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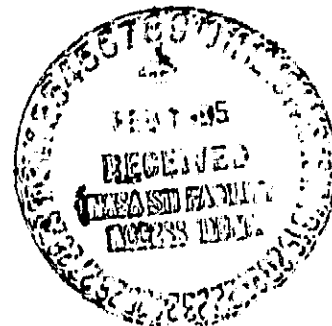
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SILICON NITRIDE AT ELEVATED TEMPERATURE

Katsutoshi Matsusue, Yoshiaki Fujisawa and Kitao
Takahara

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16. Abstract Uniaxial tensile strength tests of pressureless-sintered silicon nitride were carried out in air at temperatures ranging from room temperature up to 1600 C. Silicon nitrides containing Y2O3 · Al2O3, Al2O3 · MgO or MgO · CeO2 additives were tested. The results show that the composition of the additive used influences the strength characteristics of the silicon nitride. The tensile strength rapidly decreased at temperatures above 800 C for the materials containing MgO as the additive and above 1000 C for the material with Y2O3. When the temperature increased to as high as 1300 C, the strength decreased to about 10 percent of the room temperature strength in each case. Observations of the fracture origin and of the crack propagation on the fracture surfaces are discussed.			
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TENSILE TEST OF PRESSURELESS SINTERED SILICON
NITRIDE AT ELEVATED TEMPERATURE*

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Katsutoshi Matsusue, Yoshiaki Fujisawa and Kitao Takahara**

1. PREFACE

Ceramics which have been developed for high temperature materials include silicon nitride, silicon carbide and sialon etc. Many studies about silicon nitride have been performed at many organizations as silicon nitride has the highest strength at room temperature among the above materials, and it has been expected to be a new heat-resistant material as a substitute for a heat-resistant alloy, and application studies for practical parts have been proceeding. This laboratory has also performed strength evaluation at room temperature for these new high-strength ceramics in the past few years by using silicon nitride and pressureless sintered silicon carbide, etc., which were hot pressed, pressureless sintered and reaction sintered. And mechanical characteristics in the case when ceramics are used for strength parts have been studied [1]. The hot press method has the highest strength among the three kinds of sintering methods, and simply configured parts can be manufactured by this hot press sintering method. But, it is difficult to manufacture the parts which have complex configurations, so it is not suitable for the manufacturing of general machine parts. It, however, seems that when hot press materials are specimens, they will be able to be used for simple parts as the materials become comparatively homogenous. On the other hand, the parts which were manufactured by the pressureless sintering method and the reaction sintering method have lower strengths compared to the parts manufactured by the hot press method, but it is possible

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to manufacture complex parts rather easily. Especially, it is widely anticipated that pressureless sintered silicon nitride will become the future material for complex machine parts since there is a possibility for it to obtain a strength which is close to that of the hot press materials by choosing a suitable sintering assistant and by improving the sintering technique. Therefore, we researched in this report the characteristics of the strength of pressureless sintered silicon nitride at elevated temperature in the present developmental stage by tensile and breakage tests.

The silicon nitride which is a subject of this test is generally called a slow sintered body, and it always requires the use of an additive as a sintering assistant for the usual sintering. And the strength characteristics differs depending on the kind of the sintering agent, and the various kinds of sintering assistant which are used. It is known that the selection of these sintering assistants greatly influences the strength of the sintered body to the same degree as sintering technique.

Three kinds of materials, which are representative among silicon nitride sintered bodies and which have been made public, were applied for test materials in this report. The main component of the three kinds of materials is silicon nitride (Si_3N_4) but they are distinguished depending on the kind of sintering assistant. They are the yttria alumina type ($\text{Y}_2\text{O}_3 \cdot \text{MgO}$) and magnesia ceria type ($\text{MgO} \cdot \text{CeO}_2$).

Regarding the strength characteristics of pressureless sintered silicon nitride at elevated temperature, the results from room temperature to 1300-1400°C by bending test has already been reported [2]. But, the bending test is a test method which shows the highest strength of the strength tests, and the decrease of the strength at elevated temperature is also pointed out in the report. But, it is shown that pressureless sintered

silicon nitride has quite a bit of strength even at 1300°C, and it gives an impression that the material may be anticipated to be useful as strength parts even at a high temperature of 1300°C. In fact, it seems that an outlook of the above has been received in every field.

The above was recognized by the results of the bending test. There is a situation that it is easier to accept the tensile test than the bending and strength tests as the strength data of materials for the machine designer who is considering the application of a silicon nitride sintered body to practical machine parts. Also, it is convenient for comparing to conventional metal materials.

Under the above condition a tensile test for pressureless sintered silicon nitride at elevated temperature was performed in this report, and strength characteristics of samples at elevated temperature was researched from the users point of view. According to the result of the test the following things were recognized. Tensile strength almost does not change from room temperature to 800-1000°C, but if the temperature becomes higher than this, the strength decreases rapidly. Also, depending on the kind of sintering assistant, the temperature at which the strength begins to decrease differs somewhat. In this result, the strength decreased when the temperature exceeded 800°C for the two kinds of materials for which the magnesia system was used, and 1000°C for the materials of the yttria system. And, when the temperature is over 1300°C, all materials become less than 5 kg/mm², and the value is less than 1/10 of the bending strength at room temperature. Also, for the result where this data is compared to heat resistant alloys which have been used, heat resistant alloy is much stronger until 1000°C, and the temperatures at which ceramics are superior are over 1100°C, but the strength at these temperatures is less than 15 kg/mm². Since this value is a mean value, if variance is considered, it is thought that this value is extremely low for use as strength materials. In addition to the above result, we

performed fracture observations after testing for all of the tensile samples, the characteristics of fractures at each temperature was studied, and the situation of the destruction and crack growth was examined.

