

## SUMMARY

The flexure strength of miniature disk specimens was evaluated for both the unirradiated and irradiated CVD SiC by equibiaxial flexural test, where a disk specimen was supported on a ring and centrally loaded with a smaller loading ring. The obtained mean flexural strength and Weibull modulus were $\sigma_f$ = 352 MPa and $m$ = 5.0 for unirradiated specimen, respectively. Both of them are relatively smaller than the typical values of uniaxial tests such as 4 point bend test previously reported. However, no stress magnification at the loading ring, which is often concerned in the biaxial tests for the disk specimens, was indicated by the observation of fracture patterns. Above the irradiation temperature of 1100˚C, the flexural strength is almost same as the unirradiated values or slightly decreased at 1500˚C in contrast to the strengthening observed previously at 300-800˚C. It is clearly seen that the smooth cleavage of large grains were frequently observed in the sample irradiated at 1500˚C comparing to specimens irradiated at 1100˚C. A substantially lower population of finer defect clusters such as loops, vacancy, and vacancy clusters may be attributed to the inhibition of the strengthening at the higher irradiation temperatures. Although, many factors, such as the effective flaw populations subject to stress, may influence the relation between equibiaxial and uniaxial strength of ceramics, the simple and rapid procedure of the ring-on-ring test may be favored for the study on irradiation effects on the mechanical properties of SiC.

## PROGRESS AND STATUS

### Test apparatus

Although miniaturized disk specimens have been used for the post-irradiation mechanical tests, the stress concentrations associated with specific loading configurations such as ball-on-ring tests can be a significant problem for most ceramics because the fracture strength greatly depends on the effective loading area/volume and statistical distribution of the potential fracture origins. The equibiaxial flexural test in a ring-on-ring configuration, where a disk specimen on a support ring is loaded with a smaller coaxial loading ring, is often utilized to mitigate the stress concentration issues. For thin, high elastic modulus/strength (E/σ) ratio specimens, the uniform maximum stress occurs within the central region bounded by the loading ring.

In this study, the loading and supporting rings were designed to utilize miniature disk specimens in accordance with ASTM C1499-05 (Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature [1]) as shown in Fig. 1. The equibiaxial stress is calculated as follows:

$$\sigma_f = \frac{3F}{2\pi h^2}\left[(1-\nu)\frac{D_S^2-D_L^2}{2D^2}+(1+\nu)\ln\frac{D_S}{D_L}\right] \quad (1)$$

, where $F$ [N] is the applied load, $h$ [mm] is the specimen thickness, $D_S$ [mm] is the supporting ring diameter, $D_L$ [mm] is the loading ring diameter, $D$ [mm] is the sample diameter, and $\nu$ is Poisson's ratio

(Test Method ASTM C1259). Both the outer diameter of loading and supporting fixtures is designed to be same as the specimen diameter for ease of alignment. No compliant layer, which is sometimes used to eliminate a stress concentration and frictional stress at the rings, were used for the present study. Alternatively, industrial lubricant was applied to the ring tips. The displacement rate was set at 0.1 mm/min. The crack patterns and fracture surfaces were examined after the tests.

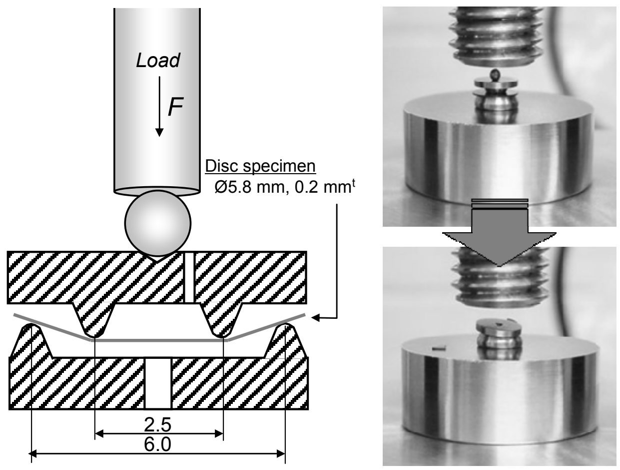


Fig. 1. Schematic and photos of concentric ring-on-ring test apparatus.

