Role of Ceramics in a
Self-sustaining Environment
R. Pampuch and K. Haberko (Editors)

DEVELOPMENT OF STRUCTURAL CERAMICS COMPONENTS AND TRANSFER TO THE MARKET

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Fine ceramics have excellent features not found in conventional materials, such as heat resistance, corrosion resistance, high strength and light weight, and are well suited for use as structural materials. NGK's structural ceramics group, led by the authors, has been developing ceramic components for structural applications in cooperation with automotive makers, gas turbine makers, machinery makers and others for over 25 years.

In starting this development, we believed that the essential followings would be for applying structural ceramics to high temperature components:

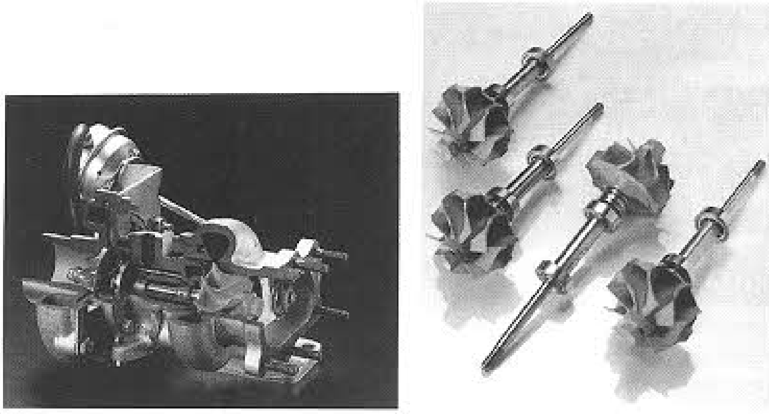
1. making clear operating conditions and required properties of materials in close cooperation with users,
2. developing test methods for evaluating required properties, accumulating a huge number of data, clarifying the mechanisms of mechanical behavior and improving the properties of materials,
3. developing design concept from the point of material properties to set the targets for the materials,
4. proving the reliability enhancement of ceramic components by field testing. Using the proposed design concept, we developed ceramic turbocharger rotors (CTR) and verified the predictions of CTR life and durability under actual operating conditions.

The success of CTR, the first ceramic component developed which rotates at high speed, and for which many ceramic scientists and engineers waited for a long time, provides a stimulus to expand the range of feasible applications of ceramic materials to many other structural components used under severe conditions. The results of our research and development, which spans from basic science, such as clarifying the failure mechanism, to engineering technology, such as design, contribute to the industry as well as the academic society.

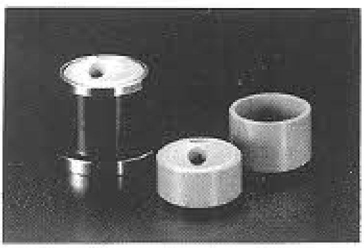
ADVANCED STRUCTURAL CERAMIC APPLICATIONS

Figure1 shows typical ceramic engine components fabricated by NGK. CTR[1,2,3] has been adopted as original factory equipment in a number of new engines by major automotive manufacturers. Using ceramics in rotors has the following advantages: improved response during acceleration, better turbocharging at low engine speeds, and increased heat resistance. An engine equipped with ceramic swirl chamber[4] met the 1987 NY US diesel particulate regulation for passenger cars. Ceramic rocker arm tip[5] marks the first original factory installation of ceramics for automobile engine valve-train parts in non-stop commercial vehicles, such as taxi cabs. Ceramic valves[6] offer high reliability.

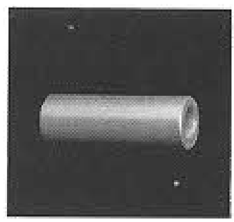
Figure 2 shows ceramic rollers and guides for hot rolling applications. Working on hot rolling lines in some ironworks, ceramic rollers demonstrated excellent performance, nonstick