There are very few embodiments for the tensile test for ceramics especially the test at elevated temperature, and standardization of specimen has not yet been performed. It is thought that the test equipment, installation tools, specimen configuration, etc. will be able to be supplied for reference work in the case when standardization of the tensile test is performed in the future.

2. SAMPLE MATERIALS AND SPECIMEN

Three kinds of pressureless sintered silicon nitride were used for sample materials. Silicon nitride (Si_3N_4) is the main component for each material, and yttria alumina ($\text{Y}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$) alumina magnesia ($\text{Al}_2\text{O}_3 \cdot \text{MgO}$) and magnesia ceria ($\text{MgO} \cdot \text{CeO}_2$) are used for the sintering assistants. Component ratio of assistant to each material is about 10%. Table 1 exhibits the main material characteristic values of each material.

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TABLE 1. Main material characteristic values of silicon nitride (per sintering assistant)

Items	Materials	$\text{Y}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$ system	$\text{Al}_2\text{O}_3 \cdot \text{MgO}$ system	$\text{MgO} \cdot \text{CeO}_2$ system
Density	g/cm^3	3.18	3.19	3.0
Young's modulus	kg/mm^2	2.8×10^4	2.7×10^4	2.5×10^4
Linear expansion coefficient	$1/^\circ\text{C}$	3.2×10^6	3.3×10^6	3.3×10^6
Coefficient of thermal conductivity	$\text{Kcal/mh}^\circ\text{C}$	12.6	9.4	25
Specific heat	$\text{cal/g}^\circ\text{C}$	0.17		0.18
Specific resistance	$\Omega \cdot \text{cm}$	$>10^{12}$	$>10^{14}$	—
Destructive toughness K_{Ic}	$\text{kg/mm}^{3/2}$	16-19	18.9	16-17

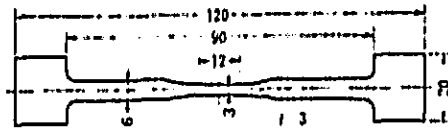
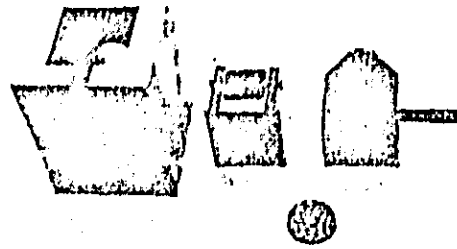


Figure 1. Tensile specimen



Picture 1. Installation tools for a three-point bending test

Bending specimen of 3 x 4 x 50 mm and the tensile specimen shown in Figure 1 were used. The bending samples were applied for the JIS (Japanese Industrial Standard) and they are standard samples to indicate the strength of ceramics and they are used at room temperature in this experiment. All of the specimens were cut from sintered plate and polished and the surface roughness is $1\mu\text{m}$ (R_{max}). Namely, 0.1 to 0.2 mm was planed off of the edges of the specimens. The configuration of the tensile samples was determined whether the equipment can be easily installed, whether fractures occur in the test region and whether manufacturing cost is cheap, etc. The fracture test region is a central parallel part (3 x 3 x 12).

3. EXPERIMENTAL EQUIPMENT AND METHODS

3.1 Bending test

4 mm width, 3 mm height and 50 mm length rectangular bars were used for specimens for a bending test, and a three point bending test was performed with 30 mm distance between supporting points. Picture 1 exhibits the installation tools for the bending test. Since the bending test has become a standard strength test for materials, 50 bars were used for each material. A shimazu universal testing machine (RS-2 type) was used for the test and loading was performed by hand. Crosshead speed at that time was about 0.5 mm/minute.

3.2 Heating equipment

There are many cases in which the strength at elevated temperatures, of the ceramics which are used for high strength materials is not much different from the strength at room temperature generally until about 1000°C. Therefore, in order to study the characteristics of the strength at elevated temperature, it is necessary to have the experiment at an elevated temperature of at least 1300°C. In this experiment an image furnace (manufactured by Shinku Riko) in which infrared rays were used was applied after studying various kinds of furnaces. And, a portion of the furnace was remodeled in order to be able to perform a tensile test at an elevated temperature up to 1600°C. Figure 2 exhibits a cross-section of the furnace which was used and a schematic drawing at the time when the furnace was used. The structure of the furnace is that four infrared ray lamps are installed at one focus of an elliptic reflection surface made of aluminum which was cooled with water and the light of the lamps is converged at the other focus and the specimens which were put at the focus are heated. Gold plating is put on the elliptic reflection surface and the efficiency of reflecting the infrared rays is raised. The diameter of the central ignition part converged is about 10mm, the effective length of the lamps is 50mm. Since the heating part of the furnace is that the vertical direction (perpendicular direction toward the paper in Figure 2) is open toward the outside air, the heat radiated by convection was decreased by installing a metallic thermal insulation board in the top and bottom of the furnace.

