Fracture analysis of porous Si–SiC ceramics with anisotropic three-dimensional network structure

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The fracture mechanism and reliability of typical dense ceramics can be statistically analyzed using a Weibull distribution as a basis for device design. However, it is not understood if the Weibull distribution can even be applied to highly porous ceramics. In this study, porous Siliconized Silicon Carbide (Si–SiC) ceramics with anisotropic three-dimensional network structures in a porosity range of 71–92% were fabricated and subjected to a fracture analysis using the results of three-point bending tests. Observations of fracture behavior during the bending tests were conducted using a high-speed camera, image analysis of stress distribution, and observation of crack distribution inside the partially damaged specimens using X-ray computed tomography. The results indicate non-linear behavior with multiple peaks in the load–displacement curves. In this regard, the fracture mechanism of the porous Si–SiC ceramics was intrinsically different from the brittle fracture of dense ceramics and did not appear to be based on the weakest-link model. However, the Weibull distribution was found to be applicable to the bending strength of the porous ceramics with a confidence coefficient of 0.90. This was because although strain and cracks were generated sporadically during loading, the catastrophic fracture of the porous Si–SiC ceramic specimens occurred with a macroscopic crack opening at the bottom of the test specimens, almost the same as a Mode I crack opening in dense ceramics. Furthermore, graded three-layer structures can be formed integrally using the proposed novel replication method with uniaxial pressing, taking the plateau of the stress–strain curve of the template polyurethane foam into account, providing a kind of damage tolerance owing to the sporadic generation and sequential propagation of cracks, manifested as the multiple peaks shown in the load–displacement curves.

Key-words : Porous Si–SiC, Fracture analysis, Weibull distribution, Anisotropic structure, Graded structure

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1. Introduction

Recently, porous ceramics have been identified as key materials for addressing environmental and energy issues.¹,² In particular, porous Siliconized Silicon Carbide (Si–SiC) ceramics have been used in filters, catalyst supports, and kiln furniture for their excellent features including light weight, low heat capacity, and good permeability.³–⁶ Previously, we fabricated porous Si–SiC ceramics with an anisotropic three-dimensional network structure in a porosity range of 83–92% using a novel replication method with uniaxial press molding.⁷ To meet the requirements of practical application, it is very important to determine the fracture mechanism and reliability of these ceramics.

It is well known particularly for dense ceramics that when load is applied during a bending test, the load–displacement curve shows linearity, indicating that instantaneous catastrophic fracture occurs immediately after the curve peak (yield point). This curve has been extensively analyzed with the Weibull distribution based on the weakest-link model, and has been used as a basis for device design.

While the fracture behavior of porous ceramics is quite complex, and has not thus far been sufficiently understood, though there are several published reports on the fracture analysis of porous ceramics in a porosity range of 30–75%. It has been reported that the Weibull statistics were effectively applied to porous alumina ceramics with a porosity range of 30–40% in a four-point bending test.⁸ However, in a different report, a two-parameter Weibull distribution was found not to be in good agreement with a four-point bending test of porous SiC ceramics with porosity of 35%, therefore the use of a Markov process was proposed instead.⁹ In accordance with Japanese Industrial Standards (JIS) R 1625:2010, Weibull statistics (single mode, two-parameter) of the strength data of fine ceramics have been specified to target brittle fracture due

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Another study found that the load–displacement curves of porous alumina in a compression test exhibited a behavioral transition between porosities below and above 60%, and found non-linearity in the load–displacement curve of porous alumina ceramics with porosity of 65%. However, there have been few detailed reports on the fracture analysis of highly porous ceramics, especially with a porosity in the range of 75–95%. Therefore, it has not yet been clarified if the Weibull distribution can be applied in the fracture analysis of these porous ceramics.

Notably, the forming process used in this study allowed for an anisotropic three-dimensional Si–SiC network structure in which the resultant porosity and mechanical function can be easily controlled by varying the compression ratio of the templates in the uniaxial press.

One example practical industrial target for this type of porous ceramic is a very lightweight kiln plate used for both degreasing and firing ceramic parts, especially in roller hearth furnaces, in which gas permeability are required in addition to strength and stiffness. The highly porous ceramic can also provide a low heat capacity to save thermal energy. When then a kiln plate is set on the rollers under load, the stress state in the kiln plate between the rollers would be similar to that applied by the bending tests.

