# INELASTIC CYCLIC STRESS-STRAIN RESPONSE IN SILICON-NITRIDE CERAMICS AT ELEVATED TEMPERATURES

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### **ABSTRACT**

The extensometer was newly developed for measuring axial displacement of ceramics and its composite materials at elevated temperatures. Then the high temperature use-closed loop type push-pull fatigue test system was equipped with this extensometer.

The push-pull cyclic loading tests were performed for silicon nitride ceramics under the controlled triangular and trapezoidal stress wave loadings at 1300°C, and its cyclic stress-strain response was measured. The inelastic strain, which is greatly dependent upon stress rate, was detected in this material at 1300°C. It was found from the shape of the cyclic stress-strain hysteresis loop that inelastic strain was easier to generate under tensile loading than under compressive one; the width in the hysteresis loop was larger on the tensile stress side than on the compressive stress side. Moreover, the cyclic stress-strain hysteresis loop showed that the positive and negative inelastic strains were generated on unloading excursions from the tensile maximum and the compressive minimum stresses as well as on the tensile and compressive loading excursions. The creep strain was also detected during the tensile and compressive stress-hold periods under the trapezoidal stress wave loading. At that time the amount of the creep strain is much larger during the tensile stress-hold period than during the compressive stress-hold one. Another notice is that the cyclic inelastic strain decreases with progress of cyclic loading process, showing that strengthening occurs during cyclic loading at 1300°C in Si<sub>3</sub>N<sub>4</sub> ceramics.

#### **KEY WORDS**

Si<sub>3</sub>N<sub>4</sub> Ceramics, Push-pull Cyclic Loading, Elevated Temperatures, Cyclic Stress-strain Response, Inelastic Strain, Fatigue Life

#### INTRODUCTION

The basic and practical studies have been extensively carried out for applying ceramics materials, which possess excellent heat-resisting mechanical properties, to high temperature machine components. Fatigue is one of the important mechanical properties to be clarified for actualizing this [1,2].

The cyclic stress-strain response is a fundamental mechanical property for fatigue life prediction.

Its

measurement, however, is extremely difficult at so called ultra-high temperatures in ceramics materials; the amount of displacement generating in these materials is quite small, and ultra-high temperature-use extensometer has to be developed to measure such a small displacement. Then the cyclic stress-strain response of ceramics materials has been little examined at elevated temperatures so far.

The inelastic deformation occurs in Si<sub>3</sub>N<sub>4</sub> ceramic at elevated temperatures [3,4]. The present study aims at clarifying the inelastic cyclic stress-strain response of Si<sub>3</sub>N<sub>4</sub> ceramic at 1300°C, which has not been little examined.

#### **USED MATERIAL AND TEST PROCEDURES**

The tested material is sintered silicon nitride ceramic which was fabricated by mixing Y- $\alpha$  sialon and Si<sub>3</sub>N<sub>4</sub> particles at ratios of 40 and 60 weight percents, and then sintering the mixed particles in N<sub>2</sub>-gas atmosphere at 1750°C. The sintered column was machined into the specimen shown in Fig.1.

The electro-hydraulic type-closed loop type test system was employed for push-pull loading test at 1300°C [2,5], where cyclic triangular stress/strain waves with stress/strain ratios of -1.0 were applied to the specimen, controlling the stress/strain rates. The trapezoidal strain wave with stress-hold time was also used in the cyclic loading test. The displacement was measured in the gauge length of 10mm settled in the parallel part of the specimen by means of the self-designed extensometer.

#### TEST RESULTS AND DISCUSSION

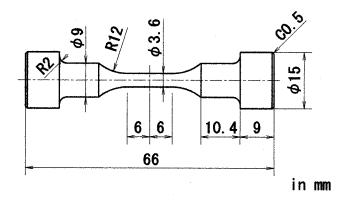


Figure 1: Configuration of test specimen

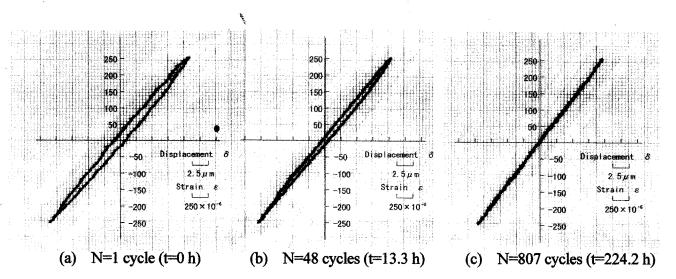


Figure 2: Change in cyclic stress-strain response with increase in number of stress cycles at  $\Delta \sigma / 2 = 250$  MPa and  $\dot{\sigma} = 1.0$  MPa/s at 1300°C in Si<sub>3</sub>N<sub>4</sub> ceramic

