Improvement of static fatigue strength of Si₃N₄/SiC crack-healed under cyclic stress

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Abstract

Si₃N₄/SiC composite ceramics were hot-pressed in order to investigate their crack-healing behaviour and the resultant static fatigue strength. Semi-elliptical surface cracks of 100 μm in surface length were made on each specimen. The pre-cracked specimens were crack-healed under a cyclic bending stress of 210 MPa in air at 900, 1000, 1100, and 1200 °C. The bending strength and static fatigue strength of the crack-healed specimens were systematically investigated at each healing temperature. The specimens which has been crack-healed and static fatigue-tested at 900 and 1000 °C showed lower static fatigue strength than those tested at 1100 and 1200 °C. Detailed investigation on the fracture surface of static fatigue-tested specimens showed that oxidation of the base material had strong effects on the static fatigue strength. It was found that if the specimens were pre-oxidized in air at 1300 °C, the surface was covered by a protective oxide layer, leading to a significant improvement of static fatigue strength at 900 and 1000 °C.

1. Introduction

Silicon nitride (Si₃N₄)-based ceramics and composites have a large number of industrial applications because of their excellent mechanical, tribological, and thermal properties. These applications include turbo charger rotors, diesel engine components, cutting tools, and bearings. Some engineering ceramics, including Si₃N₄, have a crack-healing ability.[1–7] The use of this self crack-healing ability on structural components in engineering applications may produce great benefits such as an increase in the reliability of structural ceramic members and a decrease in the inspection, machining, and polishing costs of ceramic components. We investigated the crack-healing behaviours of Si₃N₄/SiC,[8–12] mullite/SiC,[13–15] SiC[16,17] and Al₂O₃/SiC,[18] which all have high self-crack-healing abilities. Systematic studies are needed to maximize the benefits of crack-healing. We need to determine, for example, (i) the best healing conditions for high-temperature strength,[8,9,15–17] (ii) the maximum crack size that can be healed completely,[9,15,16] and (iii) the effects of environment on crack-healing.[6,8,13]

Ceramic components are often operated continuously under constant or cyclic loading at elevated temperatures. If a crack is initiated during service, the component's reliability will be reduced considerably. If the crack could be healed under service conditions, and the healed zone had sufficient strength, the reliability and lifetime of ceramic components could be increased. From this view point, we investigated the crack-healing behaviour of Si₃N₄/SiC under cyclic stress in

temperatures ranging from 900 to 1200 °C and the resultant cyclic fatigue strength at healing temperatures of 1100 and 1200 °C.[12] It was found that pre-cracks of 100 µm could be healed even under cyclic bending stress, and that the resultant cyclic fatigue strength was quite high. However, the static fatigue strength of Si_3N_4/SiC crack-healed under cyclic stress in the above temperature range was not studied.

In actual engineering applications of non-oxide ceramics, oxidation is an important issue which must be considered. The high-temperature oxidation behaviour of Si_3N_4-based materials has been the subject of much study. It has been pointed out by several researchers that the oxidation reactions of some Si_3N_4-based materials progress faster at about 1000 °C than those of higher temperature (e.g. >1200 °C).[19–23] Considering these oxidation behaviour, the crack-healing behaviour and resultant static fatigue strength should be investigated over a wide temperature range.

In this study, we tested Si_3N_4/SiC which has a very high degree of self crack-healing ability. The static fatigue strength of the specimens crack-healed under cyclic stress was investigated at healing temperatures of 900–1200 °C. The influence of oxidation on static fatigue strength and a method to improve static fatigue strength were discussed.