## Materials

The material used for this work was poly crystalline β-SiC which was produced by chemically vapor deposition by Rohm and Haas Advanced Materials (Woburn, Massachusetts) [2]. The CVD material is extremely pure, with typical total impurity concentration of less than 5 wppm. The grain size is between 5 and 10 μm in the plane parallel to the deposition substrate, with the grains elongated in the <111> growth direction perpendicular to the substrate. The material is typically free of micro cracks or other large flaws, but atomic layer stacking faults on the {111} planes are common. There is no porosity in CVD SiC, and the material is generally considered to be of theoretical density (approximately 3.21 g/cm$^3$).

For the unirradiated case, 23 specimens, which were machined and polished in the same manner as described below for the irradiated samples, were tested. In addition to the unirradiated specimens, specimens irradiated in the High Flux Isotope Reactor at Oak Ridge National Laboratory were tested. The fluence for the specimen studied here ranged from $5.1 \times 10^{25}$ to $9.7 \times 10^{25}$ n/m$^2$ (E>0.1 MeV). Irradiation temperatures were 1100, 1300, and 1500˚C, which were estimated by post-irradiation viewing of melt wires inserted in both ends of each sub-capsule. Specimens of 5.8 mm diameter with 3.2 mm thickness were sliced into thin disks and lap finished aiming to ~200 μm thickness with 3 μm diamond suspension for both the surfaces. A minimum of 10 test specimens tested validly is, normally, required for the purpose of estimating a mean biaxial flexural strength. For the estimation of the Weibull parameters, a minimum of 30 test specimens validly tested is recommended. However, only 5 to 6 specimens were tested in the case of post-irradiation experiment. Therefore, the Weibull statistical analysis was not conducted for the irradiated specimens.

## Results and discussion

*Unirradiated SiC*

The equibiaxial stresses are plotted as a function of the specimen thickness in Fig. 2, where the mean flexural stress is 352 MPa (Weibull mean; 357 MPa) and the standard deviation is 73 MPa. Although, the thicknesses of thin disks were varied in the range of 155-246 μm for unirradiated specimens, there was no evidence to prove a relation between the flexural stress and the thickness. The lowest flexural stress of 254 MPa was obtained for the specimen with 204 μm in thickness and the highest of 573 MPa was obtained for the specimen with 208 μm thickness. The significant scattered data points were generally found in a higher stress region as shown in Fig. 2. The edges of some specimens were slightly chipped, whereas the edge chip seemed not to affect the flexural strength in the present study.

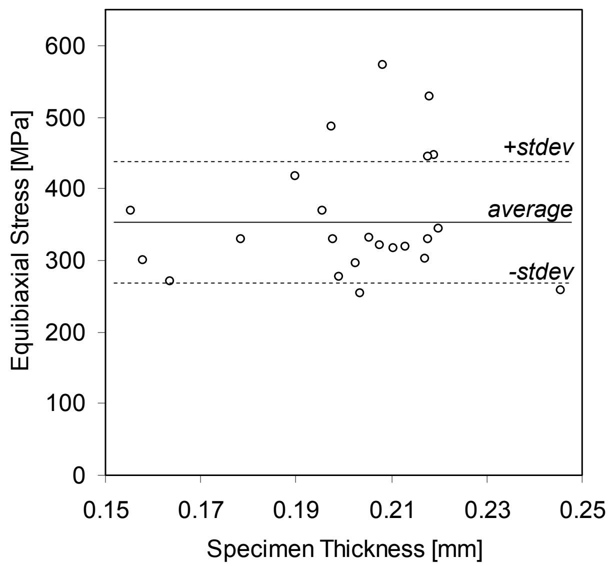


Fig. 2. Equibiaxial strength of unirradiated CVD SiC.

