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SOME ASPECTS OF MATERIALS AND STRUCTURES ENGINEERING WITH CERAMICS FOR ENGINE APPLICATIONS.

9 Final Repts.

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ABSTRACT

This paper identifies the benefits of the use of ceramics in heat engines, how one designs with brittle materials, and some key materials for heat engine application. Current engineering and materials problems in the use of ceramics for engine applications are summarized. Examples of how the materials science and engineering community is addressing two of these problems: time and environmental dependence of properties, and development of improved high temperature (ca 1400°C) materials, are presented. A case history of a successful design of a ceramic engine component is also presented. Finally, progress on a variety of ceramic engine programs is reviewed.

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CONTENTS

	Page
INTRODUCTION	1
WHY CERAMICS FOR HEAT ENGINES?	1
BRITTLE MATERIALS DESIGN	2
SILICON-BASED CERAMICS FOR HEAT ENGINES	3
CURRENT ENGINEERING LIMITATIONS	5
TIME AND ENVIRONMENT DEPENDENCE OF MECHANICAL PROPERTIES	7
THE DEVELOPMENT OF IMPROVED MATERIALS	9
CASE STUDY OF A SUCCESSFUL CERAMIC DESIGN - THE DARPA/DOE/FORD STATOR	12
RECENT PROGRESS ON CERAMICS FOR HEAT ENGINES	18
CONCLUDING REMARKS	19
ACKNOWLEDGMENTS	19
REFERENCES	20

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INTRODUCTION

It is a great privilege to have been invited to present one of the conference plenary papers on the general topic of engineering with brittle materials. I can think of few other fields which can serve as such a vivid example of the three areas which ICM-3 plenary papers should address, namely:

- Serious Engineering Problems
- Design Procedures when Data, Experience, or Codes of Practice are Lacking
- The Contribution of the Materials Science and Engineering Community to Solution of Current Problems.

In fact, I was so intrigued by each of these topics that instead of addressing only one, I thought that I would briefly address all three, based on perspective and experience gained from involvement with several ceramic-based engine programs in progress during the past eight years.

This paper will first identify the benefits to be derived from the use of ceramics in heat engines, then briefly review the brittle materials design process and identify the important candidate ceramics for these applications. A summary of the engineering and materials problems challenging the ceramic engine community will be identified and briefly described. The next portion of the paper will deal with two examples of such problems: the development of a data base on materials properties after long-time (ca 350 to 3500 hours) exposure to simulated service environments and the development of materials with improved high temperature (ca 1100 to 1400°C) strength and creep properties via grain boundary engineering (GBE). Then we will review a case history of a successful design evolution of a ceramic structure (a stator for a small gas turbine) where data was limited and design practices were developed as the program progressed. The final section of the paper will briefly review results from some key ceramic engine demonstration program.

WHY CERAMICS FOR HEAT ENGINES?

Energy, environmental, and materials supply considerations are the impetus for the current emphasis on ceramic materials technology for heat engines. The raw materials for the silicon-based ceramics (silicon nitrides, silicon carbides, and alumino-silicates) relevant to heat engines are abundant and their extraction from the earth is less disruptive of the environment than the extraction of ores for superalloys.¹ These ceramics are potentially much cheaper than superalloys and can run uncooled to temperatures well beyond superalloys. This high temperature capability of ceramics can contribute to increased engine efficiency in two ways: first, by increasing engine operating temperatures (Carnot cycle efficiency), and second, by reducing or eliminating the energy costs associated with the management of a cooling fluid, as is required in today's air-cooled gas turbines or water-cooled piston engines. Examples of the potential gains to be obtained for various generic classes of engines are presented in Table 1 (Ref. 2 to 5). It is important to note

