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Static and cyclic fatigue behaviour of crack-healed Si_3N_4/SiC composite ceramics

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Abstract

Si_3N_4/SiC composite ceramics were sintered in order to investigate their fatigue strength behavior after crack healing, Y_2O_3 and Al_2O_3 power was added as sintering additives to enhance it's sintering property. Three-point bending specimen (sized 3×4×40 mm) was hewed out according to JIS standard from sintered compact obtained. About 100 or 200 μm semi-circular surface cracks were made on the center of the tension surface of the three-point bending specimen using Vickers indenter. After the crack-healing processing of 1200 and 1300°C, 1 h, in air, cyclic and static fatigue strength behavior of these crack-healed specimen were determined systematically at room temperature and high temperature of 800, 1000, 1200 and 1300°C. The results show that Si_3N_4/SiC composite ceramics possess remarkable crack-healing ability, and crack-healed specimens showed similar cyclic and static fatigue strength behavior as smooth specimens at high temperature, not only at room temperature. Crack-healed zones had sufficient fatigue strength even though at high temperature environment, and most fractures occurred outside the pre-cracked zone in those crack-healed specimens.

Keywords: Crack-healing; Fatigue; Mechanical properties; Si_3N_4/SiC; Strength

1. Introduction

Generally, structure engineering ceramics are widely used in all kinds of engineering fields for their advantages of heatproof property, wear resistance and corrosion resistance, such as turbo-charger, ceramic gas turbine, auto heat engine, and so on. But it is still a difficulty that ceramics material were utilized in all kinds of fields more efficiently, because of their machining performance and high cost due to brittleness and low fracture toughness compared with metal materials. Here, some engineering ceramics possessing the ability of crack healing were discovered.[1–7] If this ability could be used in engineering ceramics components, their reliability will be increased and their lifetime will also be prolonged, correspondingly.

We have studied the static strength behavior of crack-healed mullite/SiC ceramics,[8] Si_3N_4/SiC ceramics[9–11,13,15] and SiC ceramics.[12] We have also verified that the crack-healing ability of Si_3N_4/SiC composite ceramics is better than that of monolithic Si_3N_4 ceramics.[11] Furthermore, we have been found that the Si_3N_4/SiC with Y_2O_3 as sintering additive has a crack healing ability under loading.[14]

In actual engineering utilization, ceramic components are often operating in a variable load environment, continuously. Consequently, it is very important to make clear the fatigue strength behavior of crack-healed ceramics. This paper focuses on investigating the cyclic fatigue behavior at room and elevated temperature, and the static fatigue behavior at elevated temperature of crack-healed Si_3N_4/SiC composite ceramics.

2. Material, specimen and test method

In accordance with research findings heretofore, Si_3N_4/SiC composite ceramics with Y_2O_3 powder added as sintering additive possess the most remarkable crack-healing ability, but it also have a low strength. If Al_2O_3 powder was also added as sintering additive at the same time with Y_2O_3 powder, denser sintering compact could be obtained, and the strength would be improved.

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However, in the case that Al_2O_3 powder was added as a sintering additive, the grain boundary phase of the silicon nitride would be non-crystalline, and there is the possibility of a reduction of the high temperature strength. In this experiment, these two kinds of Si_3N_4/SiC composites, which have different sintering additives, were made in order to investigate their fatigue strength behavior after being crack-healed.

The silicon nitride powder used in this study has the following properties: mean particle size = 0.2 μm, the volume ratio of α-Si_3N_4 is above 95% and the rest is β-Si_3N_4. The SiC powder used has a 0.27 μm mean particle size, and the quantity of SiC powder added is 20 wt.% in contrast to Si_3N_4 powder.

The samples were made with a mixture of silicon nitride, SiC powder and 8 wt.% Y_2O_3 or 5 wt.% Y_2O_3 and 3 wt.% Al_2O_3 as sintering additive powder. To this mixture, alcohol was added and blended completely for 48 h. with a nylon ball, after that the mixture was placed in an evaporator to extract the solvent and then in a vacuum desiccator to produce a dry powder mixture. The mixture was subsequently hot-pressed at 1850°C and pressure of 35 MPa for 2 h in nitrogen gas, and then 5×90×90 mm sintered plate was obtained; SN-SC-Y5A3 (SiC: 20 wt.%, Y_2O_3: 5 wt.%, Al_2O_3: 3 wt.%), and SN-SC-Y8(A),(B),(C) (SiC: 20 wt.%, Y_2O_3: 8 wt.%). Where the (A),(B) and (C) indicates the specimen that was made from each different batch.