Generally, the bending test is often performed for strength test at elevated temperature, and a closed type furnace is often used for heating equipment. Since the installation tools are heated at the same time as the specimen for this closed type furnace, the characteristics of the the resistance also must be

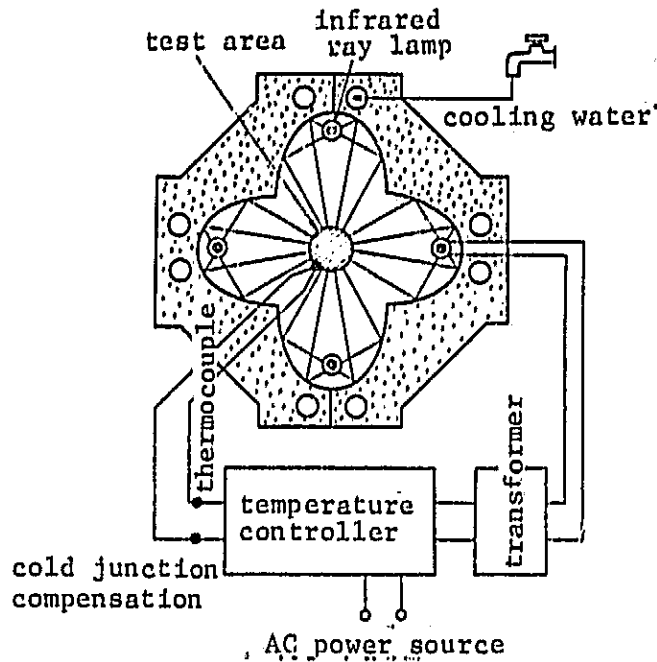


Figure 2. Heating equipment

considered such as by manufacturing the installation tools with black lead, etc. Also, it is necessary to heat the furnace for a long time to elevate the temperature of the atmosphere of the inside of the furnace. But, there are some advantages in the image furnace of infrared rays applied in this report. For example, it can be heated partially such as only the test area of the specimens, so it does not spend much energy for heating. Also, since the temperature can be elevated in a short time, experiments work well. But, it is not suitable for the case when the installation tools are put in the test region such as in the bending test and also for the case when it is applied for a specimen with complex configuration or large specimen.

3.3 Temperature measurement

It is considered that measuring temperature of the material which was partially heated over 1000°C is comparatively difficult. In the case when it is heated by radiation from infrared ray lamps such as this report, it is thought that the influence of

the temperature difference between the surface and inside of the specimen, or the difference of indicated temperatures from a difference in the thermocouple installation method appears as a measurement difference. The method of measuring by coiling the thermocouple around the samples was applied in this report, but tests were compared in the case when a thermocouple was imbedded after a hole was made on the specimen in order to study whether or not the measurement method is suitable. As a result, it was confirmed that the difference of those methods was about 1%. Also, the longitudinal temperature distribution of the specimen was also studied and it was confirmed that it is heated almost to a homogeneous temperature in the range of about 30 mm in the central part. Namely, PR 13 (platinum and the platinum which includes 13% rhodium) was used for thermocouple. A digital thermometer (Takeda Riken Industry TR-2112A) was used for a temperature indicator. A programmed temperature controller with a furnace was used for the establishment of a rate of temperature increase from room temperature to the test temperature and the control of maintaining test temperature. The thermocouple which is used as a sensor for the temperature controller is different from the one which was coiled around the specimens; it is located in the position which is about 20 mm away from the center of the furnace. The indicated temperature value of the thermocouple for this control is about 1/2 of the temperature of the specimen. The actual test temperature can be assumed by the thermocouple which was used for the controller, but since this thermocouple is located away from the center of the furnace, there is a possibility that the indicated temperature changes because of even a slight movement. On the other hand, there is a possibility that the thermocouple which was coiled around the specimens may be cut at the same time as a fracture of the specimen. If this is also used as a controller, the control of the temperature after cutting becomes impossible. Therefore, the thermocouple for control is not also used as the thermocouple for measuring temperature, and the test temperature was measured by using the thermocouple coiled around samples.

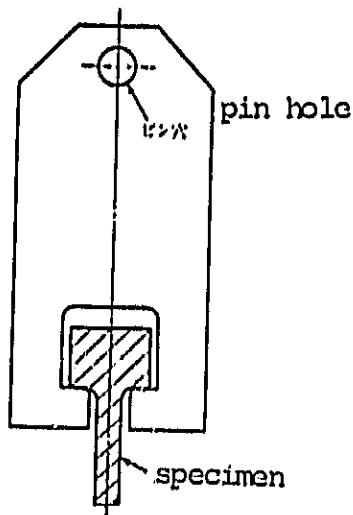


Figure 3. Tensile test

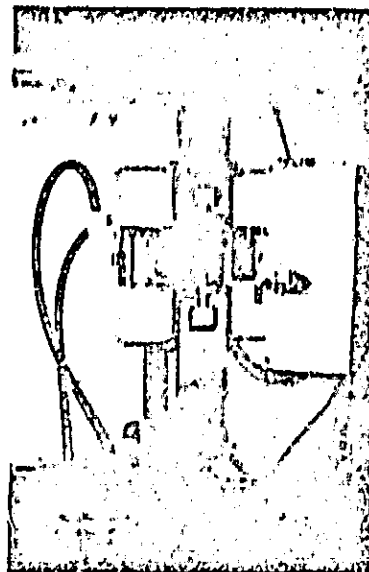
3.4 Tensile test

It is important to accurately add a uniaxial tensile load for a tensile test, but it is difficult to do that for ceramics which are brittle materials.

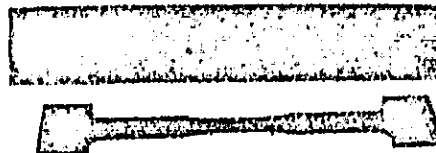
Loading method by hanging the specimen

on the chuck part of the installation tools made of mild steel through a crosspin coupler was applied in this experiment, as shown in Figure 3. The method which uses a pin couple was applied also in the previous report [1]. At that time, it was almost confirmed that a uniaxial tensile test could be performed by measuring the deformation of both surface of the specimen.