1. Davidge, R. W. "Economic and Energetic Considerations for Nitrogen Ceramics", Nitrogen Ceramics, Noordhoff International Publishing, Leyden, 1977, p. 653-657.
2. Klann, J. L. "Advanced Gas Turbine Performance Analysis", 8th Summary Report, Automotive Power Systems Contractors Coordination Meeting, ERDA-64, May 1975, p. 173.
3. Helms, H. E., and Rockwood, F. A. "Heavy Duty Gas Turbine Engine Program Progress Report - 1 July 1976 to 15 January 1978", Report DDA EDR 9346, Prepared for NASA-Lewis Research Center (Contract NAS 3-20064) February 1978.
4. Wallace, F. B., Stone, A. J., and Nelson, R. B. "Ceramic Component Design ARPA/Navy Ceramic Engine Program" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 593-624.
5. Kamo, R. "Cycle and Performance Studies for Advanced Diesel Engines" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 907-922.

that worthwhile gains can be made for several classes of engine by use of ceramics at temperatures between 1100°C (2000°F) and 1260°C (2300°F). From a materials point of view this is a very tractable temperature range with many possible materials and processing options. On the other hand, the small automotive and very large industrial gas turbines will require materials with 3000- to 30,000-hour life at temperatures ranging between 1370°C (2500°F) and 1650°C (3000°F). At present, materials and associated processing options are extremely limited or unavailable for such life at these high temperatures and thus present a formidable but far from impossible challenge for these two applications. The important point is that the potential payoff from the use of ceramics is so great that active materials development, design, and engine feasibility demonstration programs are underway for each class of engine listed in the table and, as will be shown in the final portion of this paper, very significant progress has been made.

TABLE I. Ceramic Technology Payoff for Some Typical Heat Engines

Engine Type	H.P. Range	Configuration	~ Δ SFC ^a	~ Δ Power	Ref
AUTOMOTIVE GT	~ 200	REGENERATED, SINGLE-SHAFT, 2500°F, TIT	-27%	0	[2]
TRUCK GT	~ 350	REGENERATED, 2-SHAFT, 2265°F, TIT	-17%	+30%	[3]
LIGHT SHIP GT	~ 1000	SIMPLE-CYCLE, 3-STAGE, 2500°F, TIT	-10%	+40%	[4]
DIESEL	~ 500	ADIABATIC - TURBO COMPOUND T _{max} ≈ 2000°F	-22%	+37%	[5]

SFC - Specific fuel consumption - lb/hp-hr (gm/W-hr), based on current state-of-the-art.

BRITTLE MATERIALS DESIGN

Brittle materials can be very strong; however, they generally manifest a wide scatter in strength and since they do not yield (at least up to quite high temperatures), they do not have the capability to redistribute high local stress concentrations as do most engineering metals. These two manifestations of brittleness have, historically and justifiably, made designers reluctant to use brittle materials as structural components under tensile loading. The successful design of structural components with ceramics requires that the designer know the state of stress at every point within the component. It has only been within the last 10 to 15 years that sufficiently advanced two- and three-dimensional heat transfer and stress analysis computer programs have been available to attempt finite element analysis on the scale required for highly stressed brittle materials. Due to the scatter in strength, design with ceramics is done on a probabilistic as opposed to a deterministic basis, generally using the Weibull analysis.^{6,7} Modern stress analysis programs for ceramics now incorporate Weibull statistics in the finite element program so that a risk of failure at each element in the component may be predicted.⁸ A design is then refined and re-refined until a satisfactory probability of survival is attained. Of course, analysis is only as good as the input data on materials properties, loads, boundary conditions, gas flows, etc. Thus, all one can really expect of the stress analysis is to get the design onto the "playing field." It still takes a great deal of engineering judgment for the design team to "score" by executing a successful design. The strategy by which the brittle materials design team will eventually win is the total systems, iterative, interdisciplinary, trade-off approach. This approach is shown for the general case in Fig. 1 and 2, and will be demonstrated for an actual case history to be presented later in this paper. The design team executes a preliminary design following the logic in Fig. 1. The output of this is a proposed

6. Dukes, W. H. "Handbook of Brittle Material Design Technology", AGARDograph No. 152, North Atlantic Treaty Organization, February 1971, p. 5.
7. Tree, D. J., and Kington, H. L. "Ceramic Component Design Objectives, Goals, and Methods", Ceramic Gas Turbine Demonstration Engine - Program Review, MCIC Report #78-36, 1978, p. 41-76.
8. Nicholls, P. E., and Paluszny, A. "Techniques for Design of Small Ceramic Turbine Rotors" in Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1974, p. 63-78.

optimum design/material/processing combination for a given component in the specific system under consideration. Once the design/material/process for the given component has been selected, validation by rig and/or engine tests usually requires several passes through the iterative design scheme shown in Fig. 2. It is important to note that there is unlikely to be one optimum material/process combination that will satisfy a wide variety of systems requirements. Thus, the successful development of ceramic engine technology will require the existence of many materials and materials processing options. This leads to the next area: "What are the materials and processing options available today?"

