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STRENGTH EVALUATION TEST OF PRESSURELESS-SINTERED SILICON NITRIDE AT ROOM TEMPERATURE

K. Matsusue, K. Takahara, R. Hashimoto

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### 1. PREFACE

It is anticipated that ceramics will be used for machine parts, especially as materials for strong parts, and research and development for putting this into practice has been proceeding. In Japan, applications studies for small scale heat engines have already started being reported in newspapers and magazines. However, since there is a primary problem with the brittle character of ceramics themselves, the outlook for the completion of this study still appears to be indefinite.

Under these circumstances, this laboratory has also been studying the application of ceramics to machine parts for the last few years. Using hot pressed silicon nitride, pressureless sintered silicon carbide and reaction sintered silicon nitride, the bending strength test, tensile test, rotation test, bending test of a notched square bar and the tearing ring test with pressure are performed in order to test strength at room temperature.

From those test configurations, the dimensions of the parts vs. the strength of the ceramics, the effects of stress concentration, the effects of different loading methods and the applications of Weibull's statistical analysis for strength assessment have been studied [1-4]. It was confirmed that parts which are manufactured from hot pressed material are in accord with the strength predicted by Weibull's statistics. However, reaction sintered material and pressureless sintered material are not in accord with the predicted strength. It has been pointed out that many problems remain with the processes of manufacturing these materials. Also, regarding the relationship between the configuration and the strength of parts, the result of the rotation test of the ring specimen and the tearing test with pressure, which were employed in the previous report, did not

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correspond to the result of the simple bending test. Because of this, the strength of the small scale parts with holes was assumed to depend on the complexity of their configuration.

In this report, the bending test of a square bar, tension test of plates with holes or notches and the rotation test of a ring were performed on pressureless sintered silicon nitride which is becoming popular for practical machine parts. Weibull's statistical analysis was applied for each test and the strength assessment for each part was studied. The holed part and notched part which were employed for the tension test are often seen in practical parts. Although there is a stress concentration problem in holed and notched parts when a load is received, the result of the strength test does not correspond well with the flexural strength: this is because when parts are made by the pressureless sintered method, it was found that, depending on the part's configuration, the effects of the atmosphere of the furnace during sintering and of shrinkage of the

sintered body are different. Also, in this report, in order to research the effect of polishing a parts surface, for specimens with different surface processing, the relationship between polished vs. unpolished surfaces, polishing direction and parts strength are studied for the notched test and rotation test samples. It was assumed that holed and notched parts are not influenced by whether they are polished or unpolished, and therefore, the polishing process cannot be expected to affect the part too much. As in the above, there still remain several problems that should be studied. The purpose of this report is to study stress concentration and the influence of a deformed condition, and to obtain design materials for parts, although there are many problems which should be studied for application to complicated machine parts.

### 2. SPECIMEN MATERIALS AND SPECIMENS

The material is silicon nitride which is pressureless sintered. Alumina  $(Al_20_3)$  and magnesia (MgO) are the main components of the sintered material and include 10% of the total. The apparent density of the sintered body is 3.10 to 3.15 g/cm<sup>3</sup> (theoretical current consistency ratio is about 9.5%).

For specimens, a 3x3x50mm square bar for use in the bending strength test, three different kinds of tensile test specimens (shown in Figure 1), and a rotation test specimen were manufactured. Among the above specimens, the square bar used for the bending test is made from a plate sintered body which is processed by cutting and polishing. It is a standard specimen of sample material for the strength test. Also in Figure 1 the tensile test specimen 2) is cut and polished from a plate. But for specimen b) with a notch and specimen c) with a hole, the following three different specimens, which have different surface processes, are used. For baking by the pressureless sintered method, there are two baking processes, which are temporary baking and proper baking. After temporary baking, the part is processed with fixed dimension and proper baking is performed.

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Figure 3. Tensile test 1. Pin 2. glued area 3. specimen At that time, as 15-20% shrinkage will occur, the dimensions (which include anticipated shrinkage) should be processed before proper baking.