Also, in the method of hooking the specimen on the chuck part of the installation tools, there is a possibility that destruction will occur from the part which is in contact with the installation tools. Because of that the pressure of the contact area was made uniform by putting aluminum foil between the equipment and destruction at the contact part was prevented. The test was



Picture 2. Installation condition of specimen



Picture 3. The specimen for a tensile test and the installation condition of the thermocouple

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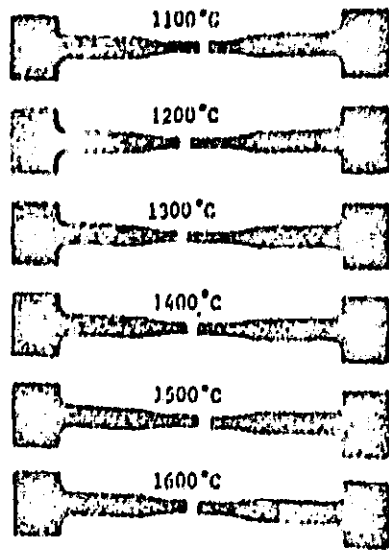
performed by the loading method by installing specimen on the installation tool and loading by installing in the furnace. The supporting stand of the furnace is separated from the testing machine, so special attention was paid to the adjustment of the relationship between specimen and furnace, and the specimen was made to be located in the center of the furnace. Picture 2 exhibits the installation condition of the specimen.

The universal testing machine which was mentioned before was used for a tensile test. Loading was performed manually. Loading speed is about 0.3 mm/minute as a crosshead speed. Some degree of rising temperature elevation of the specimen can be chosen arbitrarily by the temperature controller. The specimen in this experiment was heated at the rate of about 300°C/minute.

The procedure of the tensile test with elevated temperature in detail is as follows:

First of all, for preparation, a 0.2 mm width PR 13 thermocouple is coiled in a spiral at the central part of the specimen (test region), and the coiled thermocouple is tied to three points of the central parallel part by using other platinum lines to affix the coiled thermocouple (Picture 3).

The specimen is installed on the installation tool for which the position was adjusted beforehand and the ends of the thermocouple were connected with a digital type thermometer through a hole installed on a thermal insulation board. After preparing everything, the furnace power is turned on and temperature is raised until the set temperature at a constant rate of temperature increase (about 300°C/minute). Since the indicated temperature is unstable right after the temperature has reached a fixed test temperature, we will wait for 1-2 minutes until the digitally indicated temperature becomes stable, and after that the tensile load is added. Loading is performed at a constant



Picture 4. Representative example of the specimen after destruction (the case of $Y_2O_3 \cdot Al_2O_3$ type)

crosshead speed (about 0.3 mm/minute) and the power of the furnace is cut immediately after fracture and the highest load is read. When the two halves of the furnace are opened, the temperature of the specimen decreases rapidly, and it becomes almost room temperature after 10-15 minutes. The time which is required for one test is about 15 minutes. The load of each specimen at the time of fracture was read by the above procedure and the destruction strength at the time of high temperature was found. Picture 4 exhibits a representative example of the specimen after destruction.

4. EXPERIMENTAL RESULTS AND EXAMINATION

4.1 Bending strength

Table 2 exhibits the result of the bending strength test which was performed at room temperature by using 3 x 4 x 50 mm rectangular bars.

Since three kinds of sintered bodies which have different sintering assistants were used, the table shows results according to each assistant. Although in most cases the surface of the specimen was polished parallel to the longitudinal direction, only in the case of the alumina magnesia type material among three kinds of material a specimen was polished parallel to the longitudinal direction and a specimen which was polished at a right angle were used. However, Table 2 exhibits only the

TABLE 2. Strength of pressureless sintered Si_3N_4 at room temperature.

- 3 σ_{b3p} strength by 3 point bending (kg/mm^2), m; Weibull coefficient;
 4 V_{e3p} effective volume (mm^3) of the specimen for 3 point bending;
 5 V_{eT} effective volume (mm^3) of tensile specimen;
 6 σ_{bT} tensile strength (kg/mm^2)

sintering assistant	σ_{b3p}	m	V_{e3p}	V_{eT}	σ_{bT}	predicted value of tensile strength
$\text{Y}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$	52.9	5.3	4.5	108	26.6	29.0
$\text{Al}_2\text{O}_3 \cdot \text{MgO}$	51.3	12.8	0.95	108	34.3	37.5
$\text{MgO} \cdot \text{CeO}_2$	72.2	11.5	1.2	108	45.2	40.2

result of the parallel polished specimens. All of the results of the parallel polishing test and right angle polishing test are shown in Table 3. The resulting strength of the specimen which was polished perpendicular was about 17% less than the strength of the specimen which was parallel polished. The Weibull coefficient m shown in Table 2 is a coefficient to describe the variance of the data which was found under the assumption where the obtained strength data conforms to the Weibull probability distribution coefficient. And it was calculated by the maximum method the same as in the previous report [1].