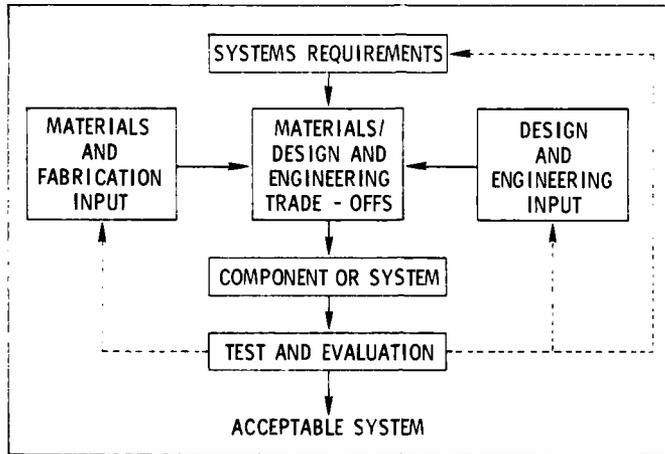


Fig. 1. "Brittle Materials Design" systems trade-off logic.

SILICON-BASED CERAMICS FOR HEAT ENGINES

As noted above, brittle materials can not relieve stress concentrations by yielding. They are consequently vulnerable to stress concentrations and the designer must avoid them, whether they are the result of mechanical or thermal loading. In practice, thermal loading is much more difficult to deal with⁶ (probably due to the difficulty

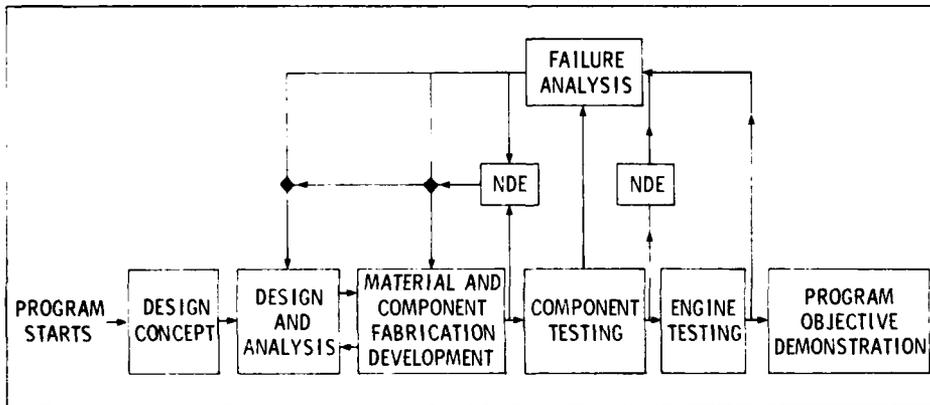


Fig. 2. Iterative "Brittle Materials Design" scheme.

in specifying thermal loads from point to point in a combustion gas stream). Thus, a key property for an engine ceramic is thermal shock resistance. Whatever other virtues a material may have, if it is susceptible to thermal shock damage in the expected thermal environment, the material is unacceptable. Thermal shock resistance is a complex quantity to measure since it is as much a function of heat transfer environment and geometry of the part as of the material. This is shown in the standard equation for the maximum instantaneous temperature change in environment that a brittle material can sustain without fracture:

$$\Delta T = \frac{\sigma_f (1-\nu) k}{\alpha E bh} S$$

where: σ_f = fracture stress
 ν = Poisson's ratio
 α = coefficient of thermal expansion
 E = elastic modulus
 k = thermal conductivity
 b = plate half-thickness
 h = heat transfer coefficient
 S = geometric factor.

