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In situ crack-healing behavior of Al_2O_3/SiC composite ceramics under cyclic-fatigue strength ☆

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ABSTRACT

Alumina/silicon carbide (Al_2O_3/SiC) composite ceramics were sintered and subjected to three-point bending. A semicircular surface crack 50–320 μm in diameter was made on each sample. Crack-healing behavior was systematically studied, as a function of cyclic-fatigue strength, crack-healing temperature, healing time, and crack size, and the bending strength of the crack-healed sample from room temperature to 1500 °C were investigated. Four main conclusions were drawn from the present study.

(1) Crack-healed Al_2O_3/SiC sample exhibited very high cyclic-fatigue limit of 700 MPa.
(2) Al_2O_3/SiC composite ceramics have the ability to heal after cracking, from 1000–1400 °C.
(3) The composite material can completely heal a 300 μm diameter semi-elliptical crack (aspect ratio 0.8–0.9).
(4) The heat-resistance limit for monotonic loading of the crack-healed sample goes up to 1300 °C, and ~ 68% of the samples fracture, outside the crack-healed zone in the testing-temperature range 600–1300 °C.

From these test results, it can be concluded that Al_2O_3/SiC has an excellent crack-healing ability and that crack healing is a desirable technology for the structural integrity of Al_2O_3/SiC.

1. Introduction

Alumina (Al_2O_3) is a very popular ceramic used in various fields. However, it has three weak points: low bending strength (σ_B) (~400 MPa), low fracture toughness (~3 $MPa \cdot m^{1/2}$), and a low heat-resistance limit for bending strength (~900 °C). These weaknesses restrict the application of Al_2O_3 for important components. Niihara [1] has proposed a new concept related to nanocomposite ceramics. Some nanocomposite Al_2O_3/SiC exhibit excellent mechanical properties [1] (bending strength of ~1500 MPa, fracture toughness of 5 $MPa \cdot m^{1/2}$ and a heat-resistance limit of ~1200 °C). However, the fracture toughness is low, thus the Al_2O_3/SiC is very sensitive to flaws, such as cracks and pores.

Al_2O_3 and Al_2O_3/SiC composites have very interesting crack-healing ability. Applying the high crack-healing ability of ceramics [2–4] to structural components for engineering use could result in great benefits, such as the increased reliability of structural ceramic components and the reduced inspection, machining, and polishing costs of ceramic components. However, when this healing ability is applied in structural engineering, many problems must be overcome, including the effect of chemical composition on the crack-healing ability [3], the effect of healing conditions on the strength of the healed zone [2], the maximum crack size that can be completely healed [5], knowledge of the high-temperature strength of crack-healed zones [2,3], the assessment of the cyclic-fatigue and static-fatigue strengths of a crack-healed ceramic component [2–7], and crack-healing behavior during service [2,5]. Several aspects of crack healing have been studied on Si_3N_4 [2,6,7] and mullite [5]. However, few studies have been made on the crack-healing behavior of Al_2O_3 and Al_2O_3/SiC composites. This paper reports on the crack-healing behavior and strength of crack-healed Al_2O_3/SiC composites at room temperature and high temperatures.

2. Sample materials and experimental method

The sample material is Al_2O_3/SiC composite ceramics (alumina composite material) and was sintered with monolithic alumina (average particle sizes 4.1 μm) purchased from the market [8]. The alumina powders, average particle sizes 0.5 μm and 99.99% purity (Sumitomo Chemical Ltd. products), are used for alumina composite material. The SiC powder has an average particle size of 0.27 μm and 98% purity (Ibiden Ltd. products). The dosage of the SiC particle is 15 vol.%.

To this mixture, alcohol was added and blended thoroughly for 48 h with a nylon ball. After that, the mixture was placed into an evaporator to extract the solvent, and then put into vacuum desiccators to produce a dry powder mixture. The mixture was

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Nomenclature

$2c$	diameter semi-elliptical crack
N_f	number of cycles to fracture
P	load by Vicker's indentation
R	stress ratio
T_R	transition temperature
$\alpha = \sigma_{t0}/\sigma_B$	a ratio of bending strength (σ_B) and the static-fatigue limit (σ_{t0})
σ_B	bending strength
σ_{t0}	static-fatigue limit
σ_{f0}	cyclic-fatigue limit

subsequently hot-pressed at sintering temperature 1600 °C and 35 MPa for 4 h inside the 800 kPa nitrogen gas.

The goal of sintering of Al_2O_3/SiC composite material is based on the following theory:

(a) Up to 15% of the dispersion of fine SiC particles, the excellent crack-healing ability is obvious.
(b) Through the dispersion of SiC particles, the strength is improved by the inhibition of the grain growth in sintering process.
(c) Through the dispersion of nano size particle of SiC, the heat-resistance limit is improved in comparison with the monolithic alumina.

The dimension of sintered plate is 5×90×90 mm. The sintered plates were cut into specimens with a length, width, and height of 3×4×22 mm. The surface of the specimens was carefully ground and polished before the test, in accordance with the Japanese Industrial Standard for bending strength test [9].