In general, a sandwich structure is widely used as a means to produce lightweight members with high strength and stiffness. For example, conventional porous Si–SiC ceramics are typically sandwiched between dense SiC plates using the paste technique. However, when such adhesion structures are used especially in structural members exposed to large heating or cooling demands, avulsion can occur easily at the boundaries between the surface layer and the intermediate layer. Moreover, the dense ceramic surface layers tend to fail in instantaneous catastrophic brittle fracture and have low gas permeability.

The purpose of this study is therefore to determine the fracture mechanism of porous Si–SiC ceramics in order to establish the appropriateness of applying a Weibull distribution to its analysis. In this report, the fracture behaviors of porous Si–SiC ceramics (with a porosity range of 71–92%) were investigated based on load–displacement curves derived from three-point bending tests, and the integration of material information including the forming condition, characteristics of the microstructure, and fracture mechanism was attempted.

### 2. Experimental procedures

#### 2.1 Fabrication of anisotropic porous Si–SiC ceramic slabs

Anisotropic porous Si–SiC ceramic slabs were fabricated using a replication method with uniaxial press molding as follows. Fine SiC powder slurry was prepared and four different slabs of 150 mm × 150 mm polyurethane foam, 6, 12, 15, and 18 mm thick with a three-dimensional network structure were prepared as templates. The slabs of polyurethane foam were impregnated with the SiC powder slurry, then excess slurry was removed so that a thin SiC outer layer was formed on the surface of polyurethane struts of the slabs.

After drying at room temperature, the replicated green bodies with their three-dimensional network structure were uniaxially pressed, controlling the final thickness to 6 mm. After the green bodies were subjected to curing and pyrolysis, liquid silicon was infiltrated into not only the boundaries between SiC grains but also into the trace cavity of the burned polyurethane at a temperature of 1500°C in Argon gas. In this manner, four types of porous Si–SiC ceramic slabs with varied anisotropic three-dimensional network structures were prepared.

The compression ratio (CR) was defined by dividing the thickness of the template by that of the pressed green body. Note that the thickness of each sintered body was almost equal to that of the green body because the shrinkage due to sintering is quite small in such Si–SiC ceramics. The four slab types were named CR1, CR2, CR2.5, and CR3 according to their respective compression ratios.

The bulk density, $D_b$, of each slab was determined by dividing the slab weight by its volume. The theoretical density, $D_{th}$, of the dense Si–SiC strut was determined to be 2.84 using the chemical composition in accordance with JIS R 2011:2007. The total porosity, $P_{total}$, was determined according to the following equation:

$$P_{total} = \left(1 - \frac{D_b}{D_{th}}\right) \times 100$$

The microstructure at the vertical cross section of each slab was observed with a scanning electron microscope (SEM) (JSM-5600, JEOL Ltd.). And then each SEM image was binarized using image analysis software (PicMap, free software) with a threshold of 60 to distinguish the struts of the anisotropic three-dimensional network structure.

#### 2.2 Testing method for bending strength

Three-point bending tests were performed and load–displacement curves were constructed in accordance with JIS R 1664:2004 and JIS R 1601:2008, necessarily preventing the one-sided contact of load points in a four-point bending test. A set of ten test specimens were cut to a length of 70 mm, width of 8 mm, and thickness of 6 mm from each sintered slab CR1, CR2, and CR3. Note that neither the top nor the bottom surfaces were polished, as the loading direction in the bending test was identical to the uniaxial pressing direction in the forming process.

The bending test apparatus (CB50–U5 and D5, AIKOH ENGINEERING Co., Ltd.) and one of the test specimens are shown in Fig. 1. The span between support points was 60 mm and the cross-head applied displacement at a rate of $8.33 \times 10^{-3}$ mm/s. Ten load–displacement curves were thus obtained for each slab type using the three-point bending strength, $\sigma_{b3}$, calculated as follows:

$$\sigma_{b3} = \frac{3PL}{2wt^2}$$
where \( P \) is the maximum load, \( L \) is the span between support points, and \( w \) and \( t \) are the width and the thickness of the specimen, respectively.