As one works with ceramics from the thermal shock resistance standpoint, it readily becomes apparent that of all the materials properties the most critical is α . One wants α to be zero or as small as possible. Thus, the three families of engineering ceramics of current interest in the heat engine area are all low thermal expansion silicon-based materials: aluminosilicates, silicon nitrides, and silicon carbides. Aluminum titanate is also a candidate for certain applications⁹ and this material also has a very low α .

Table 2 lists some members of these three families of engineering ceramics with their relevant properties. The aluminosilicates are candidates for heat exchangers on gas turbines, while the other materials are candidates for various stationary and dynamic hot flow path components such as vanes, blades, shrouds, pistons, etc. The important thing to note is that those ceramics which have the highest room temperature strengths, particularly the dense silicon nitrides, do not retain the high strengths to the 1350°C range. A similar reduction of creep resistance occurs in this temperature range for this class of materials. The grain boundary phases required to obtain

TABLE 2. Families of Heat Engine Ceramics and Typical Bulk Properties

		MOR (RT, psi)	MOR (1000°C, psi)	MOR (1375°C, psi)	RT - E ($\text{psix}10^{-6}$)	α (10^{-6} deg C ⁻¹)	K (Wm^{-1} , deg C ⁻¹)
ALUMINO SILICATES	LITHIUM ALUMINO SILICATE (LAS)	12,000	10,000	-	-	450 ppm	-
	MAGNESIUM ALUMINO SILICATE (MAS)	20,000	20,000	-	23	2.2	3.5
	ALUMINO SILICATE [†] (AS)	-	-	-	-	-	-
SILICON NITRIDE	HOT-PRESSED (MgO ADDITIVE)	100,000	90,000	48,000	46	3.0	30-15
	SINTERED (Y ₂ O ₃ ADDITIVE)	95,000	85,000	40,000	40	3.2	28-12
	REACTION-BONDED (2.45 g/cc)	30,000	50,000	55,000	24	2.8	6-3
SILICON CARBIDES	HOT-PRESSED (Al ₂ O ₃ ADDITIVE)	95,000	85,000	75,000	65	4.5	85-35
	SINTERED (a PHASE)	45,000	45,000	45,000	59	4.8	100-50
	REACTION-SINTERED (20 v/o Free Si)	55,000	60,000	40,000	50	4.4	100-50
	CVD (Lower Values)	60,000	80,000	80,000	60	-	-

Total thermal excursion in ppm from 0 to 1000°C.
[†] Properties available only in matrix form.

the full density and high strength at lower temperatures cause the reduction in strength and creep resistance at high temperatures. This subject will be dealt with in a subsequent section.

9. Rottenkolber, P. and Walzer, P. "Ceramics for Vehicular Power Plants", Presented at the 4th CIMTEC Meeting, St. Vincent, Italy, May 1979.

At this point in the paper it has been established that the benefits to be gained from exploiting ceramics for heat engine technology are significant, a design methodology is emerging, and a reasonable number of materials and processing options exist. These are the positive aspects. We will now consider some of the negative aspects, namely, those problems which are impeding the implementation of ceramic technology for engines.

CURRENT ENGINEERING LIMITATIONS

While the feasibility of ceramic utilization in heat engines is being demonstrated in a variety of programs,²⁻⁵ many engineering problems remain to be solved before feasibility becomes industrial practice. Numerous systems and materials specific problems identified over the past few years can be reduced to five broad areas, namely:

- Lack of Comprehensive Data/Experience Bases
- Lack of Service-Verified Life Prediction Methodology
- Assurance of Reliable/Reproducible Components
- Attachment of Ceramics to Metals or Ceramics to Ceramics
- Maintenance of Adequate Mechanical Properties at High Temperatures and Real Environments.

Large amounts of data have been generated on advanced ceramics in recent years¹⁰⁻¹³ yet large gaps still exist. Further, since design with brittle materials is a statistical process, one requires a high degree of statistical confidence in the data. For example, to obtain a 90% confidence level that a Weibull modulus m is within $\pm 10\%$ of the reported value requires the testing of more than 120 samples.¹⁴ Comprehensive data bases on any mechanical property with such large sample populations simply do not exist for turbine ceramics. One reason for this lack is that the resources required to generate such extensive data bases have been, in general, unavailable. Furthermore, the improvements in mechanical properties for most of the silicon-based ceramics over the past five or six years have been so marked that a large investment in property generation on a given grade material would have been inappropriate. Nevertheless, such a comprehensive data base on materials in the as-fabricated condition is now required. Several materials have reached a sufficient level of development where a fixed materials specification can be considered and generation of a comprehensive data base warranted.