The sintered plate was cut into specimens with height, width and length dimensions of 3×4×40 mm. The surface of the specimen was mirror finished by lapping after surface grinding by diamond grindstone of #600, and surface roughness of the specimen, R_{max}, was 0.05 μm. The shape and dimensions of the specimen are shown in Fig. 1.

To evaluate the bending strength, a three-point bend test was carried out at a temperature ranging from room temperature to 1400°C in accordance with JIS 1061.[16] The span length and cross-head-speed were 30 and 0.5 mm/min, respectively.

A surface crack was made at the center of the tension surface of the specimen with Vickers indenter in order to investigate strength of crack-healed zone. Vickers load was 19.6 and 49 N, and the size of surface crack was about 100 and 200 μm, respectively. The shape of crack with diameter of about 100 μm is shown in Fig. 2.

The pre-cracked specimens were heated at a rate of 10°C/min, and the time of exposure was 1 h for all specimens. Cooling was spontaneous in the furnace. The healing temperature is 1300°C for the SN-SC-Y8 specimen and 1200°C for the SN-SC-Y5A3 specimen.

The static fatigue tests were performed at 800 and 1000°C in air, and the loading was also three-point bending. The tests were stopped if rupture had not occurred by 10^6 s.

Cyclic fatigue tests were made at temperature ranging from room temperature to 1300°C in air, at a stress ratio $R = 0.2$ and using sine wave with a frequency of 5 Hz.

The fracture surfaces were analyzed by optical and scanning electron microscopy (SEM) techniques.

3. Test result and discussion

3.1. Bending strength of crack-healed specimen at high temperature

Test temperature dependence of crack-healed specimen's bending strength is shown in Fig. 3, and three pieces of specimen were used in each condition in the test. In the case of SN-SC-Y8(A) signified by symbol ○, the bending strength shows a constant value of 800 MPa up to 1000°C and above 1000°C, about 100 MPa lower bending strength showed than that at 1000°C. Moreover, most of the specimens failed outside the crack-healed zone up to 1300°C similar to the crack path in Fig. 11(a). In SN-SC-Y8(B) signified by symbol □, high bending strength of 850 MPa has been shown in 1300°C

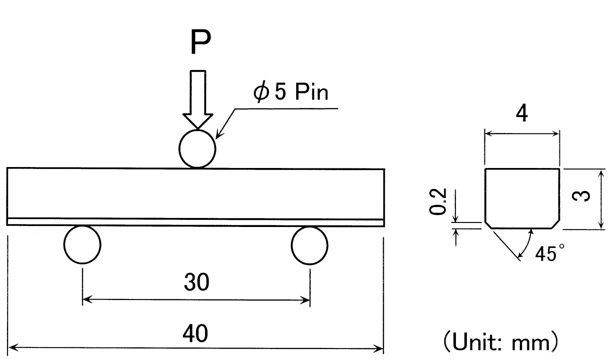

Fig. 1. Three-point bend specimen and loading system.

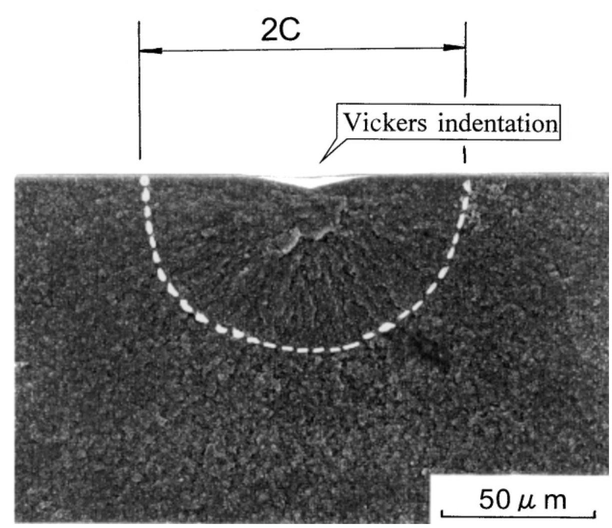

Fig. 2. Indented crack shape and fracture surface.