2.3 Observing fracture behavior during bending tests

The fracture behavior of each specimen during the bending tests was observed using a high-speed camera (FASTCAM Mini AX200, PHOTRON Ltd.) from the beginning of loading to the moment of catastrophic fracture. The obtained sequential images were correlated with the load–displacement curves in a time series. The strain distributions in the front and bottom surfaces of the specimen were analyzed using image analysis software (GOM Correlate, V8 SR1, GOM GmbH). An elemental mesh for this analysis was generated on the object area, except near the load point to exclude any strain in the area. The relative displacement of points in this mesh were then distributed to create a counter map.

2.4 Observing internal microstructure of partially damaged specimens

In the early stage of the bending test, loading operations were stopped immediately after fluctuations were detected in the load–displacement curve before catastrophic fracture. The partially damaged test specimens from slabs CR1, CR2, and CR3 were then observed using microfocus X-ray computed tomography (SMX-160CT, SHIMADZU Corp.) to scan inside the specimens vertically from the bottom surface towards the top surface, obtaining horizontal cross sections around the center of each. In this manner, the observation area of the horizontal cross section was cylindrical in shape with diameter of 11 mm and a slice thickness of 19.8 \( \mu \)m.

3. Results and discussion

3.1 Weibull analysis of bending strength

The physical and mechanical properties of CR1, CR2, CR2.5, CR3, and the dense Si–SiC ceramic equivalent to the outer layer of the strut are summarized in Table 1. An SEM image taken at the vertical cross section of CR1, CR2, and CR3 are shown in Fig. 2. The typical load–displacement curves of CR1, CR2, and CR3 are shown in Fig. 3(a), while that of the dense Si–SiC ceramic is shown in Fig. 3(b). Note that non-linearity, indicated by characteristic multiple indents, appears in each load–displacement curve of the subject porous ceramics. Therefore, it was not clear at this point if Weibull statistics could be applied to these strength data because these statistics are specified to target brittle fracture due to a single cause, assuming linearity in the load–displacement curve.

To determine the applicability of Weibull statistics, first, the three-point bending strength data from the ten test specimens were analyzed. Table 1 lists the physical and mechanical properties of the slabs.

| Table 1. Physical and mechanical properties of slabs |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
|                | Porous Si–SiC slabs | Dense Si–SiC     |
|                | CR1   | CR2   | CR2.5 | CR3   | CR1   | CR2   | CR2.5 | CR3   |
| Bulk density, \( D_b/\text{g cm}^{-3} \) | 0.24  | 0.47  | 0.63  | 0.81  | 0.24  | 0.47  | 0.63  | 0.81  |
| Total porosity, \( P_{\text{total}}/\% \) | 91.5  | 83.5  | 78.0  | 71.6  | —     | —     | —     | —     |
| Apparent porosity, \( P_a/\% \)     | —     | —     | —     | —     | 0.68  | —     | —     | —     |
| Bending strength, \( \sigma_b/\text{MPa} \) | 2.4   | 5.7   | 6.1   | 12.6  | 343   | —     | —     | —     |

Fig. 1. Apparatus for the three-point bending test and a porous Si–SiC ceramic test specimen.

Fig. 2. SEM images at the vertical cross section of slabs (a) CR1, (b) CR2, and (c) CR3, in which white lines indicate seams between sequential SEM images.
specimens for each slab type were analyzed using a two-parameter Weibull distribution, as shown in Fig. 4(a), in which the regression line of each is expressed by:

\[ \ln \ln \left( \frac{1}{F} \right) / \ln C_0 = m \ln \left( \frac{b_3}{C_0} \right) \]  

where \( m \) and \( \beta \) are the shape parameter and scale parameter, respectively. The cumulative fracture probability, \( F_i \), was calculated with median rank, and is expressed by:

\[ F_i = \frac{i - 0.3}{n + 0.4} \]  

where \( i \) and \( n \) are the order in the rank and the total number of data points, respectively.