Three different kinds of specimen are used for this report. One of them is processed by polishing on all surfaces after proper baking, another one is polished on only the notch edge or hole edge surface, and the last is without any processing after baking. There are two different kinds of rotation test specimens. One is polished only on the edge; the other is not polished. Surface roughness of the polished surrace of each specimen is 1  $\mu$ m (Rmax) and each edge is beveled 0.1-0.3 mm. Some deformation occurred in specimens which were only baked and did not undergo the polishing process, as there is aeolotropic shrinkage during baking. The effect of the deformation was thought to be an error in test strength which would be from eccentricity, or bending and twisting of the load axis from the

tensile test, but according to the following results, not much effect of this deformation has appeared.

### 3. EXPERIMENTAL METHOD

For the bending strength test, a 3-point bending strength test of a 3x3x50 mm square bar (distance between support points is 30 mm) was performed. Since this test is to become a standard strength test of specimen materials, many samples were used. For the tensile test as shown in Figure 3, the following load method was used: both sides of the sample end were glued with soft steel and a pin was passed through the glued ends. In addition, there is a connector for which a cross pin is used. For each test, a Shimazu Universal testing machine (RS-2 type) was used, and loading was done manually. Cross-head speed of the testing machine is in the range of 0.02-0.05 mm/min. It was confirmed that if the test is performed at room temperature, the effect of the load speed on strength can be almost Therefore, in this report even if the manual loading ignored [5]. method is used, it is assumed that this does not influence the rupture strength.

As in the previous report [1], as shown in Figure 4, the rotation test is performed by measuring the breaking rotation of a specimen attached to a step shaft of soft steel material. The core of the spinpit is braced with adhesive tape, although a skip rarely occurs between specimen and shaft, as the pressure in the core of the spinpit is reduced to 0.1 mm Hg. In the case of the rotation test, in order to obtain good centrifugal breakage rotation, the rotation balance of the specimen is very important. Some of the specimens that are used this time are slightly deformed since the surface processing after sintering was eliminated, and rotation balance is not considered to be very good. However, during the test there was no specimen which had any problem such as oscillation. The reason is thought to be that since the mass of the shaft used was large and since the shaft itself is well balanced, imbalance of the specimen

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| test           |                         | N  | ōb   | m    | V <sub>E</sub> po | lishing direction |
|----------------|-------------------------|----|------|------|-------------------|-------------------|
| 3-point bendir | full face<br>g polished | 40 | 55.4 | 9.2  | 1.3               | =                 |
| tensile        | Ħ                       | 13 | 16.9 | 7.0  | 300.              | J.,               |
| tensile (with  | 11                      | 10 | 32.6 | 7.4  | 13.2              | <u>ـ</u> ــ       |
| notch)         | partially<br>polished   | 10 | 23.8 | 3.8  |                   | Ŀ                 |
|                | unpolished              | 9  | 28.8 | 9.1  |                   |                   |
| tension (with  | full face<br>polished   | 10 | 31.4 | 6.9  | 2.9               |                   |
| 11040/         | partially<br>polished   | 10 | 32.2 | 6.4  |                   | =                 |
|                | unpolished              | 10 | 36.7 | 7.3  |                   |                   |
| rotation       | partially<br>polished   | 10 | 31.0 | 7.7  | 2050              | =                 |
|                | unpolished              | 8  | 27.6 | 11.0 |                   |                   |

### TABLE 1. TEST RESULT

### Notation

N: number of specimens  $\overline{\sigma}_b$ : mean breaking at principal axis (kg/mm<sup>2</sup>) m: Weibull coefficient V : effective volume (mm<sup>3</sup>), polishing direction; =: parallel to main tension stress,  $\perp$ : right angle to main tension stress

which has a small amount of mass did not affect the balance of the whole body of rotation. The behavior of the rotating specimen is observed from the observation door, which is above the upper part of the spinpit. Also, cushioning material which is laminated from paper and felt, was installed in the core of the spinpit to prevent secondary failure and to make withdrawal easy. Most of the fragmentation was caught by the cushioning material and recovered.