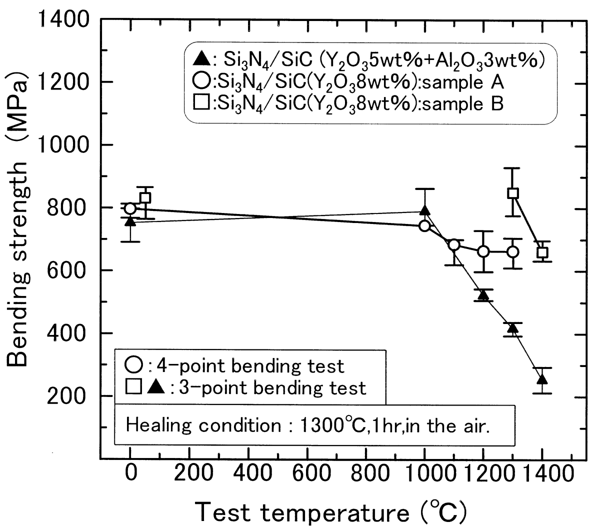

Fig. 3. Effect of test temperature on the bending strength of crack-healed specimen.

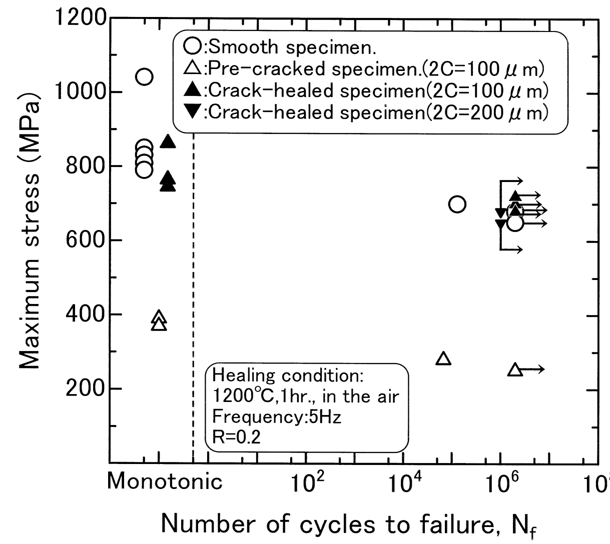

Fig. 4. Cyclic fatigue test result of SN-SC-Y5A3 at room temperature.

compared to SN-SC-Y8(A), and all of the specimens ruptured from the outside of the crack-healed zone. However, bending strength was reduced by about 200 MPa at 1400°C, and all the specimens ruptured from the pre-crack area. Therefore, the heat resisting limit temperature, in terms of the monotonic load of crack-healed SN-SC-Y8 material is about 1300°C the same as the parent material, and the possession of adequate heat resistance could be concluded. On the contrary, in the case that the composite SN-SC-Y5A3 containing Al_2O_3 signified by symbol ▲, the bending strength is almost same up to 1000°C and the value is about 800 MPa. Above 1000°C, the bending strength decreased dramatically with an increase in the test temperature. Moreover, all specimens ruptured from the crack-healed zone. From these results, it could be concluded that the heat resisting limit temperature in monotonic load is about 1000°C.

3.2. *Cyclic fatigue strength behavior at room temperature*

The cyclic fatigue test result in SN-SC-Y5A3 specimens to which Y_2O_3 and Al_2O_3 was added as sintering additives are shown in Fig. 4 by the S–N curve as a correlation with maximum bending stress (σ_{max}) and number of cycles to failure (N_f). Three kinds of smooth, pre-cracked and crack-healed specimens were used for the cyclic fatigue test. Three-point bending strength of smooth, pre-cracked and crack-healed specimen was shown on the left side of dashed line, which signified by symbols ○, △ and ▲, respectively. A mean bending strength of the smooth specimen is about 860 MPa. The bending strength decreased to about 380 MPa after cracking. By healing treatment, however, the bending strength was recovered to a level similar to the smooth specimen, which is about 800 MPa of the mean fracture stress. The cyclic fatigue tests were stopped at $N=2\times10^6$ cycles. The specimens which did not fracture in the test are marked by an arrow symbol (→). The maximum stress at which a specimen did not fracture up to at $N = 2 \times 10^6$ cycles is denoted as σ_{fc}. The σ_{fc} for smooth specimen (○) is about 680 MPa, and is about 250 MPa for the pre-crack specimens. In crack-healed specimens, two kinds of specimens with about 100 and 200 μm in crack diameter were tested in order to investigate the effect of pre-crack size on cyclic fatigue strength behavior. The σ_{fc} for crack-healed specimen with pre-crack size of 100 μm (▲) is about 720 MPa, and is about 680 MPa for crack-healed specimen with 200 μm crack (▼), which is 3 times approximately as large as that of the pre-cracked specimens. The value of crack healed specimen's σ_{fc} showed nearly equal to that of the smooth specimen. Also, the value of σ_{fc} is not dependent on pre-crack size by 200 μm.