The room-temperature failure strength of β-SiC have been well reviewed in [3], which were mostly obtained by uniaxial and ring-compression tests and varied from 200 to over 3100 MPa. The failure strength of SiC particles is quite larger than that of the SiC bulk form. Such a large variation is due primarily to the specimen size effect. It is well known that the strength at the inner weakest flaw determines the overall strength of the brittle materials. The flaw population and distribution are strictly dependent on its volume as expressed by the Weibull distribution function. Byun et al. [4] investigated the size effect on tensile hoop strength for tubular alumina specimens using the internal pressurization and diametrical loading test techniques. A major conclusion in his work is that the failure strength of the tubular brittle specimens can be determined by the effective surface area rather than the volume [5]. Therefore, the strength obtained in the present work is strongly depended on the surface finish of the tension side. The likely thickness independent fracture strength indicates that the samples studied here were thick enough to avoid stress magnification at the loading ring. If the sample thickness is too small, the stress magnification and resultant localized deformation at the loading ring may occur due to the deviation from a linear relationship between load and displacement [6]. Although, many factors, such as the effective flaw populations subject to stress, may influence the relation between equibiaxial and uniaxial strength of ceramics, the simple and rapid procedure of the ring-on-ring test may be favored for the study on irradiation effects on the mechanical properties of SiC.

In Fig. 3, Weibull stastical plots of the flexural strength of unirradiated samples are shown. The Weibull modulus of SiC at room-temperature is reported to be widely ranged from 2 to 12, depending on the condition of the SiC material [3]. The high Weibull modulus (*m* = 7–11) was often measured by the flexural and tensile tests, while the lower values (*m* = 3–9) were obtained in the ring compression test. Cockeram [7] assumed that the flaw distributions were quite different between a flexural bar and a small tubular (ring) specimen. The lower *m* values obtained by the ring compression test were therefore considered primarily as a consequence of the changed flaw distribution. Additionally, irregularity of the ring specimen [8] or surface roughness [4] would make a significant impact on the failure probability. The obtained Weibull modulus of 5.0 for the present study is within the data band of ring compression tests and is below the data band of tensile test results. More than one failure mechanism may be involved for the ring-on-ring tests because the array of plots in Fig. 3 has an upward knee.

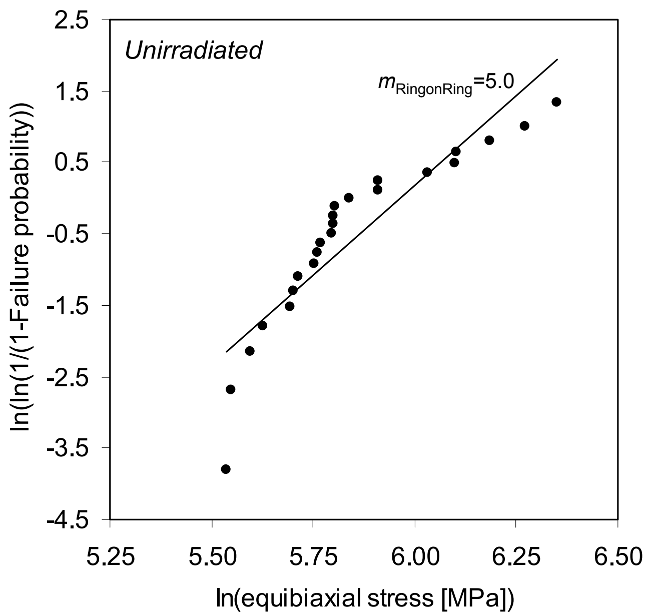


Fig. 3. Weibull plots of flexural strength of unirradiated CVD SiC.

