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DEVELOPMENT AND EVALUATION OF SILICON NITRIDE COMPONENTS FOR CERAMIC GAS TURBINE

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ABSTRACT

NGK Spark Plug Co., Ltd. has been developing various silicon nitride materials, and the technology for fabricating components for ceramic gas turbines (CGT) using these materials. We are supplying silicon nitride material components for the project to develop 300 kW class CGT for co-generation in Japan. EC-152 was developed for components that require high strength at high temperature, such as turbine blades and turbine nozzles.

In order to adapt the increasing of the turbine inlet temperature (TIT) up to 1350 °C in accordance with the project goals, we developed two silicon nitride materials with further improved properties: ST-1 and ST-2. ST-1 has a higher strength than EC-152 and is suitable for first stage turbine blades and power turbine blades. ST-2 has higher oxidation resistance than EC-152 and is suitable for power turbine nozzles.

On applying these silicon nitride ceramics to CGT engine, we evaluated various properties of silicon nitride materials considering the environment in CGT engine. Particle impact testing is one of those evaluations. Materials used in CGT engine are exposed in high speed gas flow, and impact damage of these materials is considered to be a concern. We tested ST-1 in the particle impact test. In this test, we observed fracture modes, and estimated the critical impact velocity.

This paper summarizes the development of silicon nitride components, and the result of evaluations of these silicon nitride materials.

INTRODUCTION

300 kW CGT class engines have been developed in research commissioned by NEDO. This project was started in 1988 fiscal year and is planned to end in 1998 fiscal year. The aim of the project is to improve the thermal efficiency of small gas turbines to the point where they rival that of large gas turbines by using ceramic components. One of these CGTs, the CGT301 engine, was designed by IHI. The engine is a recuperated single-shaft CGT. We supplied the first stage turbine blades for this CGT engine, and in 1997 we started supplying the second stage turbine blades also. The CGT303 engine was designed by YDE and its development was completed in 1995. We supplied the power turbine blades and power turbine nozzle for this engine.

As the first step in the 300 kW CGT project, a basic CGT with a turbine inlet temperature (TIT) of 1200°C was developed. However, it was anticipated that in order to adapt the increasing of the TIT to 1350 °C in accordance with the project goals, which was to develop a pilot CGT with a TIT of 1350 °C, a material with more outstanding characteristics than the conventional material - EC-152 - would be required. Therefore, in order to further improve the material characteristics, we developed silicon nitride materials ST-1 and ST-2 for the pilot CGT. ST-1 has greater strength than EC-152 at high temperatures, and ST-2 has a higher oxidation resistance than EC-152 at high temperatures.

In addition to the general evaluations of strength and other factors, we also conducted a special evaluation based on the conditions inside the CGT engine. The results of a part of this evaluation, a particle impact test, are reported in this paper. This test was conducted to evaluate the impact resistance of silicon nitride materials to Foreign Object Damage (FOD). The test was carried out by using the impact test apparatus at the Mechanical Engineering Laboratory. On the basis of the results of this test, we evaluated the mechanism of impact fracture and the critical impact velocity for fracture.

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NOMENCLATURE

CGT Ceramic Gas Turbines
IHI Ishikawajima-Harima Heavy Industries Co., Ltd.
NEDO New Energy and Industrial Technology Development Organization
TIT Turbine Inlet Temperature
YDE Yamaha Diesel Engine Co., Ltd.
SILICON NITRIDE MATERIALS AND CGT COMPONENTS

The material properties of the developed materials EC-152, ST-1 and ST-2 are shown in Table 1, and the four point bending strength is shown in Figure 1. EC-152 is the material developed for the basic CGT (TIT = 1200°C), and gave favorable results with the basic CGT. ST-1 has higher strength than EC-152 over the entire temperature range from room temperature to 1300°C. ST-2 has the highest strength at temperatures over 1300°C, and has high oxidation resistance at high temperatures.