2. Experimental procedures

2.1. Material, test specimen and pre-cracks

The silicon nitride powder (SN-E10, Ube Industries Ltd., Ube, Japan) used in this study has a mean particle size of 0.2 µm; the volume ratio of α-Si_3N_4 is about 95%, the rest being β-Si_3N_4. The SiC powder (Ultrafine grade, Ibiden Co. Ltd., Ogaki, Japan) used has a 0.27 µm mean particle size. The samples were prepared using a mixture of silicon nitride, with 20 wt.% SiC powder and 8 wt.% Y_2O_3 as an additive powder. The Y_2O_3 powder (Fine grade, Nippon Yttrium Co., Ltd., Oomuta, Japan) used has a 0.4 µm mean particle size. To this mixture, alcohol was added and blended completely for 48 h. The mixture was placed in an evaporator to extract the solvent and then in a vacuum to produce a dry powder mixture. The mixture was subsequently hot-pressed at 1800 °C and 35 MPa for 2 h in nitrogen gas. The relative density of the hot-pressed material determined by the Archimedes method was 99.5%. The value of fracture toughness (K_{IC}) evaluated by indentation fracture (IF) method (JIS R1607)[24] was K_{IC} = 6.5 MPa m$^{1/2}$. The hot-pressed material has the following microstructure: average grain size of the matrix Si_3N_4 = 0.44 µm, average aspect ratio = 5.0. The grain boundary crystals are $YSiO_2N$ and $Y_{20}N_4Si_{12}O_{48}$. Most SiC particles are located in grain boundaries and distributed uniformly. This silicon nitride was selected as a test material because it has excellent crack-healing ability, with the excellent high-temperature strength of the crack-healed zone being up to 1300 °C.[9,10]

The hot-pressed material was cut into test specimens measuring 3 mm × 4 mm × 20 mm. These specimens were subjected to three-point bending with a span of 16 mm. The length of a span for three-point bending prescribed by the Japan Industrial Standard (JIS R1601) is 30 mm.[25] The length of the bending span adopted in this study is shorter than that prescribed in the standards for the following two reasons: (a) In previous studies, most of the specimens with a bending span of 30 mm failed outside the crack-healed zone.[9–11] The objective of this study was to measure the bending strength of the crack-healed zone rather than the matrix. In order to reduce the effective volume, we selected a shorter bending span. (b) The strain energy of a sample increases as its length increases. Consequently, a longer sample tends to break into many pieces, which makes it very difficult to identify the crack initiation site. In order to reduce the strain energy, we selected a shorter bending span.

Semi-elliptical surface cracks of 100 µm in surface length were introduced at the center of the tension surface of the test specimens using a Vickers indenter at a load of about 20 N. The ratio of depth (a) to half surface length (c) of the crack (aspect ratio) was $a/c = 0.9$. After the introduction of the pre-cracks, crack-healing tests under cyclic stress were carried out.

2.2. Crack-healing process

Table 1 shows the crack-healing process adopted in this study. The healing processes I to IV were selected in order to investigate the static fatigue strength of specimens crack-healed under cyclic stress at 900–1200 °C. In healing processes I to IV, pre-cracked specimens were crack-healed under a cyclic bending stress of σ_{max} = 210 MPa in air at 900, 1000, 1100, and 1200 °C, respectively. The healing processes V and VI were selected in order to investigate the effects of pre-oxidation on the static fatigue strength of the crack-healed specimens. In the healing processes V and VI, as-received smooth specimens were heat-treated in air at 1300 °C for 1h prior to the introduction of pre-cracks, followed by the crack-healing in the processes of I and II, respectively.

In order to perform crack-healing under cyclic bending stress, we used a hydraulically controlled testing machine equipped with an electric furnace. The loading system adopted involved three-point bending with a span of 16 mm, in which the pre-cracked zone was subjected to cyclic tensile stress. The waveform of cyclic bending stress was sinusoidal at a frequency of 5 Hz with a stress ratio ($R = \sigma_{min}/\sigma_{max}$) of 0.2. In order to avoid unexpected crack-healing without stress, we first applied a cyclic bending stress and then increased the furnace temperature at a rate of 10 °C/min and maintained that temperature for a given time. The specimens were furnace-cooled to room temperature. After the specimens had cooled completely, the cyclic bending stress was removed, and we then performed bending tests or static fatigue tests.

Table 1
Process of crack-healing

Healing process	Pre-heat-treatment		Healing condition		
	Temperature (°C)	Time (h)	Temperature (°C)	Time (h)	Cyclic stress, σ_{max} (MPa)
I	–	–	900	70	210
II	–	–	1000	15	210
III	–	–	1100	15	210
IV	–	–	1200	5	210
V	1300	1	900	70	210
VI	1300	1	1000	15	210

2.3. Bending tests and static fatigue tests

After crack-healing, bending tests or static fatigue tests were carried out at each healing temperature. Normally, three specimens were used to establish the monotonic bending strength, and two to five specimens were used to investigate the static fatigue strength. The loading system in monotonic bending tests and static fatigue tests involved three-point bending with a span of 16 mm. Monotonic bending tests were carried out using a universal testing machine equipped with an electric furnace. The cross-head speed for the monotonic bending tests was 0.5 mm/min. Static fatigue tests were carried out using a hydraulically controlled testing machine. The static fatigue limit (σ_{t0}) was defined as the maximum value of the applied stress under which specimens endured 5×10^5 s.