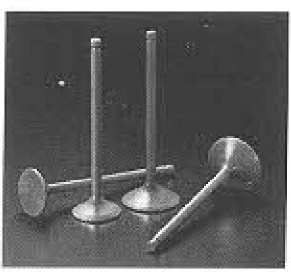
Ceramic turbocharger rotor



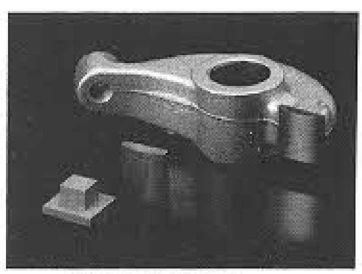
Complete ceramic swirl chamber



Piston pin



Ceramic valve



Rocker arm tip

Fig. 1. Ceramic components for engine applications

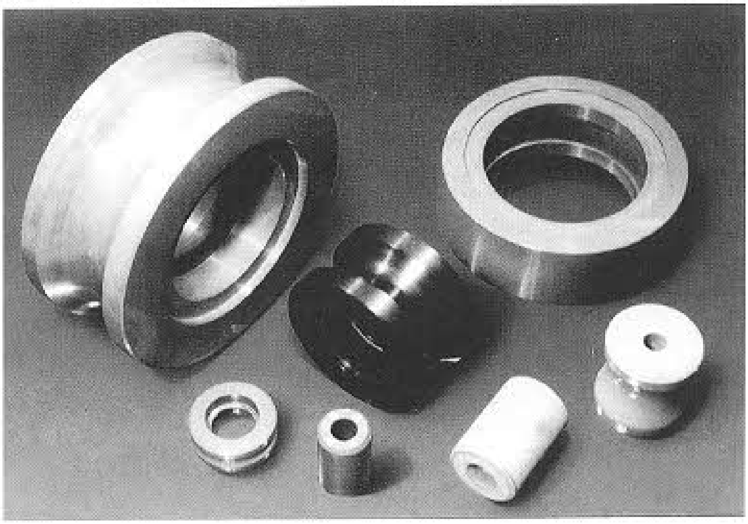


Fig. 2. Ceramic rollers and guides for hot rolling applications

Table 1. Properties of NGK's silicon nitride materials

	SN-55	SN-63	SN-73	SN-84	SN-88
Density	3.2	3.2	3.2	3.2	3.5
Flexural strength (MPa) (4 points, JIS R1601)					
RT	850	1100	1150	860	790
800 C	750	900	950	800	780
1000 C	380	650	600	760	770
1200 C	-	500	410	780	770
1400 C	-	-	-	-	760
Young's modules (GPa)	260	310	290	260	300
Poisson's ratio	0.27	0.27	0.27	0.27	0.26
Fracture toughness ($MPam^{1/2}$, JIS R1607)	7	7	7.5	6	7
Thermal shock resistance (C)	800	-	1000	1000	1200
Typical applications	tappet	pin	valve	CTR	guide

properties between roller and rolled material, good abrasion resistance, and easy handling due to their light weight.

The use of reciprocating engine is limited by the need to use special fuel, such as gasoline and kerosene, but ceramic gas turbine is characterized by its adaptability to a large variety of fuels, its compact size, light weight and the small amount of pollutants generated. NGK has been supplying ceramic components to some national projects[7] and joint works[8] in Japan, the US and Europe.

DEVELOPMENT OF SILICON NITRIDE AND ITS FABRICATION PROCESS

In the development of silicon nitride material, many kinds of raw materials have been evaluated and many additive compositions have been examined using phase diagrams in order to develop silicon nitride[9-14] with excellent high temperature strength and durability.

Through strict control of the microstructure, adjustment of the crystalline phase content, and optimization of size distribution of grain boundary phases in the sintered body, we achieved a 4-point flexural strength of more than 700 MPa at 1400°C (SN-88) - the highest strength at high temperature. Table 1 shows current silicon nitride materials of NGK, and Figure 3 shows the microstructure of SN-88 with crystalline phase in grain boundary.

In developing the fabrication process of ceramic components, many forming methods such as injection molding, slip casting and pressing are used, depending on shape required. The injection molding method was improved to be suitable for making complex three dimensional shapes such as blades for CTR. Figure 4 shows the fabrication process of CTR.

A near-net-shape processing technique was developed which satisfies profile tolerances and mechanical strength requirements at the same time. As a particular achievement the as fired CTR exhibited the required mechanical strength without the necessity of any surface treatment like grinding, and the profile tolerance of the rotor blade surface reached 200 microns[15], which is comparable to that of metal gas turbine components.