### 4. EXPERIMENTAL RESULTS

The result of each test is shown in Table 1. The table shows the types of tests, how specimen surfaces were polished, the number of specimens, mean strength, Weibull's coefficient, the effective volume which is enumerated by the three point bending test, and the

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Picture 1. Specimen after breakage

direction of polishing on the specimen surface. Also, a specimen after breakage is shown in picture 1.

### 4.1 Breakage strength

For the breakage strength of each test, the maximum stress value at the time of breakage is used. In the case of strength of brittle materials such as ceramics, the position where maximum stress occurs does not coincide with the point of breakage, as it depends on defects in the part's surface, cracks of the core, holes and impurities. However, most of the time maximum stress is defined as breakage strength.

Strength in the bending test is obtained from  $\sigma_b = 3\ell P/(2bh^2)$  where  $\ell$ . P. b. h are fulcrum distance, breakage load, amplitude and

thickness of the specimen, respectively. For strength in the tension test, first of all, the average stress of the central parallel part is used for an ordinary tensile specimen (Figure 1 part a)). In Figure 1 parts b) and c), which are the specimens with a hole and with a notch, stress distribution was obtained by the finite element method, and the maximum stress at the edge of the holed or notched part is used to determine the breakage strength. The rotation test was enumerated by the following equation:

$$\sigma_{\rm b} = \frac{3+\nu}{4} \rho \omega^2 \left( R_2^2 + \frac{1-\nu}{3+\nu} R_1^2 \right) \tag{1}$$

where  $\rho$  = consistency, w = angular speed, r = Poisson's ratio, R<sub>1</sub> = inside radius, R<sub>2</sub> = outside radius.

The breakage strengths which were acquired from each test for all specimens are shown in Table 2.

Among these test results, the result of the bending test is a representative of strength among the materials used. Therefore, the mean and dispersion of bending strength is a result which becomes a basis for examining the characteristic strengths of material.

### 4.2 Stress distribution and effective volume

Ceramic materials basically have many defects. Since each defect has its own corresponding strength, if it receives a load, the part starts breaking from the defect which reaches its strength limit first. Since there are dispersions in the strength of each defect, the area which has high stress does not always correspond to the most dangerous defect, and there is a danger of breakage even in low stress areas. However, as the kinds of defects and their sizes and distributions are random, generally in cases where stress is high and the area is wide, the probability of the existence of a dangerous defect is usually considered high.

As above, the strength of ceramics is related not only to maximum stress, but also to its distribution. Also, in relation to the

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## TABLE 2: BREAKAGE STRESS OF EACH SPECIMEN

NO--number of specimens, l--span (mm);  $\bar{\sigma}_{l}$ --mean breakage stress (kg/cm<sup>2</sup>); m--Weibull's coefficient Notation:

[3-point bending]  $3 \times 4$ ,  $\ell = 30$  mm,  $\overline{\sigma}_{\rm b} = 55.4$  kg/mm<sup>2</sup>, m= 9.2

| NO    | σb    | NO    | σb    | NO    | σb    | NO    | σь    | NO     | σb             |
|-------|-------|-------|-------|-------|-------|-------|-------|--------|----------------|
| 1-1   | 6 2,5 | 5 - 1 | 4 0.0 | 9-1   | 4 5.0 | 13— r | 5 8.2 | 17-1   | 61.2           |
| 1-2   | 56,5  | 5-2   | 4 6.3 | 9 ~ 2 | 56.0  | 13-2  | 5 5,5 | 17 - 2 | 59.8           |
| 2-1   | 4 1,8 | 6-1   | 4 9,5 | 10-1  | 6 0.3 | 14-1  | 5 3.8 | 18-1   | 4 6.0          |
| 2 - 2 | 50.3  | 6-2   | 5 9.2 | 10-2  | 4 9.3 | 14-2  | 5 2,8 | 18-2   | 60.7           |
| 3 1   | 6 6.3 | 7-1   | 5 6.2 | 11-1  | 3 4.0 | 15-1  | 5 7.3 | 19-1   | 5 6.5          |
| 3-2   | 48.0  | 7-2   | 5 2.7 | 11-2  | 54.7  | 15-2  | 64.0  | 192    | 5 7 <i>.</i> 5 |
| 4-1   | 58.0  | 8-1   | 5 6.7 | 12-1  | 68,3  | 16-1  | 66.3  | 20-1   | 61.8           |
| 4 - 2 | 6 3.7 | 8-2   | 63.2  | 12-2  | 61.3  | 16-2  | 5 2.7 | 20 - 2 | 54.0           |