Also, Weibull coefficient m is used for V_{eT} of Table 2 and it is an effective volume [3] of the specimen for the three point bending test which is calculated by the following equation:

$$V_e = \int_p \left(\frac{\sigma}{\sigma_b}\right)^m dv \quad (1)$$

Here, σ and σ_b are the stress and the maximum stress in the test region respectively and integration is performed only in the region of $(\sigma > 0)$

Different values of bending strength were obtained depending on the sintering assistant. Especially, the $\text{MgO} \cdot \text{CeO}_2$ type material became high value. This is not simply the influence of the assistant, but it is related to every condition at the time of sintering: so, whether or not the material is good, and also whether or not sintering assistant is suitable, cannot be determined only from this result. Also, the fact that the Weibull coefficient of $\text{Y}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$ type material became an extremely

TABLE 3. Destruction strength data
(three point bending test) (dimension of specimen; 3x4, span 30mm,
unit (kg/mm²))

(Y ₂ O ₃ ·Al ₂ O ₃ type Si ₃ N ₄)					(MgO·CaO type Si ₃ N ₄)				
50.0	38.1	54.4	73.1	38.8	70.0	73.8	75.0	76.3	60.0
43.8	36.3	60.0	45.0	58.8	72.5	80.0	66.3	72.5	66.9
42.5	56.9	43.8	43.8	46.3	71.9	70.0	74.4	76.9	80.0
54.4	67.5	63.1	49.4	61.9	63.1	68.1	62.5	67.5	70.6
80.0	63.6	60.4	61.9	68.8	74.3	82.1	79.1	78.0	78.4
54.6	30.4	65.4	43.3	31.4	73.0	75.0	81.1	87.6	75.0
62.0	49.2	63.0	61.8	49.5	52.1	82.3	70.0	65.1	77.3
37.9	46.8	51.4	53.3	68.5	71.5	68.9	71.9	69.3	66.8
67.1	64.1	51.1	40.9	48.4	86.0	79.5	68.1	63.3	62.3
59.8	40.8	53.8	50.9	38.4	69.8	76.3	72.4	74.4	86.3

mean value 52.9, Weibull coefficient 5.3 mean value 72.7, Weibull coefficient 11.5

(Al₂O₃·MgO type Si₃N₄) (Al₂O₃·MgO type Si₃N₄)
the one polished in the direction of the main stress the one polished perpendicular to the main stress

55.0	56.3	58.1	51.3	55.0	40.0	38.8	45.0	47.5	37.5
51.9	45.6	57.5	53.8	47.5	40.6	37.5	40.0	43.8	45.0
54.5	55.0	45.0	54.1	45.0	39.4	39.6	50.3	51.6	41.9
51.1	54.9	63.1	59.3	58.6	50.5	47.5	41.1	50.4	49.0
63.5	56.3	52.6	58.7	53.9	52.3	47.0	51.0	42.5	52.0

mean value 54.3 Weibull coefficient 12.8 mean value 44.9, Weibull coefficient 10.3

small value among the three kinds of material is not related to the assistant, and it is considered that there was probably a problem in setting a condition at the time of the sintering process. Selection of sintering assistant is important for manufacturing hard sintered material like silicon nitride, but setting and managing every sintering condition during the sintering seems to exert an important influence toward the strength characteristics of materials.

Table 3 exhibits all of the results which were obtained in the bending test and in the tensile test which is mentioned in the next section.

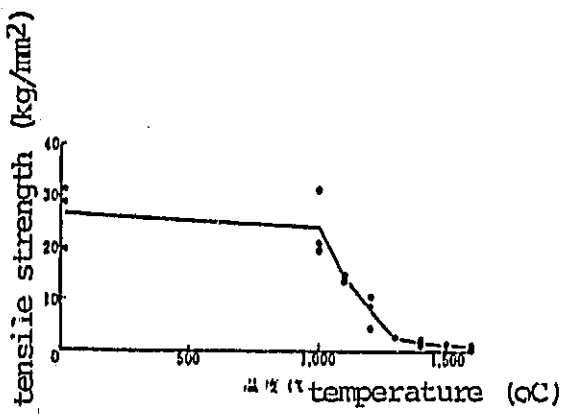


Figure 4. Tensile strength of Si_3N_4 for which $Y_2O_3 \cdot Al_2O_3$ is sintering assistant

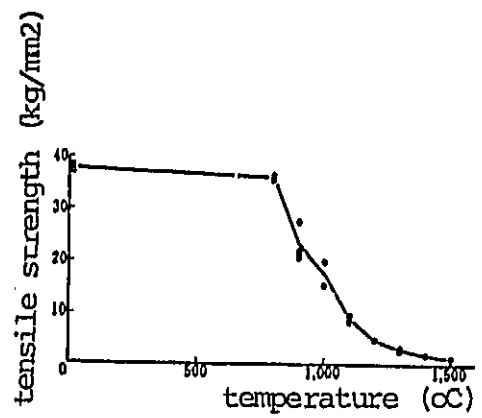


Figure 5. Tensile strength of Si_3N_4 for which $Al_2O_3 \cdot MgO$

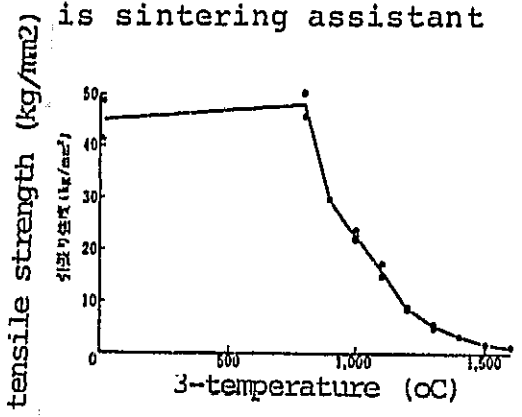


Figure 6. Tensile strength of Si_3N_4 for which $MgO \cdot CeO_2$ is sintering assistant