The effect of crack healing was investigated using specimens whose defects were created by Vicker's indentation method. Loading P= 19.61 N enabled us to create semi-circular cracks about 100 μm in diameter (aspect ratio 0.9).

To determine maximum crack size that can be healed, several crack sizes were investigated. Loading of 9.8 N to 68.6 N enabled us to generate semicircular cracks 50–320 μm in diameter.

The pre-cracked specimens were subjected to thermal treatment at different temperatures to examine the effect of healing conditions on the fracture stress at room temperature. The samples for room-temperature strength tests were healed in air at temperatures ranging from 900 °C to 1400 °C for pre-cracked specimens, and 1300 °C for smooth specimens. The samples were heated at a rate of 10 °C/min, and the time-of-exposure was 1, 10 and, 300 h.

The strength of the specimens was measured using a three-point loading system (crosshead speed = 0.5 mm/min) at room temperatures. The specimens were heated to testing temperature at 10 K/min rate, and last for 15 min. The bending tests at elevated temperatures were conducted at temperatures between 400 °C and 1400 °C. The cyclic-fatigue tests were conducted at 1100 °C, stress ratio (R) 0.2, and sine wave frequency of 5 Hz.

Table 1
Summary of monolithic alumina and alumina composite material experiment results (Section 3.1).

Sample	Symbols	Bending strength (σ_B) MPa	Cyclic-fatigue limit (σ_{t0}) MPa
Monolithic alumina, base specimen (base material, bending span 16 mm)	○	400	None
Alumina composite, base specimen	●	850	700
Monolithic alumina, crack-healed specimen (healing at 1450 °C for 1 h)	▽	370	210
Alumina composite, crack-healed specimen (healing at 1300 °C for 1 h)	▼	850	700

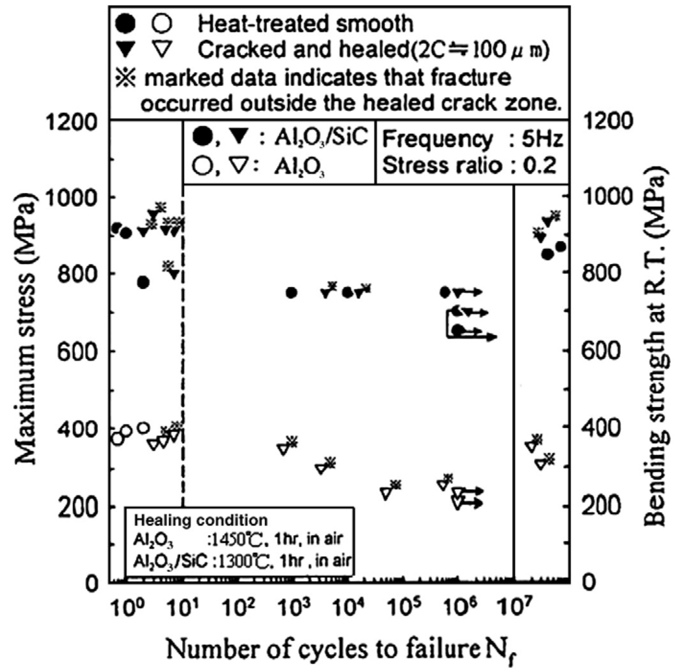

Fig. 1. Relationship between maximum applied stress and time to failure of Al_2O_3/SiC and monolithic Al_2O_3 subjected to crack-healing treatment.

3. Results and discussion

3.1. Cyclic-fatigue strength characteristics of monolithic alumina and alumina composite material

It is known that time-dependent phenomenon of fatigue happens in many structural ceramics [10], instead of cyclic-load dependence phenomenon occurring. The time-dependent phenomenon is especially noticeable in the cases of silicon nitride [11] and mullite in the crack-healed zone, but the effect of cyclic was clearly accepted in the silicon carbide. Even now, no study on the cyclic effect in the crack-healed zone of the alumina is found.

The summary of monolithic alumina and alumina composite material experiment results is listed in Table 1.

In this paper, the cyclic-fatigue strength of monolithic alumina and alumina composite ceramics crack-healed specimens is examined, and the result is shown in Fig. 1. From the left field of symbols ○ and ▽ show the base specimen and crack-healed specimen under 1 h crack-healed time at 1450 °C for monolithic alumina. Moreover, symbols ● and ▼ show the base specimen and crack-healed specimen under 1 h crack-healed time at 1300 °C for alumina composite material. In this study, the cyclic-fatigue limit (σ_{f0}) is defined as the non-fractured stress, which is cyclic stress after 10^6 cycles.

In the case of the monolithic alumina of crack-healed specimen, there are four specimens broken from outside the crack-healed zone. In the static-fatigue and cyclic-fatigue test of monolithic alumina (1723 K crack-healed specimen), all specimens had broken like the previous from the outside of crack-healed zone. Subsequently, we will discuss two reasons to examine this cause.

(1) The defect of pore (hole) exists in great numbers on the surface of the monolithic alumina. The flat part with the legible crack was selected, when the pre-cracked was introduced.
(2) 1723 K is the possible temperature range for sintering of alumina and the pre-cracked specimen can be healed perfectly.