[tensile]  $\overline{\sigma}_b = 16.9 \text{ kg/mm}^2$ . m = 7.0

| NO | σb    | NO | σb    | NÕ | σь    | NO | σb    | NO | σb    |
|----|-------|----|-------|----|-------|----|-------|----|-------|
| 1  | 17.9  | 5  | 1 1.9 | 8  | 21.5  | 11 | 1 8.1 | 14 | 1 4.9 |
| 2  | 1 9.2 | 6  | 1 5.1 | 9  | 1 7.3 | 12 | 1 9.1 |    |       |
| 3  | 1 5.2 | 7  | 20.6  | 10 | 1 3.9 | 13 | 1 4.4 |    |       |

[tensile (with hole)] (full face

polished)  $\overline{\sigma}_{\rm b} = 31.4 \, \rm kg/mm^2$ , m=6.9

|         |       |    |                  |    |                  |    |       |    | A REAL PROPERTY AND A REAL |
|---------|-------|----|------------------|----|------------------|----|-------|----|--|
| NO      | σh    | NO | $\sigma_{\rm b}$ | NO | $\sigma_{\rm b}$ | NO | σb    | NO | $\sigma_{\rm h}$   |
| 1       | 28.8  | 3  | 2 9.7            | 5  | 3 2.9            | 7  | 3 1.0 | 9  | 3 5,0  |
| 2       | 3 2.2 | 4  | 41.8             | 6  | 3 0.7            | 8  | 28.3  | 10 | 24.0   |
| <b></b> |       |    |                  |    |                  |    |       |    |  |

[tensile (with hole)] (partially polished)  $\overline{\sigma}_{b} = 32.2 \text{ kg/mm}^{2}, \text{ m} = 6.4$ 

| { | NO | σь    | NO | σb    | NO | $\sigma_{b}$ | NO | $\sigma_{b}$ | NO | $\sigma_{\rm b}$ |
|---|----|-------|----|-------|----|--------------|----|--------------|----|------------------|
| l | 1  | 1 9.9 | 3  | 3 6.3 | 5  | 26.5         | 7  | 3 0.7        | 9  | 2 6,5            |
|   | 2  | 3 3.4 | 4  | 3 3,5 | 6  | 4 1.1        | 8  | 39.2         | 10 | 3 5.1            |

[tensile (with hole)] (unpolished)  $\overline{\sigma}_{\rm b} = 3.6.7 \, {\rm kg/mm^2}, m = 7.3$ 

| NÖ | $\sigma_{\rm b}$ | NO | σb    | NO | σb    | NO | σb    | NO | $\sigma_{\rm b}$ |
|----|------------------|----|-------|----|-------|----|-------|----|------------------|
| 1  | 4 5.4            | 3  | 40.0  | 5  | 34.3  | 7  | 3 2.7 | 9  | 37.5             |
| 2  | 43.2             | 4  | 4 2.9 | 6  | 3 0.9 | 8  | 27.3  | 10 | 3 2.8            |

# (Table 2 continued)

[tensile (with hole)] (full face

|    | pointsided) $\sigma_{\rm b} = 32.6  \rm kg/mm^2 \cdot m = 7.4$ |    |      |    |      |    |       |    |       |  |  |  |  |  |
|----|--|----|------|----|------|----|-------|----|-------|--|--|--|--|--|
| NO | σь   | NO | σb   | NO | σb   | NO | σb    | NO | σb    |  |  |  |  |  |
| 1  | 39.5   | 3  | 40.6 | 5  | 30,9 | 7  | 3 4.8 | 9  | 28.0  |  |  |  |  |  |
| 2  | 3 5.6  | 4  | 27.2 | 6  | 32.8 | 8  | 3 0.3 | 10 | 2 6.0 |  |  |  |  |  |