ST-1
Since a lower sinter temperature can be used when manufacturing ST-1 (by hot isostatic pressing) then when manufacturing EC-152 (by gas pressure sintering), ST-1 can be made with a fine grain. So, ST-1 has a higher strength than EC-152 in the range from room temperature to 1300°C. During operation inside the pilot CGT engine, the turbine blades reach a temperature of between 1000 and 1200°C. Because ST-1 has a higher strength than EC-152 within this temperature range, ST-1 was used instead of EC-152 in the pilot CGT. With its exceptional strength at high temperatures, ST-1 is suitable for use in ceramic parts which are subjected to stress at high temperatures. The profile for the first stage turbine blades that we supplied for the CGT301 project is shown in Figure 2. The first stage turbine blades are fabricated by injection molding. The CGT301 uses a hybrid turbine constructed by inserting ceramic first stage turbine blades into a metal disk. The number of turbine blades is 37, and the rated rotational speed is 56000 rpm. The blade profile has as-sintered surfaces, and other parts are ground. From this fiscal year (1997), first stage turbine blades for use in the pilot CGT have been supplied.

Table 1. Mechanical properties of developed materials

<table>
<thead>
<tr>
<th></th>
<th>EC-152</th>
<th>ST-1</th>
<th>ST-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>density (g/cm³)</td>
<td>3.26</td>
<td>3.37</td>
<td>3.42</td>
</tr>
<tr>
<td>strength (MPa)</td>
<td>1020</td>
<td>1090</td>
<td>780</td>
</tr>
<tr>
<td>poison's ratio</td>
<td>0.26</td>
<td>0.28</td>
<td>0.27</td>
</tr>
<tr>
<td>Young's modulus (GPa)</td>
<td>318</td>
<td>317</td>
<td>320</td>
</tr>
<tr>
<td>toughness (MPa m¹/²)</td>
<td>6.0</td>
<td>5.6</td>
<td>5.0</td>
</tr>
<tr>
<td>hardness (HV30)</td>
<td>1500</td>
<td>1700</td>
<td>1450</td>
</tr>
</tbody>
</table>

ST-2 uses additives in order to generate grain boundaries with high resistance, and is manufactured by hot isostatic pressing. This material also shows high oxidation resistance at high temperatures. The weight gains of the silicon nitride materials EC-152, ST-1 and ST-2 in the oxidation test at 1000 to 1200°C are shown in Figure 3. The weight gain of ST-2 due to oxidation is extremely small in comparison with that of EC-152; at 1200°C it is only 1/3 that of EC-152. These properties suit ST-2 to use in members which require strength and oxidation resistance at temperatures greater than 1300°C. The power turbine nozzle for which ST-2 was used in the CGT303 project is shown in Figure 4. The power turbine nozzles were fabricated by injection molding. The number of the nozzles is 27. The blade profile has as-sintered surfaces; other parts are ground.
EVALUATION OF MATERIALS (Particle impact Test)

On applying these silicon nitride materials in CGT engines, we conducted a special evaluation based on the conditions inside the CGT engine in addition to the general material evaluation tests (strength at room temperature, etc.). One of the tests involved in this evaluation was the particle impact test. Inside an actual CGT engine, the CGT components are exposed to a flow of gas at high temperature, and it is possible that foreign objects mixed in with the stream of gas will damage the blades. Examples of foreign object damages (FODs) were reported in literature on ceramic gas turbines (Song, Cuccio and Kington, 1991). For the purpose of evaluating the impact resistance of the material and to clarify the mechanism for impact damage, we conducted particle impact tests at room temperature.

Experimental Setup

For this test, the impact test apparatus in the Mechanical Engineering Laboratory of the Agency of Industrial Science and Technology, MITI, is used. The external appearance and outline of the test apparatus are shown in Figure 5 and Figure 6 respectively. The particle acceleration equipment used in this apparatus comprised an electro-thermal (ET) gun and a micro-sabot system which accelerates a particle launched on a plastic pedestal called a "micro-sabot". The inside of the chamber was evacuated to enable the particle fly at supersonic velocities. More details are given in Ref. [2] by H. Yoshida et al.