In applying ceramic components, a ceramic-metal bonding method is one of the key technologies. A press fit method was developed[3] which takes advantage of the high compressive strength of ceramics and high tensile strength of metals. This simple method makes use of physical bonding of the ceramic shaft to the metal shaft of the CTR without any chemical bonding aids

STRENGTH AND FATIGUE OF SILICON NITRIDE

Effect of Size on Strength

To confirm the effect of size on the Weibull statistical theory, the fracture strengths of various types of silicon nitride specimen were measured[16]. Figure 5 shows the experimental results. The strength in each test is plotted against the effective volume. The Weibull theory using the strength and Weibull modules of 15, determined by the 4-point flexural test of 100 specimens, predicted a solid line. The experimental results agreed with the predicted line, as shown in Fig. 5, over the range of 4 orders in effective volume and in different cases of stress distribution such as bending, tension, torsion and rotating. These results show that the fracture strength of

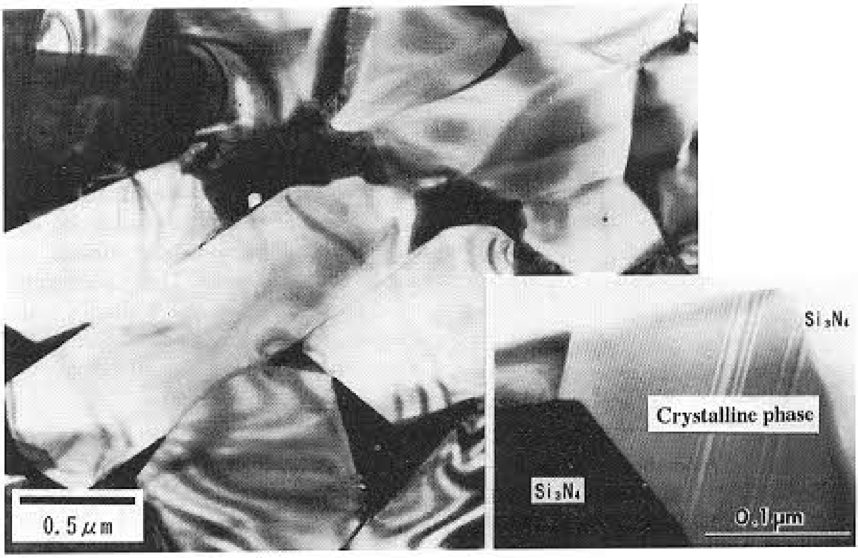


Fig. 3. Microstructure of SN-88

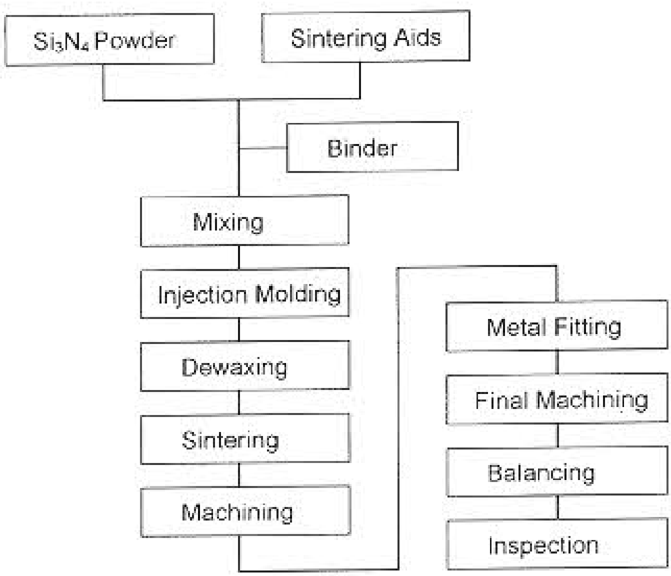


Fig. 4. Fabrication process of ceramic turbocharger rotor

components 4 orders larger in effective volume than the 4-point bending specimens can be predicted from the data of the 4-point flexural strength.