[tensile (with notch)]

(partially polished)  $\overline{\sigma}_{b} = 23.8 \text{ kg/mm}^2 \cdot \text{m} = 3.8$ 

| NO | σь    | NO | σb    | NO | σb    | NO | đb    | NO | σb    |
|----|-------|----|-------|----|-------|----|-------|----|-------|
| 1  | 3 0.6 | 3  | 31.4  | 5  | 29.4  | 7  | 1 8.5 | 9  | 1 9.6 |
| 2  | 14.1  | 4  | 2 1.7 | 6  | 2 0.2 | 8  | 14,9  | 10 | 37.3  |

[tensile (with notch)] (unpolished)  $\overline{\sigma}_{\rm b} = 28.8 \, \rm kg/mm^2, m=9.1$ 

| Í | NO | σ <sub>b</sub> | NO | σ <sub>b</sub> | NO | σb    | NO | $\sigma_{\rm b}$ | NC | σь   |
|---|----|----------------|----|----------------|----|-------|----|------------------|----|------|
|   | 1  | 28.7           | 3  | 3 2.0          | 5  | 3 0.0 | 7  | 23.2             | 9  | 27.6 |
| - | 2  | 20.7           | 4  | 3 4.2          | 6  | 30,5  | 8  | 3 1.9            | İ  | l    |

[rotation] (partially polished)  $\overline{\sigma}_{b} = 31.0 \text{ kg/mm}^{2}$ . m=7.7

| NO | σ     | NO | σb    | NO | σև    | NO | σb   | NO | σb    |
|----|-------|----|-------|----|-------|----|------|----|-------|
| 1  | 3 5.5 | 3  | 34,5  | 5  | 30.0  | 7  | 21.6 | 9  | 2 9.1 |
| 2  | 2 9,5 | 4  | 3 4.0 | 6  | 2 5.9 | 8  | 31.0 | 10 | 38.8  |

[rotation] (unpolished)  $\overline{\sigma}_{b} = 27.6 \text{ kg/mm}^2$ . m=11.0

| NO | συ    | NO | σb   | NO | σb    | NO | συ    |
|----|-------|----|------|----|-------|----|-------|
| 1  | 25.0  | 4  | 31.5 | 6  | 27.7  | 8  | 2 5.4 |
| 3  | 2 6.8 | 5  | 26,8 | 7  | 3 2.0 | 9  | 2 5.9 |

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distribution of stress, the size of the stress area affects the strength of the part.



Figure 5. Stress distribution of tensile specimen and rotation specimen

Figure 5 shows the stress distribution of two different kinds of tension tests and rotation tests which were employed in this test. The abscissa shows the distance from the maximum stress point of the cross-section that produces stress centralization. The vertical axis indicates the ratio between the stress and the average stress of the cross-section. The numeral shown on the black points on the ordinate is the stress centralization coefficient.

The stress distribution in the bending test is a straight line distribution which is divided between tension stress and compression stress adjoining the neutral plane. Tension stress within the cross-section is small. Compared to the case of the tension specimen in Figure 1a, the entire central parallel portion has a uniform tension stress.

As published in the previous report, for evaluating the strength <u>/5</u> of specimens which have different distributions, Weibull's statistical analysis is considered to be the most logical method. Weibull's breakage probability and distribution functions are shown in the following equation [6]:

$$F(\sigma) = 1 - \exp\left(-\int_{v} \left(\frac{\sigma}{\sigma_{0}}\right)^{m} dv\right)$$
(2)

where  $\sigma$  is the stress within the area and m and  $\sigma_0$  are material constants. The integral is carried out only over the area of tension stress. The significance level of breakage is shown within the brackets of equation (2). Equation (2) is a basic equation of Weibull's statistics; it is used for the stress evaluation of specimens which have different stress distributions and stress areas. If the maximum stress  $\sigma_0$  of a specimen used in the brackets of equation (2) is rewritten, we obtain the relations in the following equation [6].