The experience base in design with ceramics for engine components is miniscule when compared to the experience base with superalloys. Even though the engine design community now consider ceramics to be serious candidate materials for structural use, many years will be required for the collective experience of individual design teams to coalesce into codes of practice or routine design guidelines.

10. Ceramics for Turbines and Other High Temperature Engineering Applications, D. J. Godfrey, ed., Proceedings of the British Ceramic Society, v. 22, 1973.
11. Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1974.
12. Ceramics for High Performance Applications-II, J. J. Burke, E. M. Leno, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978.
13. Keramische Komponenten für Fahrzeug-Gasturbinen, W. Bunk and M. Böhmer, ed., Springer-Verlag, Berlin, 1978.
14. Jeryan, R. A. "Use of Statistics in Ceramic Design and Evaluation" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Leno, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 35-51.

A very important area for any future code of design practice will be credible life prediction methodology for components. Much ongoing work is focused on life prediction methodology.¹⁵⁻¹⁸ At present, all analytical life prediction methodologies are based on slow crack growth models combined with fracture mechanics approaches. It is by no means certain that slow crack growth is the only mechanism for time-dependent failure. Further, even for those materials where slow crack growth has been demonstrated to be the prime mode of time-dependent failure, the fracture-mechanics-based life prediction methodology has not been validated on engine components. One has reason for optimism here, since slow crack growth and fracture-mechanics-based life prediction have been validated on grinding wheels and electronic substrates.¹⁸ However, validation for engine components in realistic environments is still required.

Quality assurance (including NDE) of ceramic engine components in serial or mass production is an issue. Typically, when ceramic components for application in severe environments (i.e., tool bits, spark plug insulators, ignitors, grinding wheels, catalyst substrates, etc.) go into mass production, quality assurance and component reproducibility improve dramatically. However, for fabrication of ceramic engine components we are still at the small demonstration scale, which is typically beset by quality assurance, process, materials, and component reproducibility problems. This is an exceedingly important area. Designers need consistency more than the promise of a few more MPa in strength.

In nearly every ceramic engine demonstration program to date, failures of ceramic components (when not caused by an outright failure of the test rig) have resulted from an attachment scheme not working properly. This has been the case for stators ranging in size and design philosophy from the Westinghouse/DARPA 20-MW program,¹⁹ through the DARPA/Garrett program,²⁰ down to the 10-kW Solar/MERADCOM program.²¹

15. Paluszny, A., and Nicholls, P. F. "Predicting Time-Dependent Reliability of Ceramic Rotors" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 95-112.
16. Wiederhorn, S. "Reliability, Life Prediction, and Proof Testing of Ceramics" in Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1974, p. 633-666.
17. Ritter, J., and Wiederhorn, S. "Applications of Fracture Mechanics to Assure Reliability of Ceramic Components", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
18. Govila, R. K. "Materials Parameters for Life Prediction", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
19. Bratton, R. J., and Miller, D. G. "Brittle Materials Design, High Temperature Gas Turbine - Stationary Turbine Project" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 689-730.
20. Harper, J. "Overview of DARPA/NAVAIR Ceramic Engine Demonstration Program", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
21. Metcalfe, A. G., and Napier, J. C. "Application of Ceramics to MERADCOM 10-kW Gas Turbine Engine" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 827-838.

It has also been the case of ceramic rotors on the DARPA/DOE/Ford program.²² It is clear that the development of compliant attachments that maintain their compliance with time under the full rigors of the engine operating environment is a number one priority issue.