Strength under Multiaxial Stresses

Using a rod specimen 6 mm in diameter, the fracture strength was measured under the multiaxial stresses of tension-torsion and compression-torsion[17]. Figure 6 shows the fractured specimen. The fractured surface of each specimen was perpendicular to the direction of the maximum principal stress, so the maximum principal stress governed the fracture under the multiaxial stresses. There are four principal fracture theories under the multiaxial stresses. Figure 7 shows the experimental results from the tests under the multiaxial stresses, and the predicted curves from the fracture theories under the multiaxial stresses. The experimental data agree with the two curves predicted by the maximum principal stress and Weibull's multiaxial theories[18,19]. These results confirm that the components with complicated shapes should be designed based on the maximum principal stress theory or Weibull's multiaxial theory.

Static Fatigue Behavior

Static fatigue behavior of silicon nitride using tensile specimens has been investigated, because these data determined under tensile stress are very useful for designing the structural components[20-22]. Figure 8 shows the results[21] of the tensile static fatigue tests for high heat resistant silicon nitride (SN-88). SN-88 showed delayed fracture and creep deformation at temperatures over 1200°C. From this figure, the static fatigue strength under tensile stress for 1000 hr at 1200, 1300, and 1400°C are estimated to be 300, 200, and 100 MPa, respectively. The rate of static fatigue strength degradation increased with temperatures. The fatigue behavior of SN-88 shown in Fig. B is divided into two stress regions, higher and lower. In the higher stress region, the power law crack growth formulation to predict the life time can be applied. In this region, slow crack growth from pre-existing flaws caused the strength degradation. However, in the lower stress region, SN-88 showed creep deformation. In the creep deformation region, the Larson-Miller parameter[23], widely used to predict life time of creep rupture in metallic materials, can be applied to predicting the life of SN-88. Figure 9 shows the master Larson-Miller curve of SN-88, rearranged from the data in Figure 8 using a Larson-Miller constant of 30.

Cyclic Fatigue Behavior

Figure 10 shows that the cyclic fatigue behaviors of silicon nitride at room temperature are strongly dependent on the number of cycles, even with a wide range of frequencies from 0.03Hz to 3kHz[24]. These results show that the cyclic fatigue mechanism at room temperature is not only slow crack growth (SCG) fatigue but also another fatigue mechanism. These data also show that the strength degradation is not strongly dependent on time.

Figure 11 shows the experimental data from the cyclic fatigue tests of silicon nitride at room temperature, changing the ratio of minimum stress to maximum stress. The ratio of R=1 indicates a tension-compression perfect alternating cyclic fatigue test. R=0 indicates a perfect pulsating cyclic fatigue test. The closed circles indicate failure up to 10^7 cycles. The open circles indicate survival at 10^7 cycles. The solid line is the modified Goodman line[25,26], which is well known as the fatigue limit in the field of metals. We must use this material in the stress level area under the modified Goodman line. It is noted that the cyclic fatigue concept of the

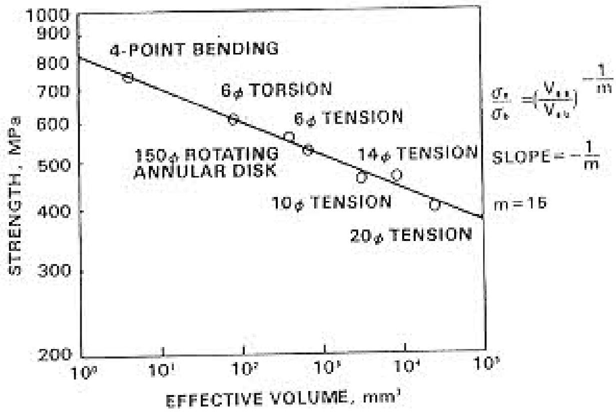


Fig. 5. Effect of size on fracture strength

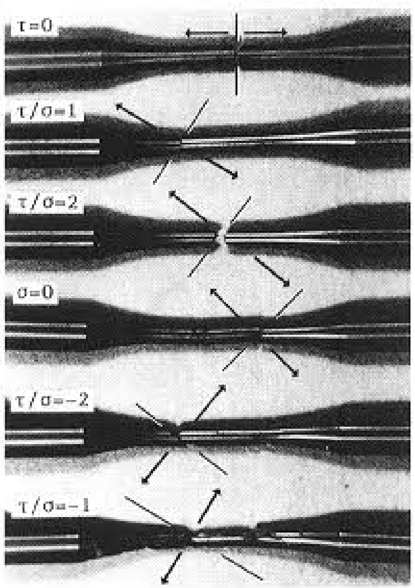


Fig. 6. Typical fractured silicon nitride specimen in tension, combined tension/torsion, torsion and combined compression/torsion. Arrow indicates the direction of maximum principal stress.