The fracture surfaces of crack-healed specimens were analyzed by using an optical microscope (OM) and a scanning electron microscope (SEM). The oxidation products were investigated by X-ray diffraction (XRD). The radiation used in XRD was Cu Kα at 40 kV accelerated voltage and 60 mA electric current.
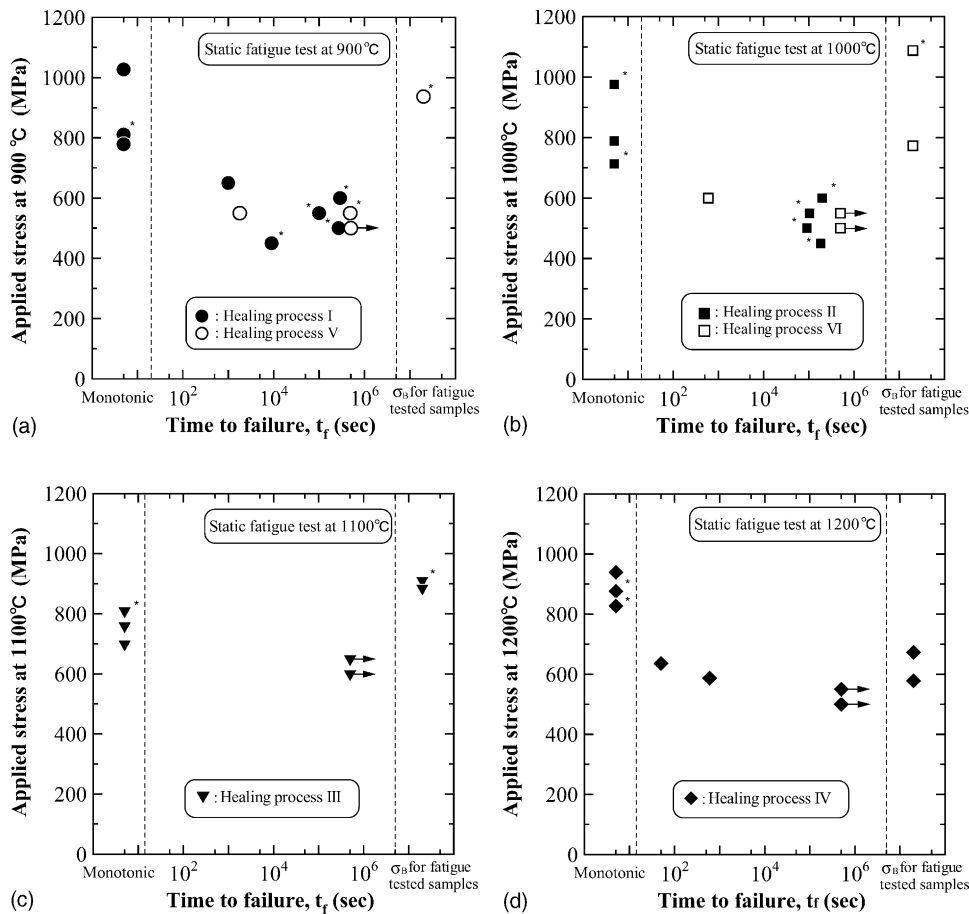

Fig. 1. Results of static fatigue tests for Si_3N_4/SiC specimens crack-healed under cyclic bending stress at (a) 900 °C, (b) 1000 °C, (c) 1100 °C, and (d) 1200 °C. Data marked with an asterisk indicate that fracture occurred outside of the crack-healed zone.