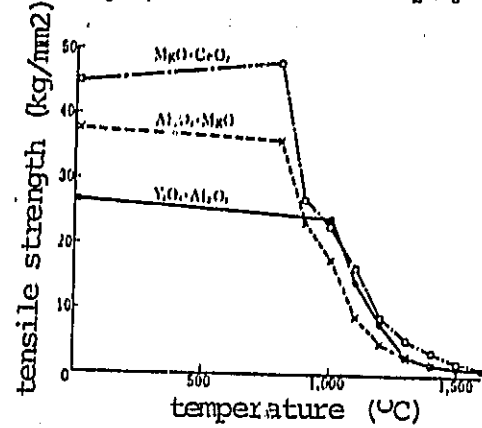


Figure 7. Tensile strength of pressureless sintered Si_3N_4

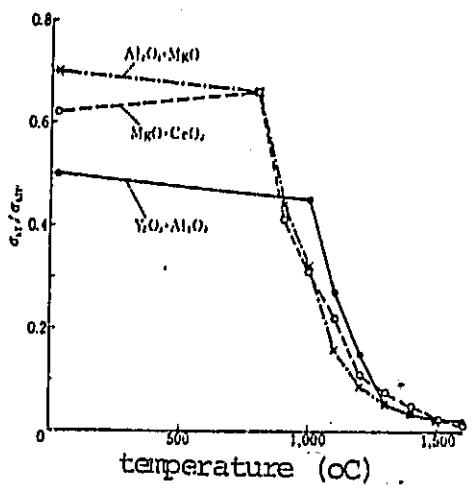
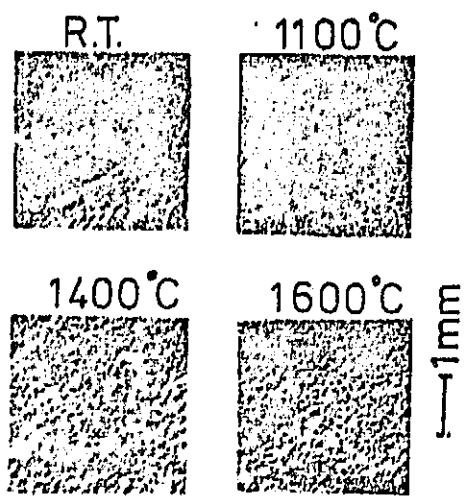


Figure 8. Tensile strength of pressureless sintered Si_3N_4 described by a comparison with bending strength at room temperature



Picture 5. Expanded pictures of fracture (in the case of $Y_2O_3 \cdot Al_2O_3$ type)

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4.2 Tensile strength

Table 2 exhibits tensile strength at room temperature to compare with bending strength. 2-3 mean values are shown in the table for each material. Effective volume V_e in the table is the volume (3 x 3 x 12 mm) of the central parallel part of the specimen. In the case of the tensile test, the stress distribution of the test area becomes uniform, so the integral of equation (1) becomes equal to the volume of the test area. The result of the bending test was used for the predicted tensile strengths of Table 2, and it is a value which was calculated by the relative equation of the effective volume, described by the following equation, and strength [3].

$$\frac{\sigma_{b1}}{\sigma_{b2}} = \left(\frac{V_{e2}}{V_{e1}}\right)^{1/3} \quad (2)$$

Here σ_b and V_e describe strength and effective volume respectively and subscripts 1 and 2 show different kinds of specimens. Since the bending test was used for the standard test in this report, the value which was obtained by the bending test was used for the σ_b value. As is clear in the table, the predicted value of the tensile strength for any material is about 9% higher compared to the experimental result. Prediction is dangerous from the point of view of designing. However, it is common that the results of the test at room temperature have a large variance. Since the number of samples is small, the above statements, such as prediction is dangerous, etc., are not entirely accurate conclusions.

Figures 4, 5 and 6 exhibit the tensile strength of each material in the case when the temperature is changed from room temperature to 1600°C.

The continuous lines for each figure are lines which link the mean strengths at each temperature in straight lines. In the case of the sintered bodies when magnesia (MgO) was used for sintering assistant as in Figures 5 and 6, there is almost no change of the strength from room temperature to 800°C. But,

when the temperature becomes 900°C, the strength definitely decreases. The sintered body for which yttria (Y_2O_3) is assistant shows the same strength as at room temperature until 1000°C. The strength rapidly decreases for any material after the temperature exceeds a certain temperature limit, and when temperature becomes 1500-1600°C, destructive stress becomes about 1 kg/mm². Figure 7 is a comparison of the relationship between the mean strength and temperature for the three kinds of material at each temperature. /8

The variance by the material from room temperature to about 1000°C is especially remarkable, but it is considered that this is caused by each condition and process, etc. at the time of sintering. Figure 8 exhibits the mean value of tensile strength of each material compared with the bending strength in a room temperature. It is considered that bending strength at room temperature is basic data for evaluating the strength of materials. The test method is specified also in JIS, and bending strength is always required for the step of developing material at every material manufacturer, and it is printed as a catalog value. Therefore, it is convenient to describe tensile strength at elevated temperature by a comparison with bending strength as in Figure 8 as the tensile strength at elevated temperature can be assumed from the catalog value from the point of view of using materials. Also, it is convenient for the comparison of the strength characteristics at elevated temperature toward the strength of each material at room temperature.