The maintenance of mechanical properties at high temperature and long times is especially crucial for the application of ceramics in engines with operating temperatures above 1150°C (2300°F). Although several candidate engine ceramics can exhibit intermediate temperature oxidation or phase instability problems, treatments to correct these problems have been identified.^{23,24} At the higher temperatures, problems of fall-off in strength or acceleration of creep due to the nature of grain boundary phases, or problems of slow crack growth and/or oxidative degradation pose a considerable challenge to the materials scientist. The next sections of this paper will illustrate some approaches being pursued to identify the effects of environment on high temperature properties and efforts to design materials with higher strength creep properties for the 1300 to 1400°C range.

TIME AND ENVIRONMENT DEPENDENCE OF MECHANICAL PROPERTIES

One of the major problems confronting designers wishing to utilize ceramics in engine applications is the lack of a comprehensive body of data on time and environmental dependence of mechanical properties, particularly strength and creep data. For proper design, the designer needs the materials properties existent at the end of the projected service life time, not "virgin" materials properties. This is an area where the materials science community can be of great help. Information concerning both time-dependent strength and creep can be obtained from stress rupture tests. For ceramic materials both stress rupture and creep are usually obtained from modulus of rupture (MOR) bars, as tension testing of brittle materials is difficult to conduct. Typically, total creep strain is determined by measuring the curvature in a flexural specimen which has survived a given time in a stress rupture test. Stress rupture curves, such as the one for hot-pressed silicon nitride (Fig. 3), can be used in the classical way to predict a safe stress level at a given temperature for a given life. Alternatively, if the time dependence can be shown to be due to slow crack growth, as is the case with HPSN, then the data can be analyzed to provide input into a fracture mechanics life prediction model as discussed by Jakus and

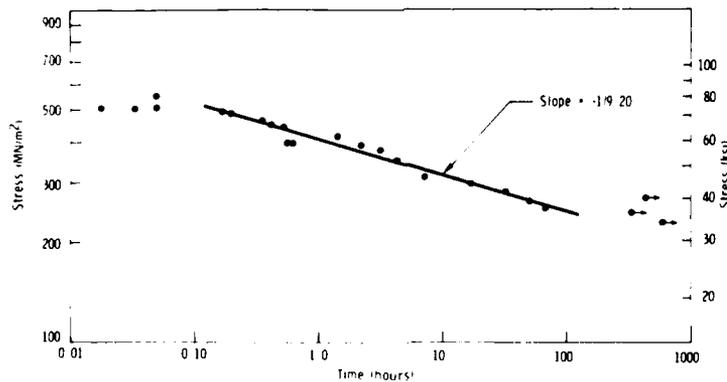


Fig. 3. Flexural stress rupture of NC-132 hot-pressed silicon nitride, 1200°C.

22. McLean, A. F., and Secord, J. R. "Brittle Materials Design, High Temperature Gas Turbine", Ford Motor Co., Interim Report, Army Materials and Mechanics Research Center, AMMRC TR 79-12, May 1979.
23. Gazza, G. E., Knoch, H., and Quinn, G. D. "Hot-Pressed Si_3N_4 with Improved Thermal Stability", Amer. Cer. Soc. Bull., v. 57, no. 11, 1978, p. 1059-1060.
24. Fisher, E. A., and Trela, W. A. "Evaluation of Ceramic Turbine Stators", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).