Flying Particle

The flying particle was a magnetized sphere made of samarium-cobalt (Sm-Co). When the flying particle passed pickup coils installed on its trajectory, induction current signals were detected. The particle velocity was calculated by measuring the time taken for the particle to pass two pickup coils. The material properties of Sm-Co are given in Table 2. Sm-Co is softer than silicon nitride ceramics.

Specimen

The shape and setting of the specimen is shown in Figure 7. The specimen was a circular disk specimen with a diameter of 40 mm and a thickness of 1.0, 1.5, or 2.0 mm. The surface of the specimen was lapped to preclude any effects caused by grinding scrapes. The material of the specimen was ST-1, which is used as a material for rotor blades. The material characteristics of ST-1 were presented previously in Table 1. The test piece was secured by gripping both sides of its periphery with a jig. The tightening torque applied to hold the test piece was 1 kg/m. The flying particle was directed at the center of the test piece.

RESULT AND DISCUSSION

Crack Morphology

In this test, it was found that fractures of specimens occurred in two kinds of fracture modes: Hertzian fracture mode which developed on the impact face and flexural fracture which developed on the rear face of the specimen. The fracture origins of Hertzian and flexural fracture were observed as ring cracks on the impact face and radial cracks on the rear face, respectively.
The typical crack morphologies observed in this test are shown in Figure 8, 9, and 10. We classified crack morphologies in following 3 types: 1. Hertzian Fracture, 2. Flexural Fracture, 3. Combination of Hertzian Fracture and Flexural Fracture.

Now, we describe these crack morphologies below.

Crack morphology 1: Hertzian Fracture (Figure 8)

When only Hertzian fracture occurs, ring cracks are observed on the impact face. Hertzian crack progresses from ring crack into the specimen. If the flying particle has sufficient velocity, a Hertzian crack reaches the rear face of the specimen, and then a cone-shaped hole and a cone-shaped fragment are formed. In cases where Hertzian crack reached the rear face, most of specimens fractured by cracking from Hertzian cracks.

Crack morphology 2: Flexural Fracture (Figure 9)

When only flexural fracture occurs, radial cracks are observed on the rear face. Radial cracks are caused by flexural tensile stress on the rear face. As the velocity of the flying particle increases, the radial cracks formed by the flexural fracture become larger, until finally the test piece fractures. The radial cracks of flexural fracture have the characteristic that they spread from a single central point on the rear face of the specimen.

Crack morphology 3: Combination of Hertzian Fracture and Flexural Fracture (Figure 10)

When both Hertzian fracture and flexural fracture occur, the crack morphology can be classified into below three levels.

- In the low-velocity range, ring cracks caused by Hertzian fracture on the impact face and radial cracks caused by flexural fracture on the rear face are observed.
- As the velocity increases, Hertzian cracks which occur on the impact face progress to the rear face, and form ring cracks on the rear face. (shown in Figure 10(a))
- As the velocity increases still further, cone-shaped hole is formed by the penetration of Hertzian cracks. Then corresponding cone-shaped fragments are produced. (shown in Figure 10(b) and Figure 11) Radial cracks caused by flexural fracture are observed on the rear faces of recovered cone-shaped fragments. When only Hertzian fracture occurs, no radial cracks are observed on the rear face of the cone-shaped fragments.

These crack morphologies consist of Hertzian fracture and/or flexural fracture. Then, critical impact velocities for these fracture are discussed below.
Critical Impact Velocity for Hertzian Fracture
(Vc for Ring Cracks on the impact face)

Hertzian fracture has the following levels. First, when the flying particle collides with the specimen, tensile stress is generated around the contact area. When this tensile stress exceeds the material strength, ring cracks are formed in the impact face of the specimen. When the velocity of the flying particle is increased further, Hertzian cracks originating from these ring cracks propagate toward the rear face of the specimen. Therefore, ring cracks on the impact face are a guide to Hertzian fracture.

The presence or absence of ring cracks on the impact face of the specimen is shown in Figure 12. Symbols • represent specimens which have ring cracks of Hertzian fracture. It is found that the impact velocity for initiation of ring cracks is roughly about 400 m/sec. Here we assume that the critical impact velocity of Hertzian fracture is equal to 400 m/sec.