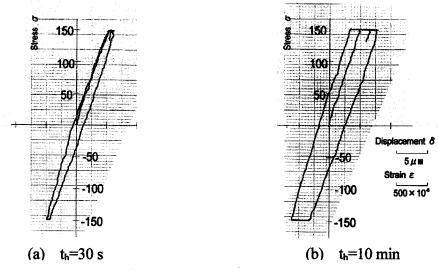


Figure 3: Cyclic stress-strain response under trapezoidal stress wave loading at 1300°C in Si<sub>3</sub>N<sub>4</sub> ceramic

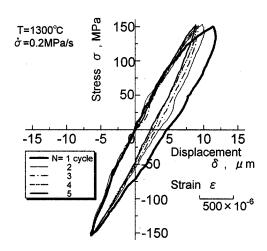
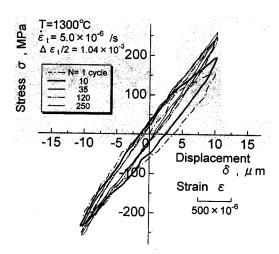


Figure 4: Cyclic stress-strain response at early stage in cyclic triangular stress wave loading process at  $\Delta \sigma / 2 = 150$  MPa and  $\dot{\sigma} = 0.2$  MPa/s at 1300°C in Si<sub>3</sub>N<sub>4</sub> ceramic

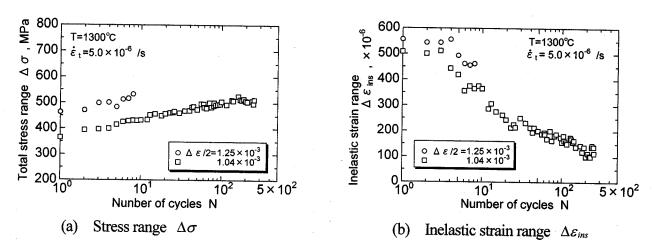
Figure 2 shows the variation in cyclic stress-strain response measured during cyclic loadings at the stress amplitude  $\Delta\sigma/2=250$  MPa controlled at the stress rate  $\dot{\sigma}=1$  MPa/s. The inelastic strain detected at N = 1 cycle is gradually reduced with progress of fatigue process, and then almost disappears at N = 807 cycles. This shows the occurrence of the increased resistance against cyclic deformation during cyclic loadings at 1300°C. Figures 2 (a) and (b) also show that much larger inelastic strain is induced by tensile stress than by compressive stress.

Figures 3 (a) and (b) show the cyclic stress-strain response measured for the first one cycle under the cyclic trapezoidal stress wave loadings at  $\Delta\sigma/2=150$  MPa with the stress rate on loading and unloading excursions,  $\dot{\sigma}=2$  MPa/s and the stress hold times  $t_h=30$  s and 10 min. The creep strain is generated for the stress held at  $\sigma=\pm150$  MPa, of which amount is dependent on the hold time  $t_h$  and the sign of the stress; the creep strain is much greater for the longer  $t_h$  and on the tensile stress side than on the compressive stress one.

The change in the stress-strain hysteresis loop measured under cyclic triangular stress wave loadings at  $\Delta \sigma / 2 = 150$  MPa and  $\dot{\sigma} = 0.2$  MPa/s is shown in Fig.4. The much larger inelastic strain is shown in the figure than in Fig.2, in which the cyclic stress-strain response is obtained under cyclic loadings at



**Figure 5:** Cyclic stress-strain response at early stage in cyclic triangular strain wave loading process at  $\Delta \varepsilon_t / 2 = 1.04 \times 10^{-3}$  and  $\dot{\varepsilon}_t = 5.0 \times 10^{-6} / \text{s}$  at  $1300^{\circ}\text{C}$  in Si<sub>3</sub>N<sub>4</sub> ceramic