The cyclic fatigue test result at room temperature for SN-SC-Y8 specimens, to which only Y_2O_3 was added as sintering additive was shown in Fig. 5. Symbols ○, △ and ▲ indicate data for smooth, pre-cracked and crack-healed specimens, respectively. The σ_{fc} of the smooth specimen is about 670 MPa, and that of the pre-crack specimen ($2C=100$ μm) is about 200 MPa which shows about 70% degradation in strength in comparison with smooth specimen. After healing treatment, however, the specimen's σ_{fc} got to about 735 MPa, which shows the same level as that of the smooth specimen, even a little high. Furthermore, in the case of two specimens which were fractured during the test, marked by (※) in Fig. 5, fracture occurred from outside the crack-healed zone. From these results, it could be concluded that the crack-healed zone is not sensitive to cyclic fatigue at room temperature.

On the other hand, the σ_{fc} of crack-healed specimen (2C = 200 μm) is about 650 MPa, which is a little degrading compared to that of the 100 μm crack-healed specimen. But that still showed nearly equal value to 670 MPa of the cyclic fatigue limit (σ_{fc}) of smooth specimens. A reason of the degradation is resulted in stress concentration due to Vickers indentation which is located on the specimen surface even though after crack-healing treatment (see Fig. 6). The indentation size is about 90 μm and is larger than that of the 100 μm crack (about 50 μm). Two specimens fractured in the test, and both of them failed from the crack-healed zone. However, the strength shows high values in comparison with

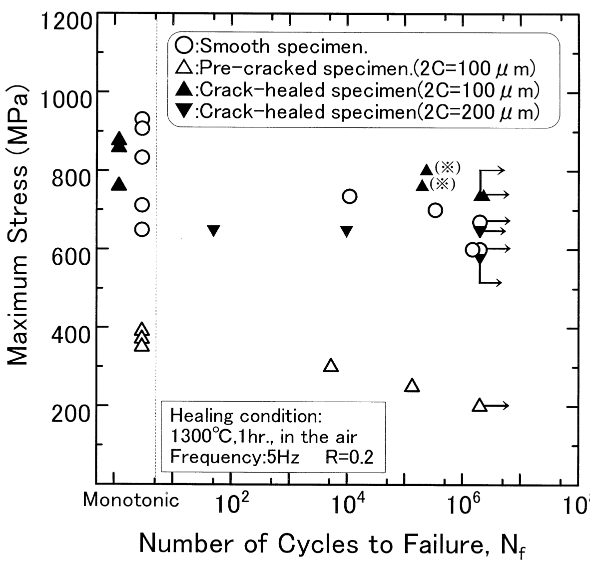

Fig. 5. Cyclic fatigue test result of SN-SC-Y8(B) at room temperature (※ marked data indicates that fracture did not occur at site of Vickers indentation).

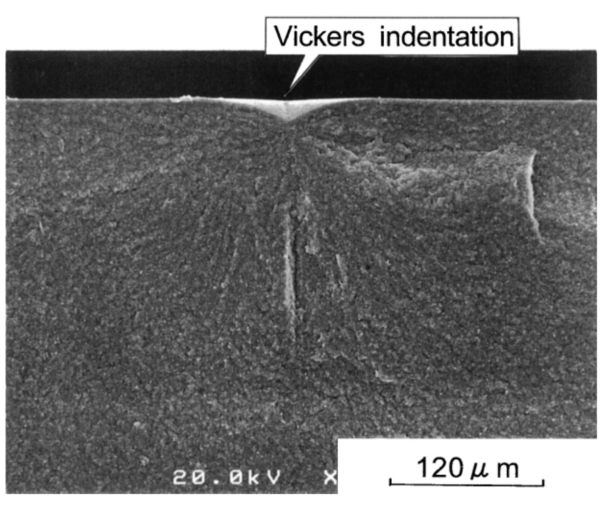

Fig. 6. SEM photograph of fracture surface of the SN-SC-Y8 crack-healed specimen which failed under cyclic load (σ_{max} = 650 MPa, N_f = 9810 cycles, $2C$ = 200 μm).