Critical Impact Velocity for Flexural Fracture
(Vc for Radial Cracks on the rear face)

The presence or absence of radial cracks of flexural fracture observed on the rear face of the specimen is shown in Figure 13. Symbols ■ represent specimens which have radial cracks of flexural fracture. As shown in Figure 13, for 1.0 and 1.5 mm, the particle velocity at which radial cracking occurred became higher as the thickness of the test piece was increased. For 2.0 mm thickness, the radial crack of flexural fracture was not observed in tested particle velocities. So, according to increasing thickness of specimen, critical impact velocity for radial crack initiation became higher. It is found that the impact velocity for radial crack initiation is influenced by thickness of specimens.

Tsuruta et al have proposed a critical impact velocity equation for the flexural fracture of circular disk specimens (1).  

Equation 1

\[ V_c = C \cdot \sigma^{5/6} \cdot t^{5/3} \] (1)

where, \( V_c \) : Critical impact velocity, \( C \) : Coefficient, \( \sigma \) : Bending strength of material, \( t \) : Thickness of specimen

This equation is derived from the equation for the impulse load in the Hertzian theory of elastic contact 4) and the equation for the maximum stress equation of the circular disk specimen 5). The equation shows that the critical velocity \( (V_c) \) is proportional to \( (\sigma : \text{material strength})^{5/6} \cdot (t : \text{thickness of specimen})^{5/3} \). We applied this critical velocity equation to the test results of this test. The result is shown in Figure 14. In Figure 14, the X-axis shows \( \sigma^{5/6} \cdot t^{5/3} \), where the material strength \( \sigma \) obtained by the four point bending test is used instead of the biaxial bending strength of the disc specimen. And the Y-axis shows the velocity of the flying particle. The results of the presence or absence of radial cracks are plotted in the graph. From Figure 14, the empirical line with 0.85 slope seems divides the presence/absence of radial cracks in specimen with thickness of 1.0 and 1.5 mm. The result of applying the equation for critical impact velocity showed that \( V_c \) for radial crack initiation is proportional to the 5/3 power of the specimen thickness.
Crack Morphology Zones in the FOD Test

By combining the critical velocities for Hertzian fracture and flexural fracture, a zone diagram for crack morphology can be created. Figure 15 shows this zone diagram for crack morphologies. It was created by combined use of two types of experimentally obtained results: the velocity at which ring cracks of the impact face are generated ($V_c = 400 \text{ m/sec}$) and the critical impact velocity equation for flexural fracture ($V_c = 0.85 \sigma^{5/6} \cdot \frac{t^{5/3}}{6}$). In the velocity zone higher than the critical impact velocity for ring crack initiation, Hertzian fracture occurs. And in the zone higher than the critical impact velocity for radial crack initiation of flexural fracture, flexural fracture occurs. Therefore, the crack morphology can be classified into the four zones indicated in the figure: Hertzian cracks only, flexural cracks only, coexistence of Hertzian cracks and flexural cracks, and "no damage". The test point of specimens fractured by Hertzian cracks with 2.0 mm thickness fall within the "Hertzian fracture only" zone of Figure 15 and matches this crack morphology.

![Figure 15. Crack morphology zones](image)

CONCLUSIONS

We participated in the 300 kW CGT project, which was commissioned by NEDO, and supplied many CGT parts for it. Later we developed the new materials ST-1 and ST-2, in addition to the conventional material EC-152, for a pilot CGT with a TIT of 1350 °C.

We have also conducted various evaluation tests based on the actual environment in which the turbine blades are used inside the CGT engine. In one of these tests – the particle impact test – by analyzing the crack morphology and critical velocity, we were able to identify zones for crack morphologies. Specifically, crack morphologies were classified into four zones – Hertzian cracks only, flexural cracks only, coexistence of Hertzian and flexural cracks, and "no damage" – on the basis of the relationship between the critical velocity for flexural fracture and the velocity at which ring cracks were generated.

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REFERENCES