The application of the two-parameter Weibull distribution seems to be valid for both \( CR_1 \) and \( CR_3 \) because their respective confidence coefficients, \( R^2 \), were equal to or greater than the criterion of 0.90.\(^{10}\) The bending strength of \( CR_3 \) was more clearly analyzed than that of \( CR_1 \). While the value of \( R^2 \) for \( CR_2 \) was slightly less than the criterion, and thus was not clearly analyzed. Therefore, \( CR_2 \) was subsequently analyzed with a three-parameter Weibull distribution, resulting in an \( R^2 \) value greater than 0.90, showing good fit when using a location parameter, \( \gamma \), as shown in Fig. 4(b). The regression line of the three-parameter Weibull distribution can be expressed by:

\[ \ln \ln \left( \frac{1}{F} \right) / \ln C_0 = m \ln \left( \frac{b_3}{C_0} \right) + \ln \gamma \]  

Accordingly, the application of a two-parameter Weibull distribution to \( CR_1 \) and \( CR_3 \) appears adequate, while for \( CR_2 \) a three-parameter Weibull distribution was required. Note that a three-parameter Weibull distribution cannot be applied to \( CR_1 \) and \( CR_3 \) because an extreme value of \( R^2 \) that would require fitting with a location parameter was not obtained. The parameters of each Weibull distribution are summarized in Table 2.

### 3.2 Relationship between the multiple load-displacement peaks and the maximum bending strength

In order to separate significant load-displacement peaks from noise, the aberrations observed in the load-displacement curves were defined quantitatively using the following method. First, differential values expressed as a change in load over time (N·s\(^{-1}\)) were calculated. These differential values were then filtered with five thresholds: 2.5, 5, 7.5, 10, and 20% of the maximum value, which in most cases was at the last peak. When the differential value of the individual aberration was larger than the applied threshold, it was determined to be a peak, and when it was

\[ \ln \ln \left( \frac{1}{F} \right) / \ln C_0 = m \ln(\sigma_{b3} - \gamma) - m \ln \beta \]  

Fig. 3. Typical load–displacement curves of (a) \( CR_1 \), \( CR_2 \), and \( CR_3 \); and (b) dense Si–SiC ceramic.

Fig. 4. (a) two-parameter Weibull distribution of the three-point bending strength data for \( CR_1 \), \( CR_2 \), and \( CR_3 \); and (b) three-parameter Weibull distribution for \( CR_2 \).
equal to or smaller than the threshold, it was determined to be noise.

The relationship between the number of peaks and the maximum bending strength was plotted in a scatter diagram representing number of peaks on the horizontal axis and the maximum bending strength on the vertical axis. The number of peaks, inclination and confidence coefficient, $R^2$, of the regression line of each distribution are summarized in Table 3. Note that the numerical values in the table are the averages of all ten specimens in each data set. The correlation between the number of peaks and the bending strength was quite low in every specimen, even though bending strength tended to be slightly lower as the number of peaks increased. Additionally, there was no statistical significance for a two-way classification between the CR type and thresholds.

However, investigation into the state change before and after individual peaks, the inclination of each peak (determined by connecting the zero-point to the first peak-top, then from the first peak-bottom to the second peak-top, and so on) found that inclination had a high probability of increasing before and after the individual peaks in every specimen. This indicates that the apparent elasticity of the test specimens continuously increased during the bending test. In this manner, the fracture mechanism of these porous ceramics should be considered to be intrinsically different from that of dense ceramics. Therefore, it cannot be considered that the fracture occurred as the result of any one cause during the bending test, as required when applying the weakest-link model.

3.3 Image analysis of strain distribution, and capturing the moment of catastrophic fracture

The strain distributions in the front and bottom surfaces of the CR2.5 test specimens were obtained at 60 fps using a high-speed camera and subsequent image analysis, with the results shown in Fig. 5. Note that the observations of the bottom surface were accomplished using a mirror set beneath the test specimen. Between 40 and 60 s after the beginning of loading, strain appeared sporadically all over the front and bottom surfaces. After 77 s, the specimens showed localized strains at the center of the bottom face, and after 82 s, larger strains began to appear in the center of the bottom face before the specimen catastrophically fractured 83 s into loading. The generation and elimination of sporadic local strain continuously occurred up to catastrophic fracture. It is theorized that the generated local strain was eliminated by the corresponding local fracture of struts in the specimen.