3. Test results and discussion

3.1. Static fatigue strength of the crack-healed specimen

Fig. 1a–d shows the results of static fatigue tests together with the monotonic bending tests at healing temperatures of 900, 1000, 1100, and 1200 °C, respectively. The pre-cracked specimens were crack-healed under a cyclic bending stress of $\sigma_{max} = 210$ MPa at each healing temperature. The results of the monotonic bending test are shown on the left-hand side of Fig. 1a–d. Asterisks show that the fracture occurred outside the crack-healed zone. The monotonic bending strength at each healing temperature is comparable to the room-temperature bending strength of smooth specimens, i.e., ~800 MPa. Thus, pre-cracks of 100 μm, which reduce the bending strength of smooth specimen by ~50%,[12] can be completely healed even under a cyclic stress of $\sigma_{max} = 210$ MPa. Moreover, the crack-healed zone has substantial strength at temperatures ranging from 900 to 1200 °C. The crack-healing of Si_3N_4/SiC occurs as a result of local oxidation at the crack surfaces; i.e., the surface cracks were filled and bonded by oxidation products, such as SiO_2 and $Y_2Si_2O_7$, formed on the crack surfaces.[8–12]

The specimens that did not fracture in the static fatigue tests up to 5×10^5 s are marked by arrow symbols (↪). The static fatigue limits (σ_{t0}) for the 900 °C (●) and 1000 °C (■) crack-healed specimens without pre-oxidation cannot be determined because all the specimens fractured during the static fatigue tests. It should be noted that most of the fractures occurred in the base material. On the other hand, for the specimens pre-oxidized in air at 1300 °C for 1 h prior to the pre-cracking and subsequent crack-healing at 900 °C (○) and 1000 °C (□), the static fatigue limits were successfully determined to be 500 and 550 MPa, respectively. Thus, the pre-oxidation improved the static fatigue behaviour of the crack-healed specimens. The mechanism governing this is discussed in the next section. The bending strength of the specimens that survived the static fatigue tests was also investigated at each healing temperature and is shown on the right-hand side of Fig. 1a and b. The fatigue-tested specimens exhibited bending strengths similar to those of the monotonically tested specimens, indicating that significant crack growth from the crack-healed zone or from internal defects did not occur.

For the specimens crack-healed at 1100 °C (▼) and 1200 °C (◆), the static fatigue limits at healing temperature were determined to be 650 and 550 MPa, respectively. These static fatigue limits were sufficiently high. As previously mentioned, the static fatigue limit for 900 and 1000 °C specimens (with no pre-oxidation) cannot be determined. Thus, the values of the static fatigue limit of the specimens tested at 1100 and 1200 °C were higher than those of specimens tested at 900 and 1000 °C.

3.2. Fracture surface of the crack-healed and static fatigue-tested specimens
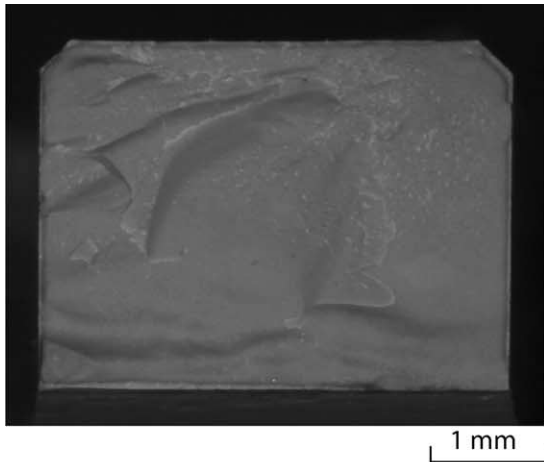
The fracture surfaces of crack-healed specimens were observed by using an optical microscope (OM). The results of OM observation revealed that the specimens which were crack-healed and static fatigue-tested at 900 and 1000 °C had thicker oxide layers than those tested at 1100 and 1200 °C. Fig. 2a–c shows examples of the macroscopic fracture surface of the specimens crack-healed under cyclic stress followed by static fatigue test. The specimen tested at 1000 °C, shown in Fig. 2a, has a thicker oxide layer than that tested at 1100 °C, shown in Fig. 2b, in spite of its shorter total oxidation time (t_{total}). Fig. 2c shows the macroscopic fracture surface of the static fatigue-tested specimens subjected to pre-oxidation prior to the pre-cracking and subsequent crack-healing at 1000 °C. For this specimen, the total exposure time at 1000 °C was $t_{total} = 5.54 \times 10^5$ s, which was longer than that of the specimen shown in Fig. 2a. However, the surface oxide layer of the specimen shown in Fig. 2c is thinner than that of Fig. 2a.