$$V_{\rm E} = \int_{V}^{V} \left( \frac{\sigma}{\sigma_{\rm b}} \right)^{\rm m} \, \mathrm{d} \, v \tag{3}$$

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 $\boldsymbol{V}_{_{\rm E}}$  is proportional to the level of significance of breakage, and if the value is smaller, breakage rarely occurs. As equation (3) shows clearly, in case that  $\sigma$  is constant, since  $\sigma$  is equal to  $\sigma_{\rm b}$ , then  $V_{\rm g}$  is equal to the volume of the integral domain. Also, for the two kinds of specimen whose  $\sigma$  distribution is equal to the integral domain, if volume increases then  $V_{\rm m}$  increases. In fact, equation (3) shows a scale effect towards the level of significance. If volume increases, the significance level of breakage becomes higher, and strength decreases. As mentioned above,  $V_{\rm p}$  shows the stress distribution within a specimen as well as the relationship between stress area and breakage strength. This  ${\rm V}_{\rm E}$  is also called the effective volume of the specimen [6], as the dimension effect of the specimen is considered to be a weighted solidity which can be explained stochastically. The effective volume of each specimen is shown in Figure 1. In the 3-point bending test, since the distribution of stress is given as a function of coordinates, effective volume is enumerated by using the integral of equation (3),

 $V_B = V / (2 (m+1)^2)$ . But V and m are Weibull's coefficient, which is acquired from the volume between the fulcrums of each specimen and the result of the bending test. As in the previous report [1], Weibull's coefficient coefficient m was acquired by the maximum likelihood estimation method using equation (2). Weibull's coefficient m = 9.2 of the bending test is a material constant which was used in this report for the sintered body. For the bending test of the specimens with notches and holes, the distribution of stress becomes complicated and the integral in equation (3) becomes harder so the effective volume was enumerated by the available element method.

### 4.3 Strength evaluation

As mentioned in the last section, the strength of ceramics is not only decided by the maximum stress (considered to be the stress centralization that occurs within parts), but is also influenced by the breadth of the area which is under comparatively high stress. Therefore, it is thought that the concept of effective volume, which is derived by considering both maximum stress and the stress areas, is an effective method for evaluating the strength of each specimen and the strength of ceramic parts. Strength and effective volume between two different specimens for which the configuration, dimension and load method are different are related by the following equation, which proves the equality of the breakage probability of both specimens [6].

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{V_{\rm E2}}{V_{\rm E1}}\right)^{\frac{1}{\rm m}} \tag{4}$$

Equation (4) shows the stress distribution and strength relations between different size specimens and parts. Using a typical standard test result, the strength of another specimen or a part with a more complicated configuration can be estimated.

As shown in Table 1, Weibull's coefficient m in equation (4) has a dispersion which depends on the specimen, but as mentioned

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in the previous chapter, Weibull's coefficient of the bending test 9.2 is used as a material constant.



Figure 6. The relationship between effective volume and strength

Figure 6 shows the relationship between effective volume and breakage strength for each test result. The mean strength and dispersion are shown for each result. The black circle in the table represents a specimen which is precut from a plate and with the whole surface polished. The hollow circle represents a specimen for which only the portion where stress concentration occurs is polished. This means that only the notch edge of a notched specimen, the hole edge of a specimen with a hole, and the core of a rotation specimen are polished. The triangle represents a specimen that is baked only. Although these three different kinds of specimens have different surface situations, each of them has equal effective volume. In order to show the dispersion of each specimen's strength, Figure 6 is distinctly plotted. Also, 303P in the figure is the result of the 3-point bending test with the 3 mm square bar. The bias going through the ava. 2 3 ] 3 P result (black mark) is a line which sows the relationship between the

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effective volume and the mean strength acquired from equation (4), using the result of the bending test which was employed as a standard strength test. As defined in equation (4), the grade of the bias is -1/m (where m = 9.2). The bias going through the above mentioned  $3\square 3$  P result is a line which predicts the strength of other specimens and also of parts made of the same material. Strength predictions from the bending test, as defined in Figure 6, are conservative in the case of the rotation test but unsafe for the tension test.