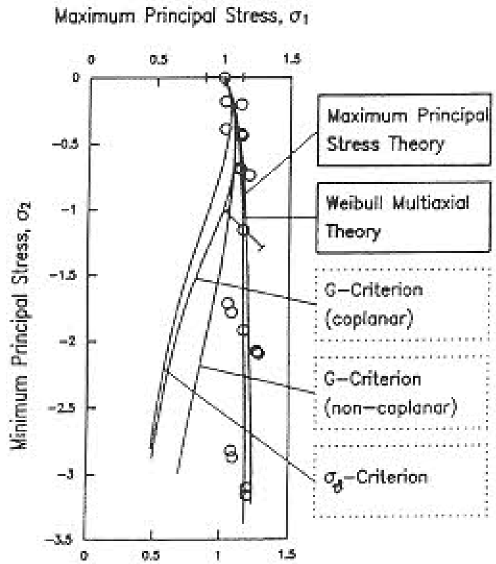


Fig. 7. Experimental (circles) and predicted (line) results under multiaxial stresses

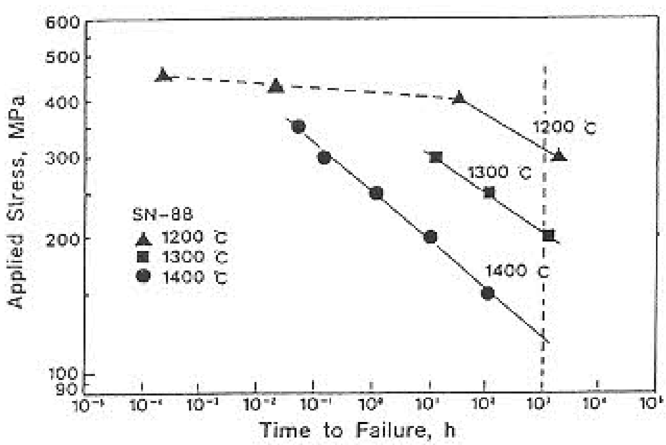


Fig. 8. Results of static fatigue test under tensile stress

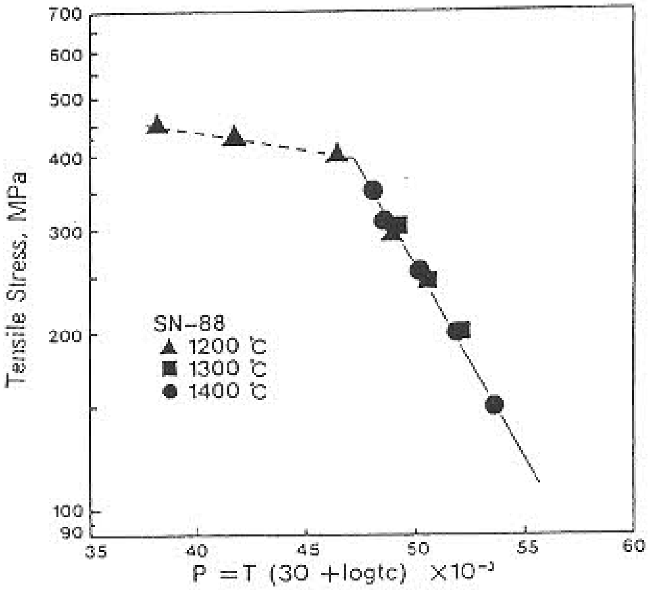


Fig. 9. Master Larson-Miller curve

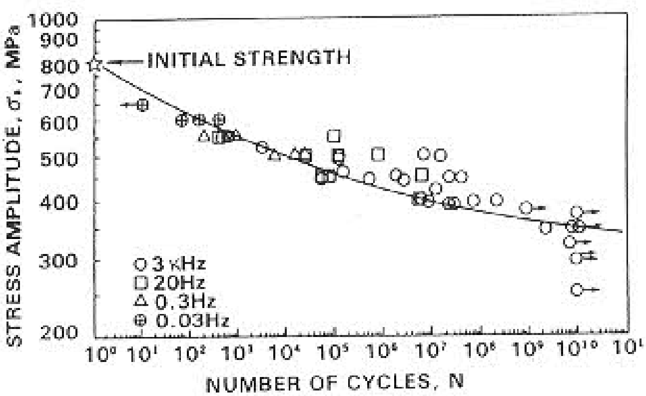


Fig. 10. Alternating cyclic fatigue behavior of a silicon nitride cantilever beam specimen