The fracture surfaces of the static fatigue-tested specimens were also observed by SEM. Fig. 3a shows the fracture surface within the thick oxide layer (20 μm) of the specimen crack-healed and static fatigue-tested at 1000 °C. This micrograph shows that many small pores indicated by arrows are formed in the oxide layer. It is considered that cracks nucleate from these pores and contribute to the specimens' fracture. Small pores are also observed in the oxide layer of the specimen tested at 900 °C. The mechanisms initiating the pores are discussed in the following section. Fig. 3b shows the fracture surface of the specimen static fatigue-tested at 1100 °C. A thin and coherent oxide layer is formed on the specimen surface. The thickness of the oxide layer is only about 2.5 μm. Small pores are not observed in these specimens. A similar fracture surface was observed in the specimen tested at 1200 °C. Fig. 3c shows the fracture surface of the specimen subjected to pre-oxidation prior to crack-healing and static fatigue testing at 900 °C. A thin and coherent oxide layer is formed by pre-oxidation at 1300 °C for 1 h. Similar fracture surfaces were observed in the specimen subjected to pre-oxidation followed by crack-healing and static fatigue testing at 1000 °C. Thus, the pre-oxidation increased the oxidation resistance at 900 and 1000 °C.

3.3. Oxidation mechanism

In order to investigate the oxidation products formed at 1000 °C, the surface oxide layer was characterized by the XRD. Specimens with or without pre-oxidation at 1300 °C for 1 h in air were subjected to heat-treatment at 1000 °C for 100 h in air.
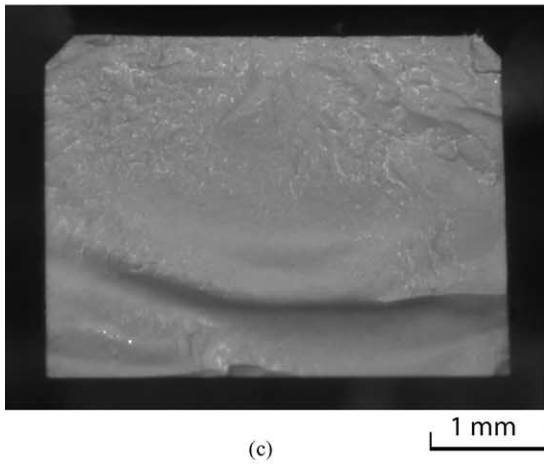
Fig. 4a shows the results of XRD analysis for the as-received smooth specimen. The XRD pattern for the oxidized specimen at 1000 °C for 100 h displays almost no X-ray peaks for SiC and $YSiO_2N$, as shown in Fig. 4b, indicating
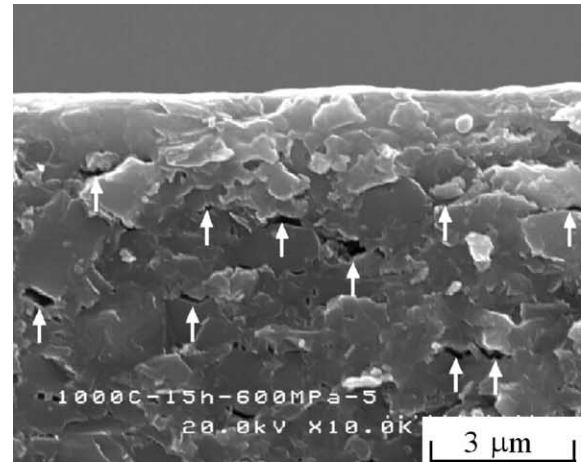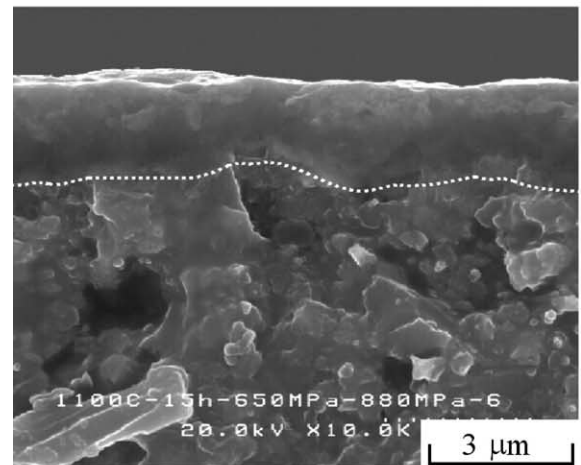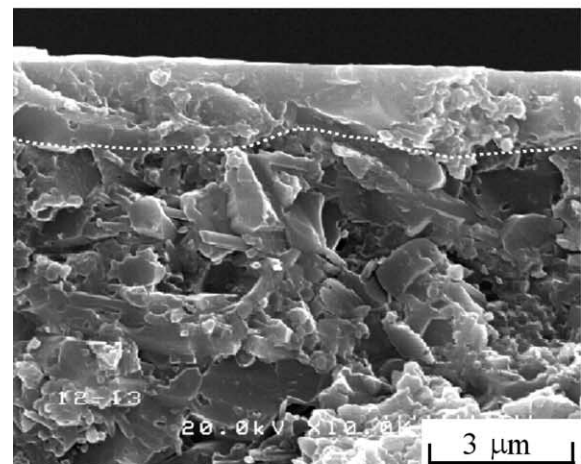
(a)
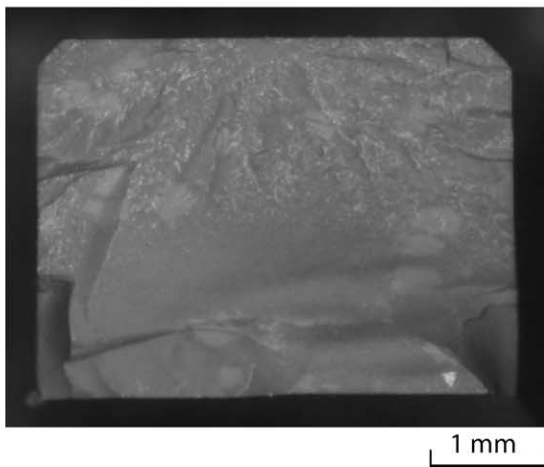
(b)