It is thought that there are several causes of error in strength predictions. Details are explained later, but this is still under development and cannot be helpful yet, since various settings are needed for optimal processing conditions of raw materials to machine parts.

### 4.4 Influence of surface polishing and polishing direction

Influence on strength of surface roughness is confirmed by the bending strength test which was performed on various surface roughnesses [7-8]. For high quality ceramics, an improvement of strength can be seen down to a surface roughness of 1  $\mu\text{m},$  but it is known that even if surface roughness is further decreased, strength does not improve. There is a relationship between polishing direction and strength. As confirmed by the bending test, differences in strength occur depending on whether, at the time of the test, the polishing direction and the direction of the principal tension stress are parallel or orthogonal. Compared to parallel polishing, orthogonal polishing decreases strength about 20%. This relationship betwen surface roughness and strength is a characteristic which, as mentioned above, is confirmed by the square-bar bending test. In the case of the bending test, the stress slope is large. Breakage occurs mostly from the surface on the tension side, and it is predicted that the influence of surface roughness will be large.

However, for the tensile test, stress is uniform over the crosssection and it is thought that, compared to the bending test, it is not influenced by surface roughness. This also assumes that the starting point of the breakage, which is shown as a fracture after tension breakage, often exists more inside than on the specimen's surface. As mentioned above, it is certain that the surface roughness and polishing direction of parts greatly influences their strength when they receive a load. Because of this, the surface polishing process is a very important issue, related to processing costs of manufacturing machine parts.

For the tensile samples with holes and with notches that were manufactured for this report, three different surface processing conditions for each specimen were used. For the rotation specimen two kinds of specimens were used. As defined in chart 1 and drawing 6, strength differs depending on surface processing. But for the results of the two kinds of tensile tests, the influence of surface polishing and polishing direction on strength is not clearly For example, a tensile specimen with a hole is polished shown. parallel to the direction of principal tension, but the strongest among the three types of specimens is an unpolished specimen. Although the notched specimen is polished at right angles (to the tension), among the three specimens the full-polished specimen is the strongest and, compared to the others, the partially polished specimen is very weak. As mentioned above, the direction of polishing and whether or not the specimen is polished are not considered to be related to the strength, but in the rotation test in which specimens were polished parallel to the tension, there was a noticeable effect from the polishing. The reason why polishing affects specimens differently should be studied. However, judging from all these results, it is assumed that for a part which has a rather small radius of curvature, strength is not much affected by whether or not the part is polished.

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### 5. PLANNING

Since the causes of dispersion to the predicted strength of hot-pressed silicon nitride and reaction sintered silicon nitride [2] are different from the previous report [1], this leads to the important fact that strength prediction using Weibull's coefficient and the mean strength acquired from the bending test becomes very dangerous for tension test predictions. In the case of hot-pressed material, compared to other sintering methods it appears to be easier to set the conditions at the time of sintering, and since shrinkage does not occur in reaction sintered material during sintering, the material is not affected too much by the complexity of its configuration. However, since pressureless sintered material shrinks 10-20% at the time of sintering, it is thought that strength changes depending on the configuration of the sintered body. Pressureless sintered materials undergo two baking processes, which are temporary baking and proper baking, after the formation of feed powder. Specimens in this report, however, are processed to dimensions that include anticipated shrinkage during proper baking after the temporary baking. Since only surface polishing is carried out after proper baking, there is a difference in the dimensions of specimens which are not processed after proper baking and there are some bends and deflections. The cause of deformation at the time of baking is thought to be heterogeneity of the material, or a difference in the coefficient of contraction caused by crystal anisotropy, the temperature of the sintering furnace, atmospheric dispersion, etc. At this level, it is currently unreasonable to expect uniform shrinkage in the process of baking. If aeolotropic shrinkage is carried out, it is assumed that at the time of sintering, internal stress will naturally occur and that residual stress will remain inside after sintering. Also, cracks might occur, being caused by internal stress. The reason why the strength of specimens with holes or notches is unrelated to whether or not surfaces are polished or to the direction of polishing, is thought to be that residual stress is caused by the occurrence of

the above aeolotropic shrinkage and that cracks occur because of a deformed condition.