Fractographic examination of the test specimens is recommended to determine the location of test specimen fracture [9]. Typical examples of the fracture patterns at the tensioned surface are shown in Fig. 4, where the possible contact lines of the supporting and loading rings are indicated as dotted circles. The fracture initiated likely in the area bounded by loading ring or just inside the loading ring in all samples, though the difference of the complexity of the fracture patterns were observed depending on the strength. For high strength case as Fig. 4 (a), primary crack plane is located near center line of the circular sample and significant flaw branching were observed. For intermediate strength case as Fig. 4 (b), likely crack origin was generally located near the center. These support that the successful avoidance of the stress magnification at the loading ring as stated above in the most cases. For low strength case as Fig. 4 (c), however, the primary crack was initiated just inside the loading ring, and samples were divided into only 4-5 pieces. This indicates that the results showing lower strength might be attributed to the stress magnification near the loading ring rather than the being of relatively large surface flaws that can be uniformely distributed at the disk surfaces. The misalignment of the test fixtures and the sample may be suspected as one of the primary cause of the stress magnification, if any.

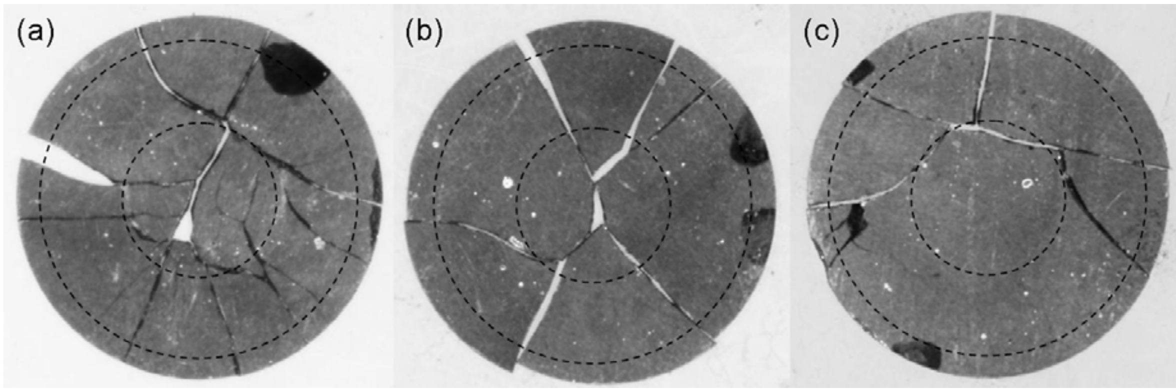


Fig. 4. Typical fracture patterns at the tensioned surface of unirradiated specimens showed a flexural strength of ; (a) $\sigma_f$ = 573 MPa,(b) $\sigma_f$ = 321 MPa, and (c) $\sigma_f$ = 270 MPa.

Table 1 Irradiation conditions and test results

| Sample ID | Irradiation Temperature [degree C] | Irradiation Fluence [dpa] | Mean Thickness [mm] | Mean Equibiaxial Stress [MPa] | Std. Dev. [MPa] | % Change [%] | Weibull Modulus | Number of samples |
|---|---|---|---|---|---|---|---|---|
| unirrad. | - | 0 | 0.202 | 357 | 87 | - | 5.0 | 23 |
| m14 | 1100 | 7.0 | 0.195 | 329 | 100 | -7.8 | - | 6 |
| m56 | 1300 | 5.1 | 0.205 | 345 | 61 | -3.4 | - | 5 |
| m20 | 1500 | 9.7 | 0.198 | 279 | 42 | -22 | - | 5 |

