Evaluation of quasi-static fracture characteristics considering surface conditions of silicon nitride for a space component

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Abstract. ISAS/JAXA is now planning to adopt a thruster made of monolithic silicon nitride (SN282 manufactured by Kyocera Co.) onto a Venus exploration probe, PLANET-C, in replacement of conventional niobium heat-resistant alloy. Silicon nitride is still brittle and requires precise analysis on multiaxial thermal stresses induced during firing, though it has high toughness among other structural ceramics. This study evaluated quasi-static fracture characteristics of SN282 considering the surface conditions through compression-torsion biaxial fracture tests as well as the conventional four-point-bending tests. The samples were applied to the mechanical tests either as-ground or after annealing at 1300°C in air for 1 h, which formed an oxidation layer of more than 250nm on the specimen surface. Symmetry four-point-bending tests showed that annealing improves flexure strength and reduce the difference caused by grinding directions. Biaxial stress fracture tests showed the high compressive stress makes the influence of facial crack insensitive.

1. Introduction

A thruster is a small rocket engine to control attitude or orbit of a spacecraft such as a planetary exploration probe or a satellite. Thruster materials are required oxidation resistance and low density. In addition particularly, propulsion power of a thruster is improved by using higher heat resistance material. Although a conventional thruster has been made of niobium heat-resistant alloy, this alloy must have oxidation resistant coating. Therefore, now ISAS/JAXA is planning to adopt a thruster made of monolithic silicon nitride (SN282 manufactured by Kyocera Co.) onto a Venus exploration probe, PLANET-C, in replacement of niobium heat resistant alloy [1].

SN282 has been developed for a ceramics turbine, exhibits good resistance to oxidation and has larger heat-resistance than niobium heat-resistant alloy. However, it is still brittle and requires precise analysis on multiaxial thermal stresses induced during firing, though it has high toughness among other structural ceramics. It is well known that surface condition strongly effects on mechanical quasi-static strength of ceramics. In addition, for brittle materials, fracture analysis is determined by combination of fracture statistics theory [2] and mixed mode crack propagation criteria [3] [4].
This study evaluated quasi-static fracture characteristics of SN282 silicon nitride considering the surface conditions and multiaxial stress condition through compression-torsion biaxial fracture tests as well as the conventional four point bending tests.

2. Experimental Procedure

2.1 Test machine and specimens

The experiments were conducted using a compression-torsion combined test machine, which is based on a tension/compression tester (AG1 manufactured by Shimadzu) and a retrofitted torque cell with a torque motor. Materials were commercial gas-pressure sintered silicon nitride, SN282. This material is toughened with microstructures tailored to achieve elongated grain structure.

2.2 Symmetric four-point-bending test

Symmetric four-point-bending tests were performed by using a four-point flexure fixture with 10 mm inner and 30 mm outer spans in the ambient temperature and atmosphere condition. The specimens were machined into flexure test specimens of 3 mm × 4 mm × 40 mm bars. Two kinds of finishing were applied: ground parallel or perpendicular to the longitudinal direction of the specimen with #400 grind stones. Half of them were annealed at 1300°C in air for 1 h prior to testing. Flexure strength was determined as follows:

\[\sigma = \frac{3PL - l}{2\omega t} \]

where \(P\) is the load, \(L\) and \(l\) are the outer and inner spans, respectively, and \(\omega\) and \(t\) are the specimen width and depth, respectively.

Brittle materials have a large variety of flexure strength between each specimen. Therefore, flexure strength was determined using a total 30 test specimens through evaluating the Weibull plot.

2.3 Biaxial stress fracture test

Biaxial stress fracture tests were performed in compression-and-torsion mode in ambient temperature and atmosphere condition. This specimen and loading mode are shown in Figs. 1 (a) and (b), respectively. These samples were machined and finished perpendicular to the longitudinal direction of the specimen with #400 grind stones. At first, the compression force, \(\sigma\), was applied. Next, torsion, \(\tau\), was introduced on the specimen with the compression force being kept.