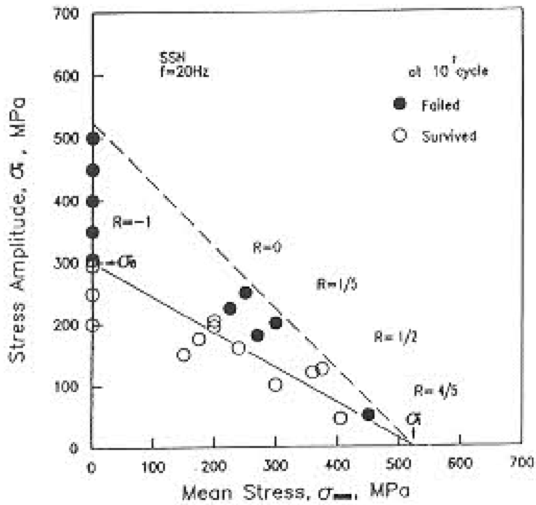


Fig. 11. Stress amplitude vs. mean stress for fatigue failure of a silicon nitride at room temperature

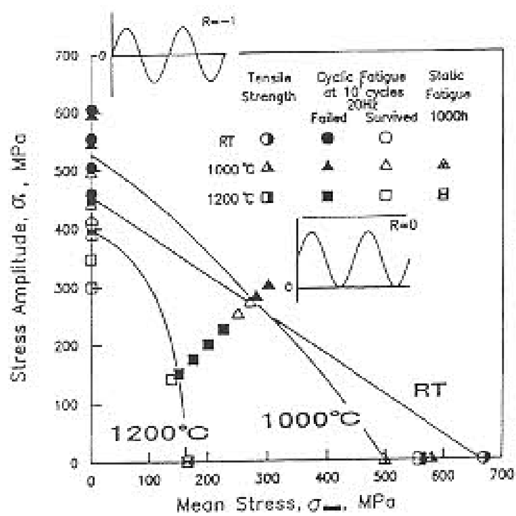


Fig. 12. Stress amplitude vs. mean stress for fatigue failure of a silicon nitride at high temperature

modified Goodman line is applicable in ceramics as well as in metals. The cyclic fatigue data at high temperatures[27,28] shown in Figure 12 imply that perfect alternating cyclic fatigue is more severe than static fatigue at room temperature, but as the temperature increases static fatigue becomes more severe.

DESIGN CONCEPT FOR CERAMIC COMPONENTS

Figure 13 shows the design concept[2,29] for high heat resistant ceramic components. First, the initial design is determined, including factors such as geometric design, material selection, and operating conditions. Then FEM techniques analyze stress and temperature distributions in the component. After that, mechanical properties such as strength, Weibull modules, SCG behavior, creep failure, and oxidation fatigue are evaluated to obtain the allowable stress-temperature critical conditions for each failure mode.

In a graph of stress vs. temperature, the Fracture Map shows the stress-temperature distribution in the components and the allowable stress-temperature condition for each failure mode. Comparing these variables in Fracture Map provides either the required material properties for the component or new information for design. If necessary, the same analysis is performed on new materials and designs.

Fracture Map for Ceramic Turbocharger Rotor

Figure 14 shows a fracture map[29] of a CTR in operation at a gas temperature of 950°C and a tip speed of 500 m/s. The allowable stress-temperature values of two silicon nitrides are estimated for each failure mode. Failure probability is taken into account for fast fracture and static fatigue failure modes. As shown in this fracture map, durability against oxidation fatigue failure and creep failure is comparable between SSN-A and -B; however SSN-A has an advantage over SSN-B in the fast fracture condition and in the durability against static fatigue failure. Therefore SSN-A is suitable for the application in which long-term durability is required. The point A corresponding to the root portion of rotor blades is close to the allowable stress curve, with a failure probability of 10 ppm for the static fatigue failure condition of SSN-A. Therefore, the stress-temperature condition at the root portion of the rotor blades is critical for the static fatigue failure mode of SSN-A, whereas those at the highest temperature and the highest stress point are not critical. As the allowable stress-temperature condition for oxidation fatigue of SSN-A and -B is not critical in contrast to that for other failure modes, oxidation fatigue conditions can be ignored.