*Irradiated SiC*

The irradiated flexural strengths normalized to unirradiated values are plotted with neutron data reported previously as a function of irradiation temperature in Fig. 5. Above 1100˚C, the flexural strength is almost same as the unirradiated values or slightly decreased at 1500˚C. Since the strength of irradiated SiC is strongly dependent on the form of the material tested, results except for pyrolytic β-SiC are excluded from the comparison. For all the case except for the present results, the values are for Weibull's mean with error bars indicating ±1 Weibull's standard deviation [10-13]. The irradiation-induced strengthening is statistically observed at 300-800˚C. The substantial increase of fracture energy was found to be primary cause of the strengthening [14]. The formation of strength peak was attributed to the reduction of the Young's modulus accompanied by point-defect swelling (lattice expansion) [14], which was generally observed in irradiated SiC at 300-800˚C [15]. The increase in effective fracture energy is probably due primary to the dense nano-sized defect clusters [16] such as interstitial-type faulted loops formed on {111} family planes, though the fracture energy increase may be attributable to the combined effect of a number of mechanisms operating simultaneously. Meanwhile, the damage microstructure above ~1000˚C was characterized by both the voids and much larger interstitial type faulted loops in contrast to the smaller defects densely formed below 1000˚C [17]. The significant reduction in the population of irradiation-induced finer defects may prohibit the increase in fracture strength in addition to the less lattice expansion.

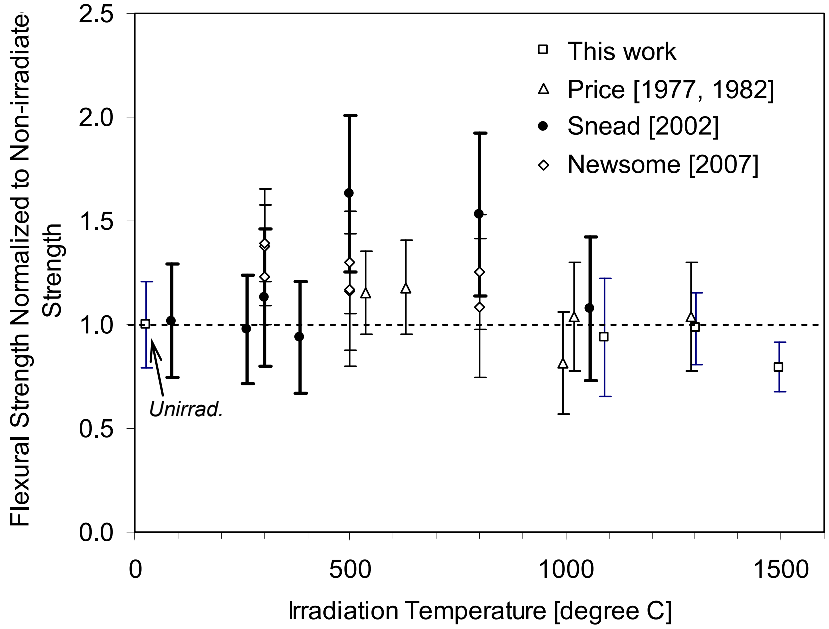


Fig. 5. Effect of irradiation temperature on normalized flexural strength of CVD SiC.

Fracture surfaces of a couple of samples tested were examined for each irradiation condition by scanning electron microscopy as typically shown in Fig. 6; irradiation temperature of 1000˚C for (a), and 1500˚C for (b), where tensioned surfaces are located on the upper side of the SEM images. In each case, fracture likely initiated at relatively large surface flaws indicated by black arrows. It is clearly seen that the smooth cleavage of large grains were frequently observed in the sample irradiated at 1500˚C as shown in Fig. 6 (b) compared to Fig. 6 (a). The same tendency of the frequent cleavage has been reported to be observed in bend-tested SiC without irradiation [14]. It has been also stated in [14] that the irradiation might increase the cleavage energy by the irradiation toughening and the Weibull modulus reduction at the irradiation temperatures ranging 300-800˚C. At 1500˚C, however, this mechanism may not be applicable because of the significant reduction of the defect cluster density. Fracture toughness and Young's modulus obtained by micro Vickers and nano-indentation techniques, respectively, were reported to be almost unchanged by the neutron-irradiation above 1100˚C. Therefore, it may be concluded that the modification of fracture energy by high temperature irradiation is also minimal for SiC as stated earlier. It suggests that the void-crack interaction, which has been observed by transmission electron microscope [18], also very limited in irradiated SiC. Furthermore, the array of voids with {111} facets, which is preferentially formed at stacking faults, may modify the fracture energy at the cleavage plane observed. Any significant differences of fracture patterns from unirradiated samples were not observed as shown in Fig. 7. It was confirmed that the ring-on-ring configuration may applicable to both the irradiated and unirradiated miniature SiC specimens. However, the sufficient number of samples to statistical analysis is strongly recommended for obtaining clear understanding of the irradiation effects due to the complex fracture mechanisms.