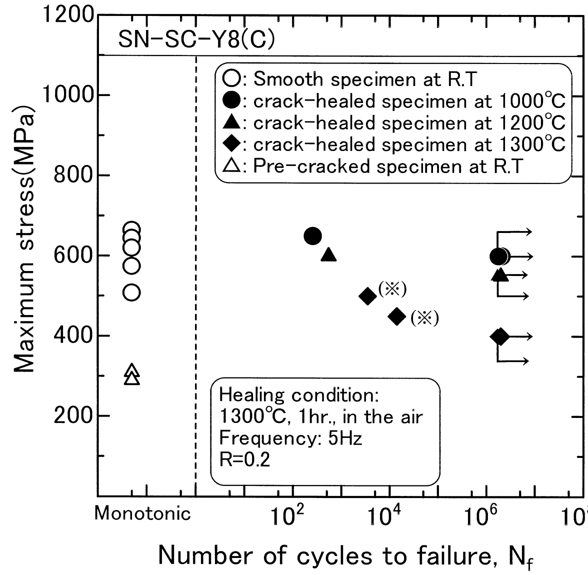

Fig. 7. Cyclic fatigue test result of SN-SC-Y8(C) at high temperature.

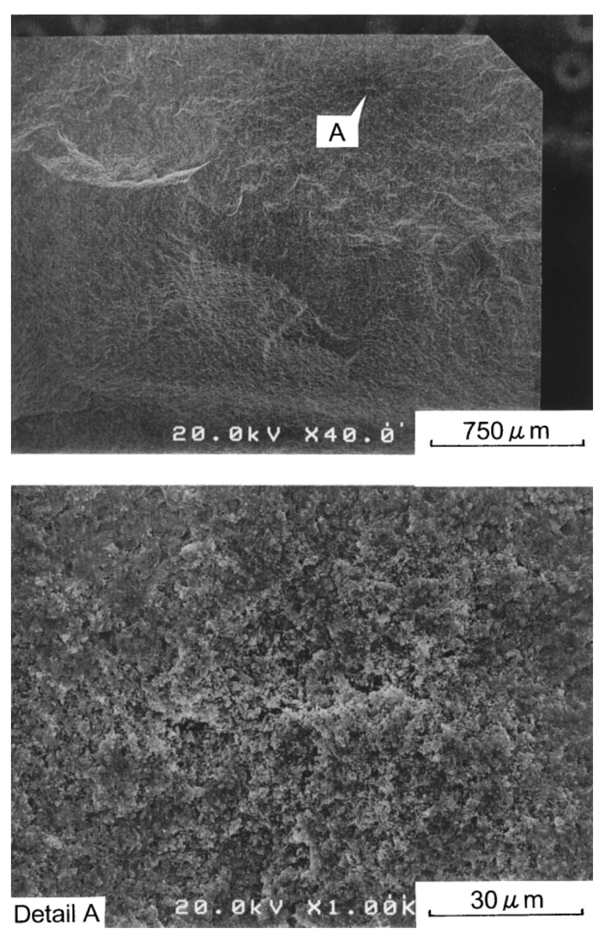

Fig. 8. SEM photographs of the fracture surface of the SN-SC-Y8(C) smooth specimen.

the crack-healed specimen. A SEM photograph of the fracture surface of one is shown in Fig. 6.

From that mentioned above, in regard to the SN-SC-Y8 specimen, it could be concluded that even a 200 μm crack could be healed, and the crack-healed zone possess enough strength under cyclic fatigue loading at room temperature.

4. Fatigue strength at high temperature

High-temperature cyclic fatigue strength of crack-healed specimens was investigated using SN-SC-Y8(C) material, and the test result is shown in Fig. 7. In Fig. 7, three-point bending strength at room temperature of smooth and pre-cracked specimen of SN-SC-Y8(C) was shown on the left side of dashed line, which signified by symbol ○ and △, respectively. The bending strength of the smooth and pre-cracked specimen is about 600 and 300 MPa, respectively. The values are smaller than that of SN-SC-Y8 (B). Using SEM, the fracture surface of specimen fractured in bend test was observed, and its photograph was shown in Fig. 8. From this SEM photograph, it can be seen that fracture occurred from a large internal processing flaw in the specimen due to sintering faults. It seems that abounding existence of these internal flaw is the reason that three-point bending strength is decreased. By the way, there was no effective difference between SN-SC-Y8(C) and (B) in strength on crack-healing behavior. The cycle fatigue tests were stopped at $N_f = 2\times10^6$ cycles. The specimens which did not fracture in the test are marked by an arrow symbol (→) in the figure. The σ_{fc} decreased with increasing test temperature; about 600 MPa at 1000°C (●), 550 MPa at 1200°C (▲) and about 400 MPa at 1300°C (◆). However, crack-healed SN-SC-Y8(C) possessed enough cyclic fatigue strength up to 1200°C in comparison with smooth specimen's bending strength at R.T and not showed any degradation in strength under cyclic fatigue. On the contrary, The σ_{fc} at 1300°C is as low as 400 MPa and, however, all specimens which were failed during the test had been fractured from outside the crack-healed zone. A SEM photograph of the fracture