Ritter.²⁵ However, it is important to independently ascertain by the use of fractography, that the time dependence of strength is due to slow crack growth. Examples of time-dependent behavior not attributable to slow crack growth have in fact been observed.²⁶ In such cases the current life prediction models are not valid. It also should be noted that, given the present experience base, there is no justification for extrapolating data beyond the longest actual test times. It is also important that tests be conducted in oxidizing or combustion gas atmospheres rather than inert environments. The last two years have seen major efforts in acquiring such stress rupture data, particularly by Quinn at AMMRC,²⁷ and Govila at Ford,²⁸ as well as in many other laboratories around the world. However, much more work is required; particularly better statistical approaches, measurements in combustion environments with relevant trace impurities, and tests extending to 10,000 or more hours. Many of the candidate engine ceramics exhibit a reduction in room temperature strength after exposure to oxidation in the 1000 to 1400°C range for several hundred hours. This was first identified as a problem with regard to static oxidation in the Westinghouse/DARPA program.²⁹ If static oxidation presents a problem, what will occur under cycle oxidation? Work is currently underway at AMMRC,³⁰ AiResearch,³¹ Volkswagen³² and elsewhere to study the effects on retained strength after cyclic exposure of engine ceramics at high temperatures, in oxidizing environments, and with large members of thermal cycles. Benn and Carruthers at AiResearch³¹ utilize a combustor rig with an oxidizing combustion gas at temperatures to 1375°C (2500°F) combined with cyclic air quenching. Quinn at AMMRC³⁰ has used a stepped temperature oxidation cycle in a furnace, in air, coupled with multiple thermal shocks accumulated in a MAPP gas/O₂ flame as illustrated in Fig. 4. Siebels at VW³² utilizes stepped temperature cycling in a furnace, in air, between RT and 900°C to 1260°C. Table 3 shows the range of materials response to such thermal cycling studies. Of particular interest is the comparison of the RBSN data. In spite of different manufacturing procedures, differing densities, and differing test procedures, the percent strength degradation for similar times falls in the 15 to 20% range. Recent data of Siebels³² indicates that newer grades of RBSN tested at VW now also fall into this range (RBSN previously tested at VW showed much greater degradation). It appears that a mutually consistent data base may be emerging on cyclic oxidation of RBSN. At present, multiple data bases do not exist for the response of other engine ceramics to cyclic oxidation. Also, specimen data on the effect of cyclic oxidation under load is nonexistent.

25. Jakus, K., and Ritter, J. E. "Static Fatigue of Si₃N₄", J. Amer. Cer. Soc., v. 61, 1978, p. 274-275.
26. Quinn, G. D., and Katz, R. N. "Time-Dependent High Temperature Strength of Sintered α -SiC", Submitted to J. Amer. Cer. Soc. for publication.
27. Quinn, G. D. "Durability Testing of Heat Engine Ceramics", AMMRC TR to be published.
28. Govila, R. K. "Methodology for Ceramic Life Prediction and Related Proof Testing", Ford Motor Co., Interim Report, Army Materials and Mechanics Research Center, AMMRC TR 78-29, July 1978.
29. Miller, D. G., and co-workers. "Brittle Materials Design, High Temperature Gas Turbine - Materials Technology", Westinghouse Corp., Final Report, Army Materials and Mechanics Research Center, AMMRC CTR 76-32, v. 4, December 1976.
30. Katz, R. N., Lenoe, E. M., and Quinn, G. D. "Durability Testing of Structural Ceramics", Proceedings of the Thirteenth Highway Vehicle Systems Contractors Coordination Meeting, DOE CONF-771037, March 1978, p. 208-223.
31. Benn, K. W., and Carruthers, W. D. "3500 Hour Durability Testing of Commercial Ceramic Materials", 5th Interim Report on Contract DEN 3-27, June 15, 1979.
32. Siebels, J. E. "Oxidation and Strength of Silicon Nitride and Silicon Carbide", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).