(c)

Fig. 2. Fracture surface of Si_3N_4/SiC specimens crack-healed under cyclic bending stress followed by static fatigue test: (a) healing process II, 1000 °C, $\sigma = 450$ MPa, $t_f = 1.84 \times 10^5$ s, $t_{total} = 2.38 \times 10^5$ s; (b) healing process IV, 1200 °C, $\sigma = 550$ MPa, ran out 5.0×10^5 s, $t_{total} = 5.2 \times 10^5$ s; (c) healing process VI, 1000 °C, $\sigma = 500$ MPa, ran out 5.0×10^5 s, $t_{total} = 5.54 \times 10^5$ s.

(a)

(b)

(c)

Fig. 3. SEM micrographs of fracture surface of Si_3N_4/SiC specimens crack-healed under cyclic bending stress followed by static fatigue test: (a) healing process II, 1000 °C, $\sigma = 600$ MPa, failed at $t_f = 2.0 \times 10^5$ s, $t_{total} = 2.54 \times 10^5$ s; (b) healing process III, 1100 °C, $\sigma = 650$ MPa, ran out 5.0×10^5 s, $t_{total} = 5.54 \times 10^5$ s; (c) healing process V, 900 °C, $\sigma = 550$ MPa, $t_f = 4.8 \times 10^5$ s, $t_{total} = 7.36 \times 10^5$ s.

that the grain boundary phases were preferentially oxidized. It is believed that extensive oxidation at lower temperature occurs due to the fact that the surface of the material is not completely covered by a protective SiO_2 layer.[19,21–23] Thus, grain boundary phases can be readily oxidized. If the SiC oxidized into SiO_2, a volume expansion of about 30% occurs.[22] In addition, the oxidation of $YSiO_2N$ also leads to a volume expansion of about 12%.[20] Such volume expansion at the grain-boundary phase causes internal stress and results in the nucleation of small pores as shown in Fig. 3a. Such pores can provide preferential paths for oxygen transport, thus producing an enhanced oxidation rate. However, at high temperatures such as 1100 and 1200 °C, these problems were not observed, probably due to the formation of a protective SiO_2 layer. Moreover, both the oxide layer and grain boundary phases were softened, allowing them to deform more easily to relieve oxidation-induced internal stress.[21,23]

For the specimen pre-oxidized at 1300 °C followed by oxidation at 1000 °C for 100 h, the XRD pattern is almost as same as that of the as-received specimen, as shown in Fig. 4b. The pre-oxidation in air at 1300 °C for 1 h formed a protective oxide layer on the specimen surface. The protective

layer was assumed to retard any further oxidation of the grain boundary phases at 900 and 1000 °C. Lange et al.,[19] Petal and Thompson,[21] and Yamashita et al.[22] also pointed out that such pre-oxidation effectively prevents extensive oxidation at 1000 °C of Si_3N_4 base ceramics.