TRANSFER TO THE MARKET

To prove the above-mentioned design concept, the CTR was evaluated by the over hot-spin test of dynamometer, the foreign object damage test, high load engine durability test under several driving conditions, and a few failure mode tests such as artificial damage on blade or shaft of the CTR or increased unbalance conditions with over 10,000 hr of total operating time. The durability of the CTR was evaluated by non-destructive tests and proof tests, then finally the CTR was launched to the automotive market successfully.

Today about a million cars equipped with CTR are on the road in Japan. The fact that no accident has resulted from a CTR proves that the CTR has excellent reliability with fracture

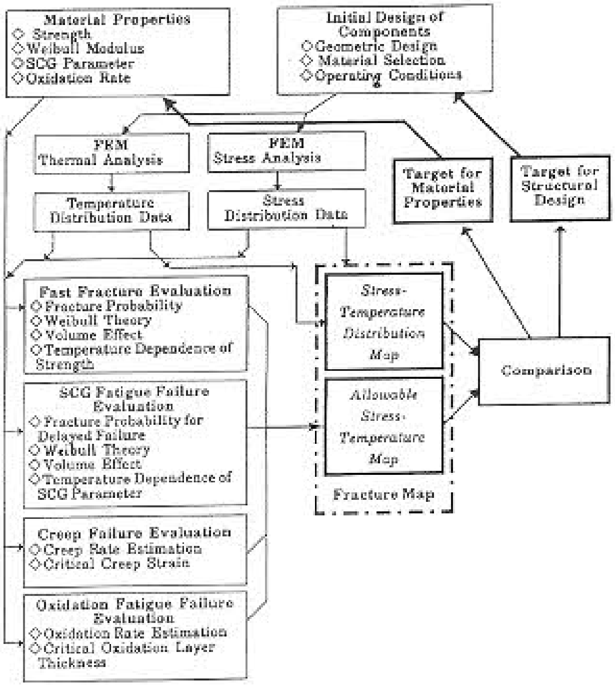


Fig. 13. Design concept for ceramic component

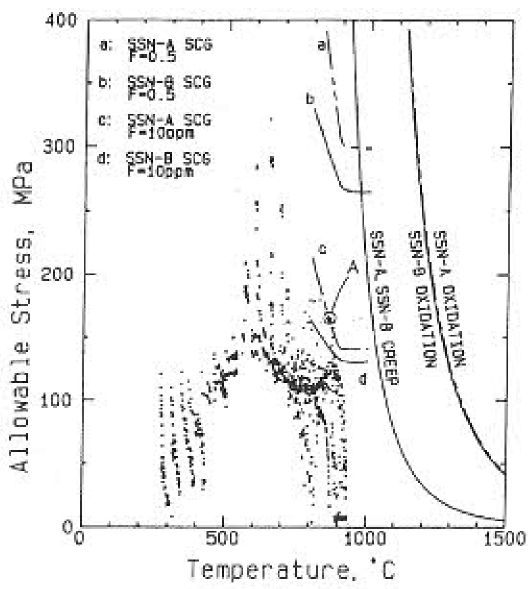


Fig. 14. Fracture map of a ceramic turbocharger rotor
TIT: 950 C, tip speed: 500 m/s, required life time: 5000 hrs