**Figure 6:** Variations in stress and inelastic strain ranges in cyclic triangular strain wave loadings at  $\dot{\varepsilon}_i = 5.0 \times 10^{-6} / \text{s}$  at 1300°C in Si<sub>3</sub>N<sub>4</sub> ceramic

 $\Delta\sigma/2=150$  MPa and  $\sigma=1$  MPa/s. Thus the inelastic strain generated at a given stress amplitude is greatly dependent upon the stress rate, and less at the larger stress rate. The great amount of inelastic strain yields at first tensile loading, and this increases even in the unloading process. Furthermore, the inelastic strain due to compressive stress is much less than that due to tensile stress. The gradual decrease in inelastic strain is also clearly found during stress cycling in the figure, of which the greater part occurs on the tensile stress side. As the result, the difference between the amounts of inelastic strain on the tensile and compressive stress sides is reduced with progress of the stress cycling process.

Figure 5 shows the change in the cyclic stress-strain response obtained from cyclic straining test controlled at strain amplitude  ${}^{\bullet}\Delta\varepsilon_t/2 = 1.04 \times 10^{-3}$  at strain rate  $\dot{\varepsilon}_t = 5.0 \times 10^{-6}$ /s. The stress and the inelastic strain increase and decrease with progress of cyclic straining process, respectively, largely on the tensile stress side and slightly on the compressive stress side in the figure. Consequently, the stress-strain hysteresis loop shifts from the configuration with the lower stress and the larger inelastic strain on the tensile stress side from the one with almost the same stress and inelastic strain in absolute value at a given tensile and compressive total strains.

Figures 6 (a) and (b) show the variations in stress and inelastic strain ranges in cyclic straining process at  $\dot{\varepsilon}_i = 5.0 \times 10^{-6}$  /s at 1300°C. The figures clearly show that the stress range increases and the inelastic strain range decreases as cyclic straining process progresses, exhibiting the increasing resistance to cyclic

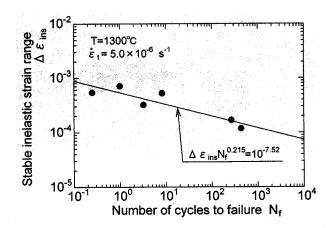


Figure 7: Relationship between inelastic strain range and fatigue life obtained from strain-controlled cyclic loading test at  $\dot{\varepsilon}_t = 5.0 \times 10^{-6} / \text{s}$  at  $1300^{\circ}\text{C}$  in Si<sub>3</sub>N<sub>4</sub> ceramic

deformation during strain cycling. Such phenomena correspond well to the cyclic stress-strain response under stress-controlled cyclic loading, which is shown in Fig.2.

The fatigue life  $N_f$  was plotted against the stabilized inelastic strain range  $\Delta \varepsilon_{ins}$  in the strain-controlled fatigue test at  $\dot{\varepsilon}_i = 5.0 \times 10^{-6}$ /s, in Fig.7, which was determined at half the number of cycles to failure. The figure shows that the relationship between  $\Delta \varepsilon_{ins}$  and  $N_f$  is expressed by a linear line on double logarithmic scales, as known in metallic materials. The exponent  $\alpha$  in the Manson-Coffin plot [6,7]  $\Delta \varepsilon_p \cdot N_f^{\alpha} = C$ , however, is 0.202 in this material, which is quite small, compared with the one in metallic materials.

## **CONCLUSIONS**

The push-pull cyclic loading tests were performed and cyclic stress-strain response was measured for Si<sub>3</sub>N<sub>4</sub> ceramic under the stress- and strain-controlled conditions at 1300°C. The main results obtained are summarized as follows.

- (1) The inelastic strain is generated during cyclic loadings, of which amount is much greater at the smaller stress/strain rates, and on the tensile stress side than on the compressive stress side.
- (2) The inelastic strain is gradually reduced with progress of the stress- and strain-controlled cyclic loading processes, showing increasing resistance to cyclic deformation in the cyclic loading process.
- (3) The Manson-Coffin plot holds in Si<sub>3</sub>N<sub>4</sub> ceramic at 1300°C, where the exponent  $\alpha$  in  $\Delta \varepsilon_{ins} \cdot N_f^{\alpha} = C$  is 0.202, being much smaller than the one in metallic materials.

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