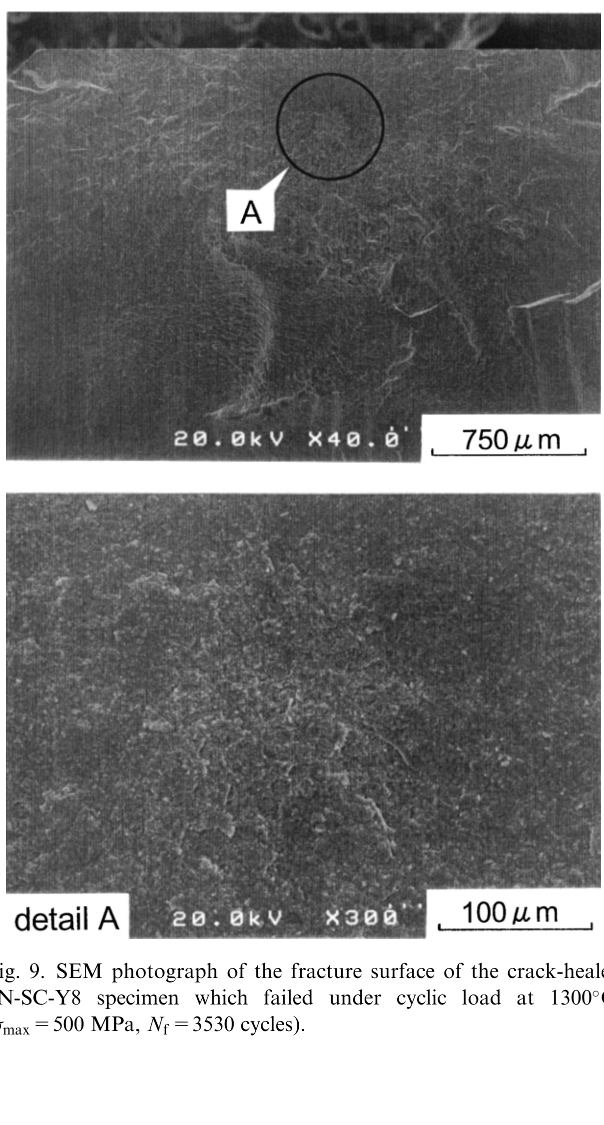

Fig. 9. SEM photograph of the fracture surface of the crack-healed SN-SC-Y8 specimen which failed under cyclic load at 1300°C. ($\sigma_{max} = 500$ MPa, $N_f = 3530$ cycles).