The moment of catastrophic fracture was captured at 2000 fps using a high-speed camera, and can be seen in Fig. 6. At the beginning of loading, slight displacement

### Table 2. Parameters of each Weibull distribution

| Threshold /% | Porous Si–SiC | Dense Si–SiC |
|--------------|--------------|-------------|
|              | CR1 | CR2 | CR3 | CR1 | CR2 | CR3 |
| Two-parameter Weibull distribution | Shape parameter, $m$/— | 9.33 | 6.97 | 5.51 | 4.72 |
| Scale parameter, $b$/— | 2.55 | 6.06 | 13.7 | 424 |
| Three-parameter Weibull distribution | Confidence coefficient, $R^2$/— | 0.904 | 0.895 | 0.94 | 0.99 |

### Table 3. Relationship between number of peaks and corresponding maximum bending strength

| Threshold /% | CR1 | CR2 | CR3 |
|--------------|-----|-----|-----|
| Number of peaks/— | 2.5 | 240 | 21 | 7 |
| 5 | 50 | 4 | 3 |
| 7.5 | 8 | 3 | 2 |
| 10 | 2 | 2 | 2 |
| 20 | 1 | 1 | 1 |
| Inclination of the regression line/— | 2.5 | 0.00 | —0.03 | —0.31 |
| 5 | —0.01 | —0.10 | —0.41 |
| 7.5 | —0.02 | —0.06 | —0.10 |
| 10 | —0.07 | —0.22 | —0.63 |
| 20 | —0.07 | —0.27 | —4.98 |
| Confidence coefficient, $R^2$/— | 2.5 | 0.49 | 0.40 | 0.26 |
| 5 | 0.39 | 0.03 | 0.10 |
| 7.5 | 0.19 | 0.01 | 0.30 |
| 10 | 0.07 | 0.07 | 0.06 |
| 20 | 0.01 | 0.03 | 0.49 |
and the falling of fine debris from the specimen were observed. With the continued progression of loading, the test specimen slightly deformed in a downward convex shape. Subsequently, catastrophic fracture occurred with the instantaneous generation and opening of a macroscopic crack on the bottom face, macroscopic crack opening, and catastrophic fracture.

In the typical brittle fracture behavior of dense ceramics, a strain concentration develops at an original defect, generating a crack that propagates instantaneously during crack opening. While in the subject porous Si–SiC ceramic, the local strain and resulting debris were generated sporadically, independent of the development of catastrophic fracture. Though the progress of the catastrophic fracture was different from that of dense ceramics, the porous ceramic specimens still exhibited macroscopic behavior similar to that of dense ceramics at the moment of catastrophic fracture.

### 3.4 Observation of sporadic cracks inside the test specimen

For test specimen CR1, because no cracks were observed inside the test specimens near the center, buckling near the support points could potentially occur. For test specimen CR3, contiguous cracks were observed on the bottom surface propagated towards the top surface, much like in dense ceramics. However, in this state the test specimens had not yet reached catastrophic fracture and thus remained undamaged. At this point, the fracture behavior of the subject porous ceramics was different from that of dense ceramics.

Slice images of the horizontal cross section of the internal microstructure of the CR2 test specimen are shown in Fig. 7. Slice images in which cracks appeared were observed alternately with those without cracks in the vertical direction. This indicates that the cracks had not yet grown substantially connected to each other. Cracks were not observed on the bottom surface, but rather were observed sporadically on the top surface and intermediate portions inside the test specimen, as shown in Fig. 8.

As a result, it was determined that the initial fracture behavior of the subject porous ceramics is intrinsically different from that of dense ceramics: it appears that...
bending stress was generated within a limited area in the top surface and intermediate portions inside the specimens so that the propagation of cracks towards the bottom surface was suppressed at macro pores in the lower intermediate portions. Moreover, although cracks were generated sporadically in the early stages of the bending tests, it is theorized that as loading progresses, macroscopic cracks would be generated on the bottom surface and then propagate at catastrophic fracture.

3.5 Appropriateness of applying Weibull statistics

In general, as Weibull statistics are based on the weakest-link model and Weibull statistics (single mode, two-parameter) of the instant break strength data of fine ceramics have been specified in JIS R 1625:2010,\(^{10}\) it is necessary to discuss if the subject porous Si–SiC ceramics exhibit single-cause instantaneous fracture and are compatible with the weakest-link model.