As is clear in the figure, there is a difference in the strength characteristics from room temperature to around 1000°C in the case when MgO is used and when Y_2O_3 is used for sintering assistant. But, the tensile strength becomes less than 1/10 of the bending strength at room temperature. It is considered that the reason why strength decreases extremely when the temperature becomes high such as this is that, the sintering

assistants Al_2O_3 , MgO , CeO_2 , Y_2O_3 , etc. used at the time of sintering exist at grain boundaries at a glassy layer, and when this grain boundary layer becomes a high temperature which is over 1000°C , it becomes soft. For strengthening grain boundaries, the method which crystalizes this grain boundary glassy layer is applied for hot press Si_3N_4 [4], but, it has not been realized for pressureless sintered Si_3N_4 . Sintering without using assistant is difficult in either case, so it is considered that unless the selection of assistant and the grain boundary strengthening method are improved and unless the materials strength does not decrease much at elevated temperature, it is difficult to use ceramics as strength parts at elevated temperature.

4.3 Observation of tensile fracture

The fracture of the material which causes destruction with brittle characteristics often has some kind of trace which becomes an origin of crack propagation. Important hints to solve the strength characteristics of materials are often given from the base point of their destruction and the configuration of the fracture. Picture 5 is a representative example of fracture after tensile destruction. This picture is the case for sintering assistant to be Y_2O_3 type material. Also, Picture 6 exhibits the examples of other fractures. Entire fractures of the specimens which have 3 x 3 mm cross-sections are pictured. As is shown in Picture 5, the characteristics of the fracture vary depending on test temperature. The fracture at room temperature has remarkable |||| , which is a typically brittle destructive characteristic and it is especially seen in high strength ceramics. In the case of this picture, it seems that the destruction exists at the position close to the upper right corner part. In the case when the test temperature is from 1000 to 1200°C , the entire fracture becomes flat like the upper right picture. And if the temperature becomes higher than that

/9

Picture 6. Expanded pictures of tensile fracture

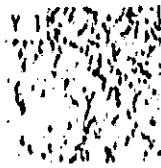
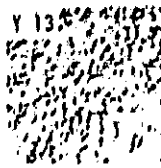
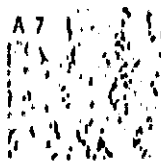
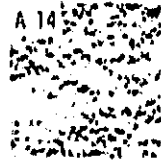

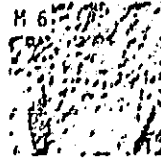
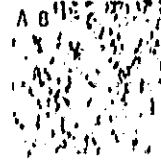
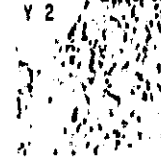
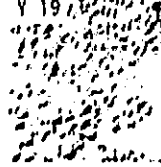

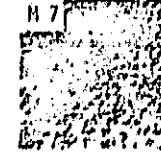
Room temperature	Y 4	Y 17		A 19
	A 20	M 15	M 16	
800°C	A 17	A 18	M 14	M 18
900°C	A 15	A 16	M 17	
1000°C	Y 8	Y 9	Y 15	A 2
	A 10	M 1	M 8	M 12

Y: Si_3N_4 for which sintering assistant is $\text{Y}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$;
 A: Si_3N_4 for which sintering assistant is $\text{Al}_2\text{O}_3 \cdot \text{MgO}$;
 M: Si_3N_4 for which sintering assistant is $\text{MgO} \cdot \text{CaO}$;
 Mark;
 number: number of specimen

ORIGINAL PAGE
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1100°C	Y 7	A 4	A 11	H 2
	H 9			
1200°C	Y 6	Y 11	Y 16	A 5
	A 12	H 3	H 10	
1300°C	Y 5	Y 12	A 6	A 13
	H 4	H 11		

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

1400 °C				
				
1500 °C				
1600 °C				

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(1400°C) small 凹凸 appear on the fracture and at the temperature of 1600°C small 凹凸 spread out in the entire fracture. Regarding the changes of the above fracture, there is the same tendency in materials of MgO type. It is considered that the configuration of this kind of fracture is related not only in the temperature itself but also to the strength of materials at room temperature, loading rate, the crack propagation rate, and softness of grain boundary layer at elevated temperature.

The fracture of the material with high strength has a part called a mirror around the origin of destruction which is comparatively flat, and as cracks become bigger 凹凸 becomes remarkable. But, it is known that entire fractures of the materials which have comparatively low strengths become flat [1]. Since the materials which have high strength have a large strain energy accumulated until right before destruction, when the strain energy is released by the occurrence and progress of cracks, the amount which is released as dynamic energy increases and the progress rate of cracks increases. On the other hand, it is theoretically required that a limiting value (60% of propagation rate of transverse wave) of material itself exists [5], and it is experimentally recognized that cracks branch when the progress rate of cracks reaches this limiting rate [6]. Therefore, it is considered that the fracture obtained in this experiment at room temperature had a big hackle formed 凹凸 on the fracture because of the branches accompanied by the progress of cracks. And it is considered that the reason why the fracture at 1000-1200°C became flat was that material strength decreased because of the elevated temperature, and the energy which is released at the time of destruction was small. Because of that destruction occurred without the occurrence of cracks. Furthermore, when the temperature becomes high, such as over 1400°C, the softness of the glassy layer which form the grain boundaries proceeds. And as the strength decreases more and cracks selectively progress in the grain boundary with smaller strength, a small 凹凸 must have appeared on the fracture. It is assumed