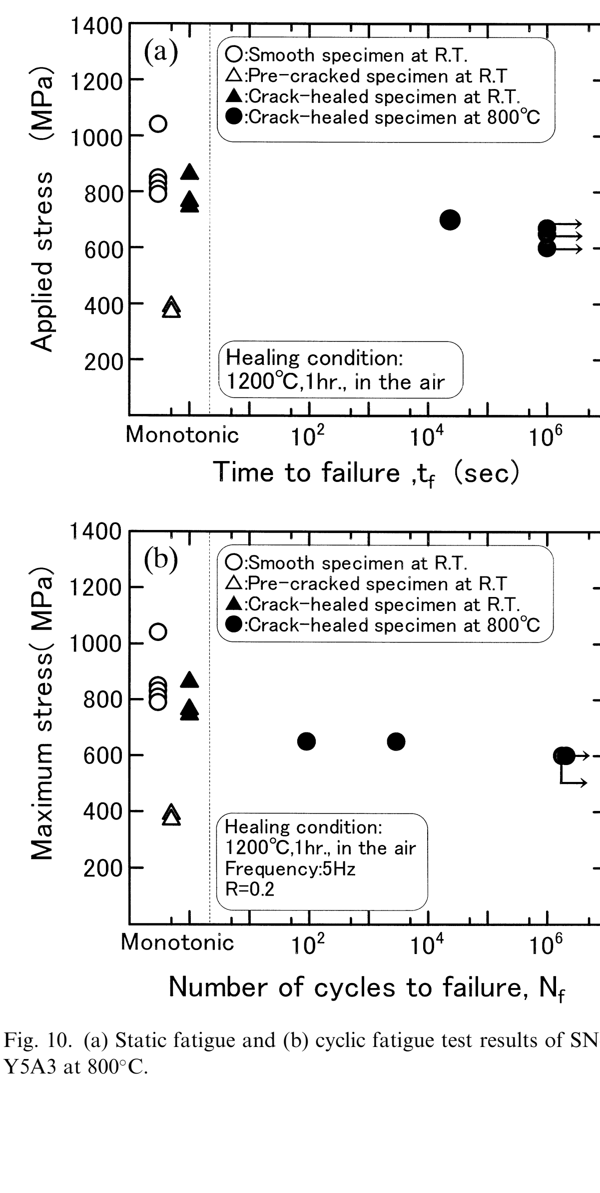

Fig. 10. (a) Static fatigue and (b) cyclic fatigue test results of SN-SC-Y5A3 at 800°C.

surface is shown in Fig. 9. The fracture initiated from internal defect in the specimen not from the crack-healed zone. From these results, it could be concluded that the heat resistance limit temperature of crack-healed SN-SC-Y8 material was 1200°C, but the material is no more sensitive to cyclic fatigue up to 1300°C.

High-temperature fatigue tests were carried out for the SN-SC-Y5A3 specimen under an ambience of 800°C, which is the temperature that an auto-engine usually works at. The test result is shown in Fig. 10.

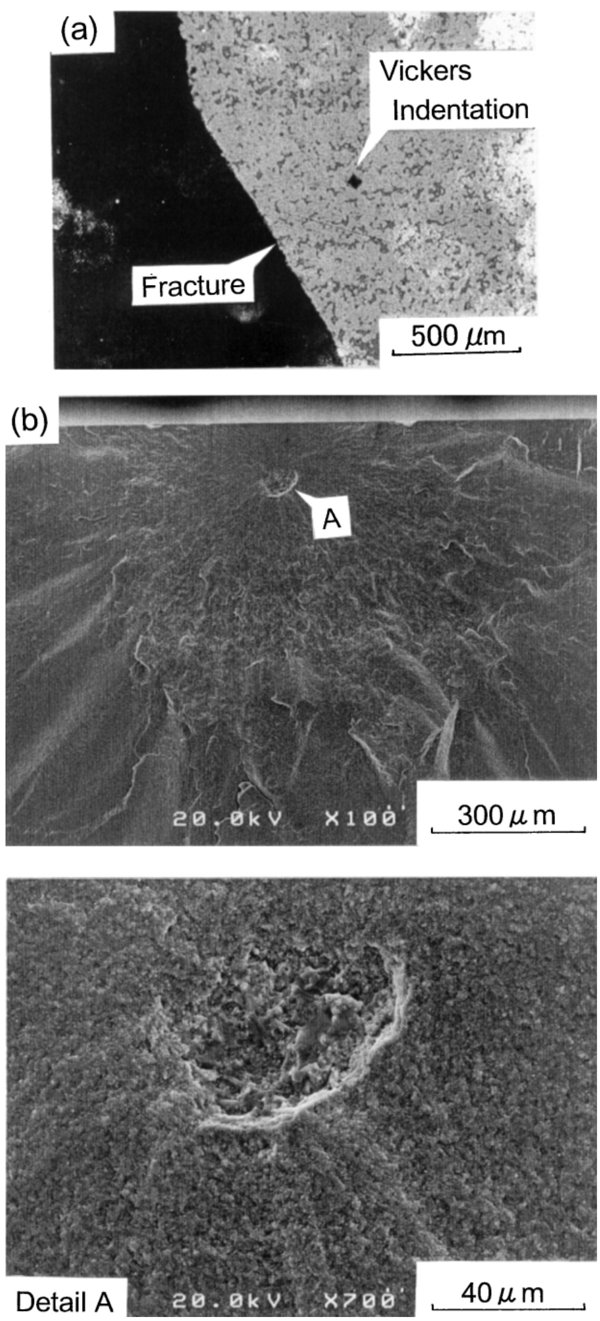

Fig. 11. (a) Fracture patterns and (b) fracture initiation site of the crack-healed SN-SC-Y5A3 specimen which survived in the cyclic fatigue test and then was bend-tested at RT after the cyclic fatigue test of $N_f = 2\times10^6$ cycles, $\sigma_{max} = 600$ MPa at 800°C.