As the dense Si–SiC ceramics basically consists of covalent bonding, it exhibits elastic deformation and brittle behavior, so the stress concentrations around a defect in the specimen cannot be released. Therefore, the tensile strength of the specimen is determined by the weakest defect (the weakest-link model), and Weibull statistics can be applicable.\(^{16}\)

In the strictest sense, three cracking modes are used in fracture mechanics: Modes I, II, and III, as well as a mixed mode.\(^{17}\) Mode I is an opening (tensile) mode in which the crack surfaces move directly apart. Mode II is a sliding (in-plane shear) mode in which the crack surfaces slide over one another in a direction perpendicular to the leading edge of the crack. Mode III is a tearing (out-of-plane shear) mode in which the crack surfaces move relative to one another and parallel to the leading edge of the crack. At brittle fracture, for Mode I, when the stress intensity factor, \(K_I\), is larger than the fracture toughness, \(K_{IC}\), a crack propagates instantaneously, reaching catastrophic fracture. Therefore, it can be considered as a form of single-cause instantaneous fracture based on the weakest-link model.

While as the subject porous ceramics, even though the local microscopic cracks are sporadically accumulated until the catastrophic fracture, Weibull statistics are applicable to analyze the reliability of the strength, according to two reasons as; one is that the porous ceramics fractured in a catastrophic manner where a macroscopic crack, around the tensile surface of a bending specimen, opens in Mode I similar to typical brittle dense ceramics. The other reason is that the microscopic cracks are owing to the local collapse of the porous ceramics near the load/supporting points where are far and independent from the macroscopic crack. The energy release by the local collapse basically does not affect the elastic strain energy which should be a driving force of the macroscopic crack generation and its propagation because the situation of macroscopic bending is soundly maintained even after the sporadic local collapse. In this way, the catastrophic fracture can be considered as a form of single-cause instantaneous fracture based on the weakest-link model.

Furthermore, these are also the reasons why the correlation between the number of peaks in the load–displacement curves and the maximum bending strength is not significant (as described in Section 3.2), and the fact supports that Weibull statistics can be applied to the subject porous ceramics.

Note that the fracture of the subject porous ceramics might be considered using a multi-mode Weibull distribution\(^{18}\) or others, because of the sporadic fractures as multiple diverse causes. Since it was difficult to distinguish between multiple causes, this study was limited to the use of a single-mode Weibull distribution, regarding all fractures to be due to a single cause for each specimen.

3.6 Graded three-layer structure of three-dimensional network

As shown in Fig. 2, a graded three-layer, sandwich-like three-dimensional network structure, consisting of a porous intermediate layer between two rather denser surface layers, was integrally formed during fabrication. This is likely because the particular polyurethane foam used as the template was not homogeneously deformed during uniaxial pressing. Thus, the top and bottom surface layers, compressing first, were more deformed and thus denser than the intermediate layer, which deformed later and thus to a lesser extent.

The results of the image analyses quantifying the graded structures in each SEM image are summarized in Table 4. Note that the struts in the three-dimensional network are expressed as a white area in each binarized image, so that the slab density is defined by the ratio of white area in each object image. The most clearly defined graded three-layer structure appeared in slab \(CR2\). In slab \(CR1\), although the thickness of the template was 6 mm (equivalent to that of the test specimen), once the template swelled and increased to 1.2 times the thickness with the slurry medium

| Table 4. Ratio of slab surface layer density to intermediate layer density |
|---|---|---|---|
| | CR1 | CR2 | CR3 |
| Defined surface layer thickness/mm | 1.0 | 1.0 | 1.5 |
| Top side | Slab density/% | Surface layer | 93.2 | 89.8 | 86.8 |
| | Intermediate layer | 74.6 | 74.7 | 79.6 |
| | Ratio of slab density/— | 1.25 | 1.20 | 1.09 |
| Bottom side | Slab density/% | Surface layer | 83.5 | 83.2 | 79.7 |
| | Intermediate layer | 75.9 | 68.0 | 71.6 |
| | Ratio of slab density/— | 1.10 | 1.22 | 1.11 |
prior to uniaxial pressing, it was compressed strictly with a CR of 1.2. The compression load could thus be too small to sufficiently deform the top and bottom surface layers, so the graded three-layer structure was not clearly formed. For slab CR3, the compression load may have been too large, deforming not only the top and bottom layers, but also the intermediate layer.