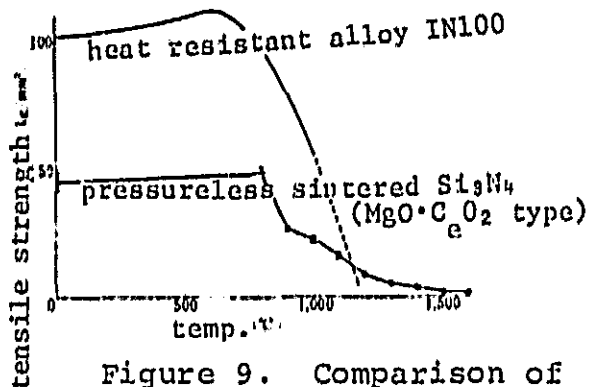


Figure 9. Comparison of tensile strengths of heat resistant alloy (IN100) and pressureless sintered Si_3N_4

like this that the characteristics of fracture changes depending on the strength of materials, destructive energy, crack progress rate, etc. A creep fracture characteristic test of the silicon nitride sintered body at 1200°C was performed for the study where tensile fracture was observed. And the relationship between fracture time and the fracture characteristics with fracture strength was studied [7]. It was confirmed in the study

that, as the fracture has a longer fracture time, the area where cracks progress slowly becomes large. The area where cracks progressed slowly in this case is the area which occurred because cracks grew in the grain boundary softened by the elevated temperature. The fracture is formed with a small σ/σ_0 the same as the case when materials were destroyed by temperatures over 1300°C in this experiment. Namely, the progress rate of cracks in the slow crack growth area is about 10^{-6} - 10^{-4} m/sec [8] and the limiting rate where cracks branch is about 3800 m/sec [5]. And, slow crack growth is observed only by destruction at elevated temperature and the limiting rate is seen when the materials with high strength are destroyed at room temperature or at a temperature below 1000°C . In the case when materials are destroyed at a medium rate, the fracture becomes comparatively flat.

5. COMPARISON WITH HEAT RESISTANT ALLOYS

Since the development of ceramics as high temperature materials has been proceeding as a future substitute for heat resistant alloys, it is considered that comparing with the strength characteristics at elevated temperatures of the heat resistant alloys presently used is important for understanding the current situation of material development. Here, we made a

nickel type alloy IN100 [9] for a heat resistant alloy as a subject of comparison. IN100 is a material which is currently often used for the turbine part of aircraft engines. The characteristics of anticorrosion, heat resistance, creep, etc. are considered for the strength characteristic to become a subject of comparison, only the tensile strength which is a subject of instantaneous destruction was compared in this report.

The changes of tensile strength of $\text{MgO} \cdot \text{CeO}_2$ type Si_3N_4 and IN100 used in this experiment with respect to temperature are shown in Figure 9. As is shown in the figure, the heat resistant alloy has a definitely higher strength from room temperature to 1000°C .

The strength of ceramics becomes higher when the temperature becomes over 1200°C . But, the strength is below 10 kg/mm^2 . It is considered that this value is too low to use as strength parts at elevated temperature.

Although the development of pressureless sintered bodies of silicon nitride has been proceeding for the future strength parts at elevated temperature, as is shown in this result, it still has not attained usefulness as strength parts at elevated temperature compared to metals.

The above is a comparison with a heat resistant alloy which is only about tensile strength, but when it is seen as general structural strength parts, comparing it to metals regarding the severity of destructive toughness, creep characteristics, stress relaxation, stress concentration, etc., metals are definitely superior to ceramics. But, there are some advantages to ceramics such as abrasion resistance, light weight, rigidity, so it is thought that ceramics can be expected to be useful for high temperature parts which do not require much strength.

6. CONCLUSION

Tensile tests at elevated temperatures were performed by using pressureless sintered silicon nitride which is anticipated to be in future high temperature parts. The strength characteristics of this material at high temperature was studied mainly by the bending test from the initial time of the development of the materials with manufacturers of raw materials. But, the strength at elevated temperature, especially the strengths at over 1300°C are not always clearly indicated and it is thought that there were many known points about the application characteristics of these materials as the parts at elevated temperature.

Three kinds of sintered bodies which have different sintering assistants were used for this experiment. Among them it is known that strength decreases if temperature exceeds 800°C in the case of two kinds of materials for which magnesia (MgO) is assistant, and strength decreases from 1000°C in the case that the material includes yttria (Y_2O_3). As a strength decrease rapidly occurs in either material, tensile strength becomes about 5 kg/mm² at 1300°C. Therefore, in the case when pressureless sintered silicon nitride is used for a high temperature material, it was understood that the temperature is limited to below 1000°C in the current development stage. It is confirmed that the cause of the decrease of strength at elevated temperature is that the component used for sintering assistant deposits in the grain boundaries and it is softened at elevated temperature. Because of that, it will be as important in the future to choose a suitable sintering assistant as to improve the sintering method for the development of materials.

Also, regarding the method of strength testing, the bending test which is usually used at the manufacturers for raw materials uses a specimen with small dimensions and it is a test of

a small stress region. Therefore, it is considered that it is not always a good testing method in the case when we think about the application of practical parts. On the other hand, the tensile test performed in this report has a large effective volume and it is easily accepted by mechanical designers. So, the tensile test is more useful than the bending test in order to know the strength of materials and it is convenient to compare with other structural materials.

The image lamp which used infrared rays used in this report works very well as the handling is simple and it was a very convenient furnace for a tensile test at elevated temperature.

High strength ceramics has been expected to be developed for the purpose of future elevated temperature materials. Therefore, it is necessary to have additional different kinds of high temperature tests such as creep, oxidation, fatigue tests, etc. and it is desirable for more data to be reported.

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