Large defects which did not appear in specimens of simple configurations were introduced and because they were defects which cannot be eliminated by polishing, then, unrelated to polishing, the results showed lower strengths than predicted. In the case of a simple specimen a) in Figure 1, for a tensile test sample, unlike the above specimens, it is predicted that the occurrence of defects from deformed conditions is small and influenced strongly by making the direction of polishing at right angles to the tension, and strength becomes lower than the predicted strength. The reason why the predicted strength of rotation specimens is often correct is that, compared to the holed and notched tensile test specimens, the dimensions are larger. Therefore, the effect of deformed conditions at the time of sintering is tempered. Also, as the direction of polishing is parallel to the direction of the principal tension stress (circumferential for a circular plate), defects which occur in the sintering process and the effect of the surface polishing are similar to the situation of bending test samples. Polished and unpolished samples are employed for the rotation test, Although when comparing the strength of both kinds of samples. the mean strength of the polished specimen is larger than the unpolished specimen, the dispersion of the specimen which is only sintered is smaller. In this case, polishing is useful for raising mean strength but, on the contrary, it makes the dispersion of strength larger.

As mentioned above, specimens with holes and notches, which have rather small curved edges, are predicted to have different sizes of defects and different distribution situations compared to other specimens. Namely, since defects in the material to which Weibull's statistics are applied are different between simple specimens and deformed specimens, the same breakage probability distribution function may be applied to some unreasonable points. Therefore, there might be substantial contradictions in predicting the

strength of parts with complex configurations from the results of the simple bending test. Although it seems that the relationship between the complexity of configuration and the strength of a part has not been systematically studied, it is informative that, regarding the result of a rotation test for a complex configuration radial turbine rotor [9], a large error is produced if the rotor's rotation strength is predicted by using the result of bending test of the same material. The breakage probability of rotor strength does not follow Weibull's distribution, and there are two distinct groups which are low strength and high strength. It has been confirmed that each group's breakage probability follows Weibull's distribution. In order to plan to manufacture a complex part by the pressureless sintered method, there are some unreasonable /9 points in using the results of strength tests which were carried out on simple configuration specimens. A strength test should be performed using specimens for which the effects on complex parts can be newly studied. Considering the manufacturing cost of samples and the ease of the tests, the bending test of a square bar with shoulder, and the bending or tensile tests of holed or notched specimens used in this report, are pretty reasonable methods. Of course, there will be no problem if sintering techniques are improved and new materials developed so that complex parts will acquire the same strength characteristics as simple parts.

### 6. CONCLUSIONS

A strength test applied for machine parts was performed on pressureless sintered silicon nitride, a material expected to be used in the future for complex machine parts. The specimens with holes and notches used in this report are applied stress concentration parts which are commonly found among machine parts. Not only changes in stress concentration were questioned; the influence of the complexity of the configuration on the strength of sintered parts was also made a subject of this investigation. Weibull's statistical analysis was applied to the result of each test.

As a basic test, the bending test was used for strength evaluation and the following results were acquired:

- Compared to the strength predicted from the results of the bending test, the tensile strength of the notched and holed specimens is very low.
- The strength of the above mentioned specimens is not related to whether or not the specimen surface is polished, or to the direction of polishing.
- 3) The results of the rotation test correspond to the results of the bending test and match predicted strength.
- 4) It is assumed that, when comparing tensile tests of holed specimens and notched specimens and rotation tests of rings, there is a possibility that the size of a defect or a difference in distribution could dominate strength, depending on radius of curvature of the circular portion. From the result of this study, it was assumed that there was a definite difference between a 5-6 mm radius of curvature and a 30 mm radius of curvature.
- 5) Holed and notched parts have a small configuration change. However, the fact that the strength of parts which have these stress centralizations does not correspond to the result of the bending test remains an important subject for parts manufacturing even though these materials are under development now.

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