These phenomena, and thus the methods for controlling the template so that the graded three-layer structure is formed, can be explained by examining the plateau in the stress–strain curve of the polyurethane foam. It is well established that the deformation of a flexible polyurethane foam exhibits three stages in its stress–strain curve: an initial linear elastic stage, a plateau stage in which small increases in load result in large additional strain (deflection), and a densification stage.10 The range of the plateau is related to the hardness and original thickness of the polyurethane foam.

For the CR2 template with an original polyurethane foam thickness of 12 mm, a plateau can be observed in its stress–strain curve within a deflection range of 16–61% (2.3–8.8 mm, considering the 1.2 times swelling, as previously discussed), determined in accordance with JIS K 6400-2:2012.20 The degree of uniaxial compression in the forming process was 58.3% (8.4 mm, considering the swelling), just within the deflection range. However, for both CR1 and CR3, each compression distance was out of the appropriate range. For CR1, because too small of a load was applied near the lower limit of the plateau, the top and bottom layers of the polyurethane foam were barely deformed, while for CR3, because too large of a load was applied near to exceeding the upper limit of the plateau, the polyurethane foam was almost wholly deformed.

Forming conditions (as indicated by the CR) may account for the multiple peaks in the load–displacement curves. Indeed, for CR2, the gradation of specimen density was the most definite, and the initial cracks were generated not in the bottom surface but in the top and intermediate layers. This could be one of reasons that the bending strength could not be effectively analyzed with a two-parameter Weibull distribution in this specimen (as described in Section 3.1).

A novel replication method considering the plateau in the stress–strain curve of the polyurethane foam is thus proposed so that the sandwich-like graded three-layer structure of anisotropic three-dimensional network will be integrally near-net formed without adhesion, potentially suppressing avulsion at the boundaries (change in density).

An advantage of the sandwich-like graded three-layer structure is that a kind of damage tolerance can be provided, such that even though a member may be partially damaged, it will not immediately reach catastrophic fracture, which may improve its apparent fracture toughness and reliability.

This improvement is based on the clarification of the fracture behavior of the Si–SiC porous ceramics as follows: (1) although some struts in the three-dimensional network may be partially fractured, catastrophic fracture of the entire specimen does not necessarily occur all at once; (2) catastrophic fracture is caused by a macroscopic crack opening in the bottom surface, similar to the Mode I crack opening in typical dense ceramics; and (3) the bending strength was affected by the specimen density, and the higher the specimen density, the higher the bending strength.

It is preferable that the surface layers should be as dense as possible, because sporadic fractures could be created first in the more porous intermediate layer, bending strength of the bottom surface layer becomes higher, and a macroscopic crack opening likely could be suppressed by refraction at the boundary between the intermediate and top surface layers.

4. Conclusions

Porous Si–SiC ceramics with anisotropic three-dimensional network structures in a porosity range of 71–92% were fabricated and fracture analyses of three specimen types, CR1, CR2, and CR3 were conducted using a three-point bending test. The results informed the following conclusions:

1) Non-linearity with multiple peaks was observed in the load–displacement curves of all specimens, and the relationship between the number of peaks in the curves and the maximum bending strength was insignificant.

2) A two-parameter Weibull distribution could be applied to CR1 and CR3, but a three-parameter Weibull distribution had to be applied to CR2 to achieve a confidence coefficient of 0.90.

3) The fracture mechanism of these specimens was different from that of dense ceramics and it could not be accurately based on the weakest-link model, because the local cracks were generated sporadically, independently of the development of catastrophic fracture.

4) The Weibull distribution was applicable because the catastrophic fracture of these specimens exhibited a macroscopic crack opening, by the intrinsic degree of stress concentration resulting from the specimen density, similar to a Mode I crack opening in dense ceramics.

5) For CR2, a graded three-layer structure could be formed integrally using a novel replication method based on uniaxial pressing by considering the plateau of the stress–strain curve of the polyurethane foam as a template.

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