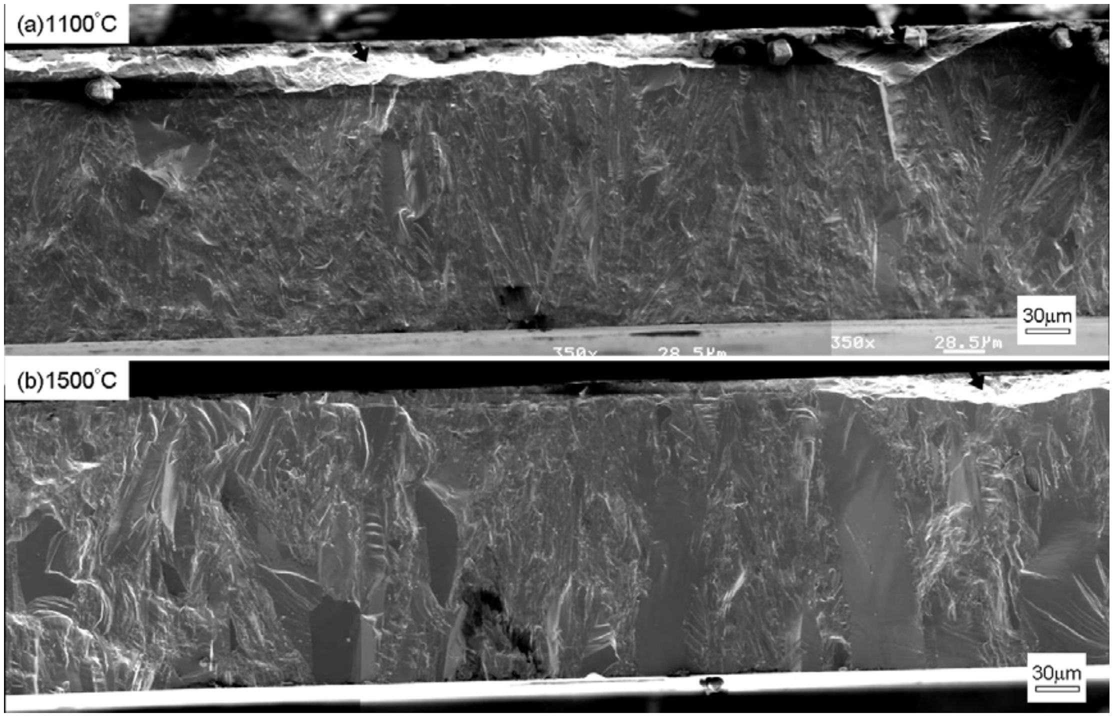


Fig. 6. Scanning electron microscope images of the fracture surface of the samples irradiated at (a) 1100˚C, and (b) 1500˚C.