We proposed a new methodology to guarantee the reliability of ceramic components.[26] This new concept consisted of the following three stages: (a) crack-healing under optimized conditions (in air at 1300 °C for 1 h for the Si_3N_4/SiC), (b) proof testing to eliminate ceramic components having harmful internal defects, and (c) in situ (in-service) crack-healing.[26] The benefits of crack-healing in air at 1300 °C for 1 h (stage (a)) are not only to heal the pre-existing small surface cracks generated by surface finishing but also to form a thin protective layer of SiO_2 which retard further oxidation and led to a significant improvement of static fatigue strength at 900 and 1000 °C.

4. Conclusions

Si_3N_4/SiC composite ceramics were hot-pressed in order to investigate their crack-healing behaviour and resultant static fatigue strength. The specimens having pre-cracks were crack-healed under a cyclic bending stress of 210 MPa in air at 900, 1000, 1100, and 1200 °C. The bending strength and static fatigue strength of the crack-healed specimens were systematically investigated at each healing temperature. The results obtained in this study are as follows:

1. The specimens crack-healed and static fatigue-tested at 1100 and 1200 °C showed sufficiently high static fatigue limits, i.e., 650 and 550 MPa, respectively. However, the specimens crack-healed and static fatigue-tested at 900 and 1000 °C showed lower fatigue strength than did those tested at 1100 and 1200 °C.
2. When the specimens were crack-healed and static fatigue-tested at 1100 and 1200 °C, the specimen surface became covered by a protective oxide layer. On the other hand, when the specimens were crack-healed and static fatigue-tested at 900 and 1000 °C, their surfaces were covered by a thick oxide layer with small pores. Thus, the oxidation of the base material had large effects on the static fatigue strength.
3. It was found that if the specimens were pre-oxidized in air at 1300 °C, the surface became covered by a protective oxide layer. This protective layer prevented further oxidation and led to a significant improvement of static fatigue strength at 900 and 1000 °C.
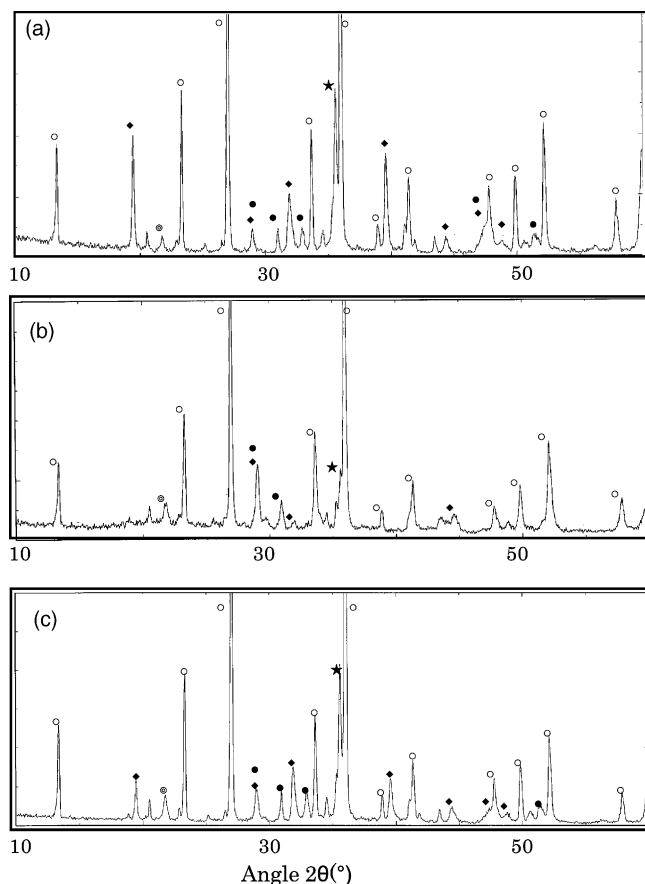

Fig. 4. XRD profiles of the surface of Si_3N_4/SiC specimens before and after oxidation in air: (a) as received; (b) 1000 °C × 100 h; (c) pre-oxidized at 1300 °C for 1 h followed by oxidation at 1000 °C × 100 h. (○) β-Si_3N_4, (★) SiC, (◉) SiO_2, (◆) $YSiO_2N$, (●) $Y_{20}N_4Si_{12}O_{48}$.

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