Fig. 10(a) shows the result of the static fatigue test at 800°C, and Fig. 10(b) that of the cyclic fatigue test. Three-point bending strength at room temperature under static loads of smooth, pre-cracked and crack-healed specimen was shown on the left side of dashed line, which signified by symbols ○, △ and ▲, respectively. All of the pre-crack lengths are about 100 μm. The static fatigue tests were stopped at $t_f = 1\times10^6$ s. The specimens which did not fracture in the test are marked by an arrow symbol (→) in the figure. The applied stress which a specimen did not fracture up to $t_f = 1\times10^6$ s is denoted as σ_{fs}. The σ_{fs} of the SN-SC-Y5A3 specimen at 800°C after crack-healing is about 670 MPa. And, the cyclic fatigue limit (σ_{fc}) of the SN-SC-Y5A3 specimen at 800°C after crack-healing is about 600 MPa. There were two pieces of specimens, which were marked by an arrow symbol (→), which did not fracture until $N = 2 \times 10^6$ cycles under maximum stress of 600 MPa. Using the specimens, a bending test at RT was carried out and their bending strength is 1016 and 841 MPa, respectively. SEM photographs of fracture pattern and fracture surface are shown in Fig. 11. The fracture occurred from the outside of crack-healed zone, as shown in Fig. 11(a). Note, the fracture surface of this specimen, and it shown that the fracture initiation site is at the internal flaw in the material, as shown in Fig. 11(b). From these results, it is concluded that the crack-healed zone has enough strength in both cyclic and static fatigue up to 800°C.

The static fatigue strength behavior at 1000°C was investigated, and the test result is shown in Fig. 12. Three-point bending strength at room temperature of smooth, as-cracked specimens and crack-healed specimens, which are signified by symbols ○, △ and ▲, are

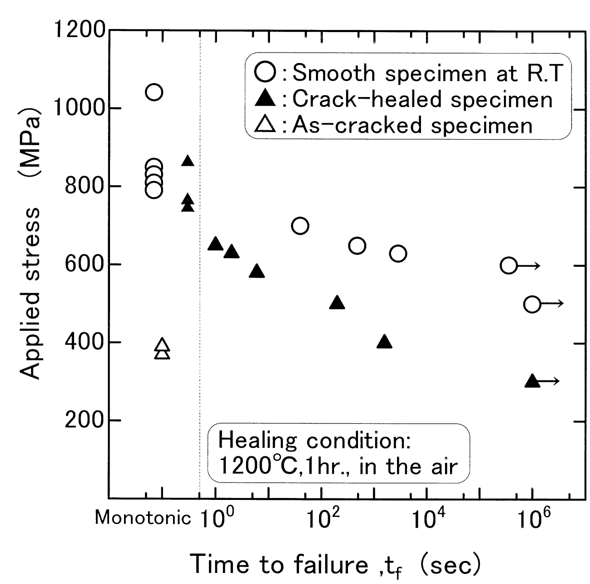

Fig. 12. Static fatigue test results of smooth and crack-healed SN-SC-Y5A3 specimens at 1000°C.

shown on the left side of the dashed line in the figure. From Fig. 6, it can be seen that the static fatigue limit (σ_{fs}) of smooth specimens at 1000°C is about 600 MPa, and the σ_{fs} of the crack-healed specimen is about 300 MPa, which shows a half value of that of smooth specimens. Furthermore all the specimens that failed during the static fatigue test fractured from the crack-healed zone. Consequently, the crack-healed zone is sensitive to static fatigue at 1000°C and the heat resisting limit temperature on static fatigue of SN-SC-Y5A3 is 800°C.

5. Conclusions

The results could be summarised as following:

1. Heat resisting limit temperature on monotonic load of crack healed SN-SC-Y8 material is about 1300°C as in the parent material.
2. The crack-healed zone of both SN-SC-Y8 and SN-SC-Y5A3 material is not sensitive to cyclic fatigue at room temperature.
3. The heat resistance limit temperature of crack-healed SN-SC-Y8 material was 1200°C, but the material is not sensitive to cyclic fatigue up to 1300°C.
4. In the SN-SC-Y5A3 material, the crack-healed specimen is not sensitive to either static or cyclic fatigue up to 800°C. But, at 1000°C, the static fatigue strength is decreased dramatically and all that specimens that failed in the test are fractured from the crack-healed zone. Consequently, it could be concluded that heat resisting limit temperature of SN-SC-Y5A3 on static fatigue is 800°C.

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