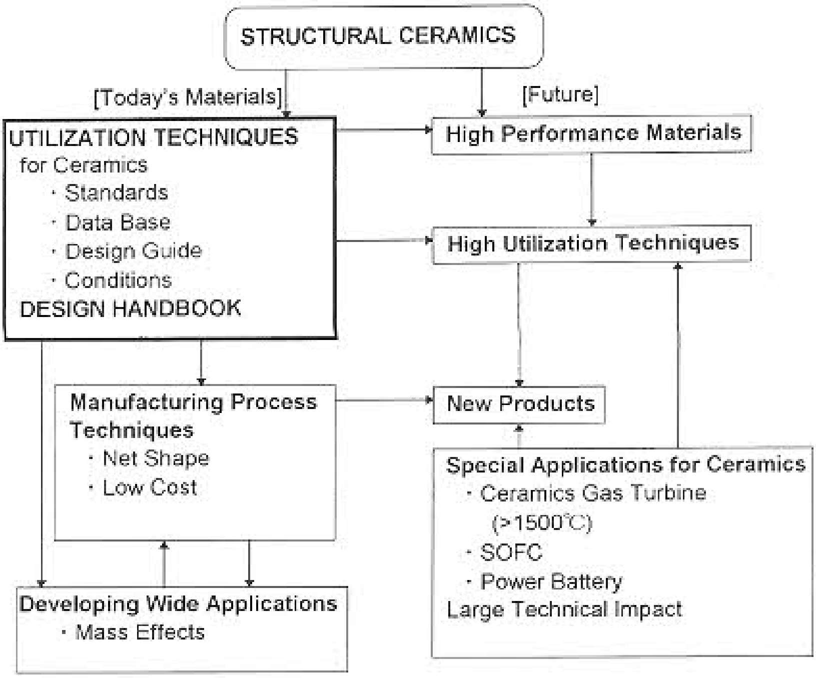


Fig. 15. Utilization techniques for structural ceramics.

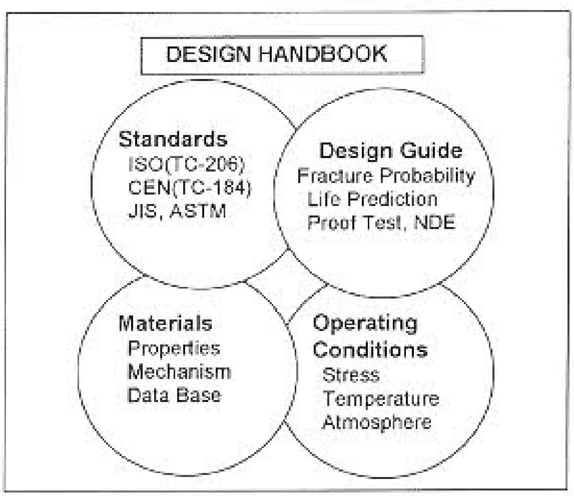


Fig.16. Main items in a design handbook of utilization

probability of less than 10 ppm. It further proves that the above mentioned design methodology is useful for developing high temperature structural ceramic components[30].

NGK has been developing ceramic components for structural applications in cooperation with automotive makers, gas turbine makers, machinery makers and others, on the basis of the materials and technologies which were investigated and innovated by NGK.

FUTURE WORKS

Through the study on the reliability of silicon nitride such as the effect of size on strength, fracture under multiaxial stress conditions, static fatigue, cyclic fatigue and life prediction using fracture map, ceramics has become understood as an excellent material, offering long life if the ceramic component is correctly designed. So, the lack of reliability often said about ceramics is not due to ceramics, but instead to the lack of utilization techniques for structural ceramics.

To promote the commercialization of structural ceramics, utilization techniques such as those shown in Figure15 should be researched and developed to establish a design handbook[31] or structural ceramics. These techniques are as follows:

1. to standardize testing methods of material properties,
2. to measure material properties and clarify the mechanism,
3. to establish a data base and standard materials, and
4. to establish a design guide: design methodology of fracture probability and life prediction, NDE, and proof test.

Except the above items, correct information about operating conditions applied to ceramic structural components is essential as fundamental utilization techniques as shown in Figure15. Establishing of a design handbook would promote not only developing applications using today's materials, but also developing higher performance ceramics and higher utilization techniques.

FINAL REMARKS

Fine ceramics which have the unique characteristics of heat resistance, anticorrosiveness and light weight compared with metals or plastics, are considered to be key materials that will contribute to solving global environmental and energy problems. Their widespread practical use is expected. However, fine ceramics is still rather a new material in the industry. Properties, database, design methodology, proof test technique, non-destructive evaluation, and standardization of testing methods should be completed to establish a design handbook.

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