![Fig.1. (a)Biaxial fracture test specimen geometry, (b)stress condition under the biaxial fracture test.](image-url)

The maximum, intermediate and minimum principal stresses \(\sigma_1, \sigma_2\) and \(\sigma_3\) at the moment of fracture were conducted by the following equations:
\[
\sigma_1 = \sigma + \sqrt{\frac{\sigma^2}{4} + \tau^2},
\]

(2)

\[
\sigma_2 = 0,
\]

(3)

\[
\sigma_3 = \frac{\sigma}{2} - \left(\sqrt{\frac{\sigma^2}{4} + \tau^2}\right).
\]

(4)

Since torsion causes non-uniform stress distribution, the fracture stresses are corrected considering effective volume \(V_e\) by the following formula (5) (6) (7)

\[
\sigma_1 = \left(\frac{\sigma}{2} + \sqrt{\frac{\sigma^2}{4} + \tau^2}\right)\left(\frac{V_e}{V}\right)^{\frac{1}{m}},
\]

(5)

\[
\sigma_3 = \left(\frac{\sigma}{2} - \sqrt{\frac{\sigma^2}{4} + \tau^2}\right)\left(\frac{V_e}{V}\right)^{\frac{1}{m}},
\]

(6)

\[
V_e = 2V\int_0^{r_0} \left(\frac{\sigma}{2} + \sqrt{\frac{\sigma^2}{4} + \tau^2}\right)\left(\frac{r}{r_0}\right)^m r dr.
\]

(7)

3. Experimental results and discussion

The two-parameter Weibull plots of flexure strengths of SN282 are shown in Fig. 2, where the variables \(F\) and \(\sigma\) are failure probability and flexure strength, respectively.

![Two parameter Weibull plots of flexure strength of SN282 test specimens with two kinds of finishing obtained by symmetric four-point-bending test.](image)

For non-annealed specimens, Weibull modulus, \(m\), and characteristic strength, \(\sigma_\Lambda\), were \(m = 11\) and \(\sigma_\Lambda = 630\) MPa for the specimen ground parallel to the longitudinal direction, and \(m = 9\) and \(\sigma_\Lambda = 508\) MPa for the specimen ground perpendicular to the longitudinal direction. On the other side, for
the annealed specimens, $m$ and $\sigma_A$ were $m = 14$ and $\sigma_A = 731$ MPa for ground parallel, and $m = 13$ and $\sigma_A = 708$ MPa for ground perpendicular. These results show that annealing improves flexure strength of SN282 and reduces the difference of finishing directions.

Auger Electron Spectroscopy (AES) was executed to measure the surface conditions of the annealed and non-annealed specimens. For the non-annealed specimens, oxygen remains 8 at.% to 50 nm in depth from the specimen surface. In contrast, for the annealed specimens oxygen remains 34 at.% to 250 nm in depth. Therefore, an oxidation layer was formed on the surface of the annealed specimens and the oxidation layer functions to infill surface micro-flaws.

For biaxial stress fracture test, as shown Fig. 3, a compression force, $\sigma$, is applied on the specimen, with varying $\sigma_3$. Next, a torsion, $\tau$, is introduced on the specimen, with varying both $\sigma_1$ and $\sigma_3$. For a low compressive stress field, annealing has a large effect on the fracture criteria. On the other side, for a high compressive stress field, annealing has little effect on the fracture criteria. AES results showed that annealing generated an oxidation layer on the specimen surface which infiltrates facial micro-flaws of the specimen. Therefore, it is considered that the high compressive stress makes the influence of facial micro-flaws insensitive.

![Fig.3. Biaxial stress fracture test result for annealed and non-annealed specimens.](image)

4. Conclusions

Quasi-static fracture characteristics of SN282 silicon nitride considering the surface conditions and multi-axial stress condition were evaluated, through compression-torsion biaxial fracture tests as well as the conventional four point bending tests. Four-point-bending tests showed that annealing improves flexure strength and reduce the difference of grinding directions. Biaxial stress fracture tests showed that the high compressive stress makes the influence of facial micro-flaws insensitive.

References

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