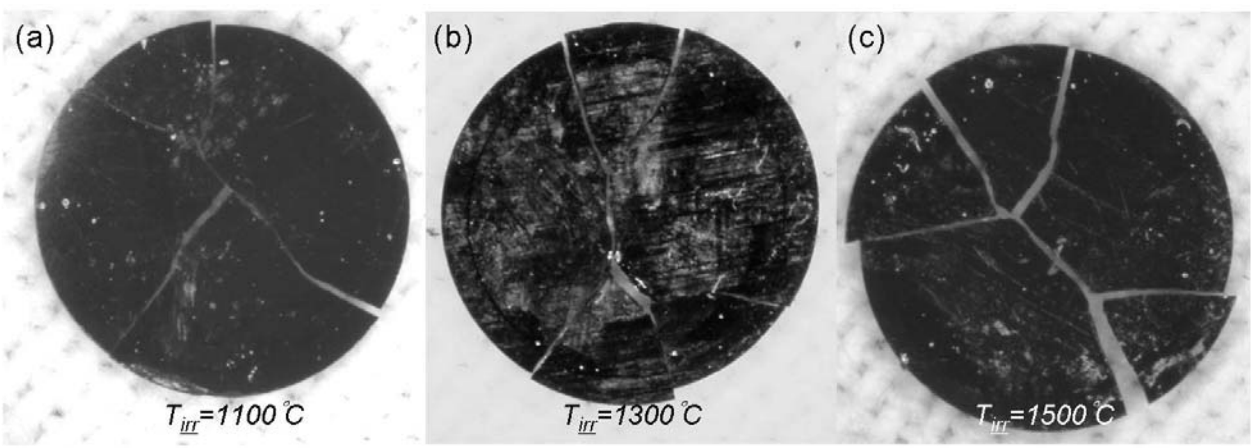


Fig. 7. Typical fracture patterns at the tensioned surface of unirradiated specimen; (a) $T_{irr}$= 1100˚C, $\sigma_f$ = 314 MPa, (b) $T_{irr}$= 1300˚C, $\sigma_f$ = 339 MPa, and (c) $T_{irr}$= 1500˚C, $\sigma_f$ = 313 MPa.

## References


[1] ASTM C 1499-05
[2] http://www.cvdmaterials.com/
[3] L.L. Snead, T. Nozawa, Y. Katoh, T.S. Byun, S. Kondo, D.A. Petti, J. Nucl. Mater. 371 (2007) 329-377.
[4] T.S. Byun, E. Lara-Curzio, R.A. Lowden, L.L. Snead, Y. Katoh, J. Nucl. Mater. 367-370 (2007) 653-658.
[5] Report CEGA-002820, Rev. 1 (July 1993).
[6] J.W. Kim (Private communication).
[7] B.V. Cockeram, J. Am. Ceram. Soc. 85 (2002) 603.
[8] K. Minato, K. Fukuda, K. Ikawa, J. Nucl. Sci. Technol. 19 (1982) 69.
[9] ASTM C 1322
[10] R. Price, Nucl. Technol. 35 (1977) 320-336.
[11] R.J. Price and G.R. Hopkins, J. Nucl. Mater. 108&109 (1982), 732-738.
[12] L.L. Snead, T. Hinoki, Y. Katoh, Report DOE/ER-0313/33 (2002).
[13] G. Newsome, L.L. Snead, T. Hinoki, Y. Katoh, D. Peters, J. Nucl. Mater. 371 (2007) 76-89.
[14] Y. Katoh, L.L. Snead, J. ASTM Intl. Paper ID JAI12377.
[15] Y. Katoh, H. Kishimoto, A. Kohyama, J. Nucl. Mater. 307-311 (2002) 1221.
[16] Y. Katoh, T. Hinoki, A. Kohyama, T. Shibayama, and H. Takahashi, Ceram. Eng. Sci. Proc. 20 (1999) 325-332.
[17] S. Kondo, et al., in proceedings of ICFRM-13, J. Nucl. Mater. (2008).
[18] F.W. Clinard, Jr., G.F. Hurley, R.A. Youngman, and L.W. Hobbs, J. Nucl. Mater. 133&134 (1985) 701-704.