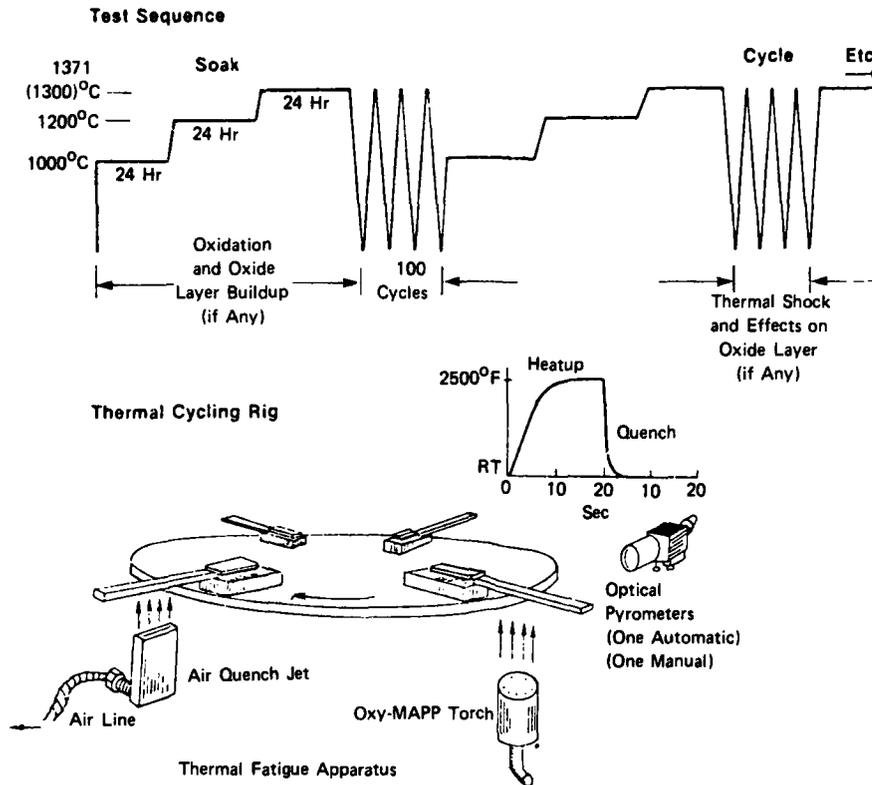


Fig. 4. Combined thermal exposure thermal cycling.

TABLE 3. Combined Thermal Exposure - Thermal Cycling Tests of Engine Ceramics to 2500°F

Material	Virgin MOR (ksi)	Exposed MOR (ksi)	% Change in MOR	Laboratory and Test Condition
NC-132 HPSN	104	50.5	-51	AMMRC - 360 hour/500 cycles in air and flame [30]
NC-203 HP SiC	99	102	+3	
NC-350 RBSN	43	35	-19	
KBI-RBSN	30	24	-20	
Silcomp - Si/SiC	47	32	-32	
Ford - RBSN	42	36	-15	AIResearch - 350 hour/1700 cycles in combustor gas [31]
ACC RBSN-101	37.4	29.4	-21	
Sintered α -SiC	45.8	45.9	0	

The entire field of time and environmental dependence of ceramics, and how such dependence should be factored into life prediction models, should be a very fruitful area of ceramic research for the next several years.

THE DEVELOPMENT OF IMPROVED MATERIALS

As illustrated in Table 2, the state-of-the-art hot-pressed and sintered silicon nitrides (HPSN's and SSN's) do not retain high strength at ~ 1200 to 1400°C . In addressing this problem the contributions of the materials scientists have resulted in the attainment, in the laboratory, of HPSN's and SSN's which do maintain appreciable strength out to 1200 to 1400°C and have significantly improved strengths at room and moderate temperatures. The method by which the improved materials were achieved is

referred to as grain boundary engineering (GBE).^{33,34} This differs from standard microstructure development in that one focuses on controlling the structure of, and reactions occurring at, the grain boundary. To understand why GBE has played a central role in the development of improved fully dense silicon nitride, we must first briefly consider how these materials are processed.

To produce fully dense high strength Si_3N_4 requires that one start with an $\alpha\text{-Si}_3\text{N}_4$ powder plus an oxide additive (usually MgO in state-of-the-art, commercial, hot-pressed material), and hot press at about 1650 to 1750°C. During hot pressing the MgO additive, the SiO_2 on the surface of the $\alpha\text{-Si}_3\text{N}_4$ powder and the Si_3N_4 itself react to form a liquid phase in which the $\alpha\text{-Si}_3\text{N}_4$ dissolves and reprecipitates as high-aspect ratio (about 8:1) $\beta\text{-Si}_3\text{N}_4$ grains (average grain size ~ 1 to 2 μm). The high strength of hot-pressed and some dense sintered Si_3N_4 's results from the elongated microstructure of the $\beta\text{-Si}_3\text{N}_4$. However, the liquid that acted as a solution-precipitation medium at the processing temperature is a grain boundary glass at room and low temperatures. As temperatures increase to near the softening point of these Mg:SiO₂ glasses (ca 1100°C), strength and creep behavior both deteriorate.^{33,34} Figure 5 is a lattice-imaging TEM from the work of Clarke and Thomas,³⁵ which shows the amorphous grain boundary phase at a triple point and along one grain boundary. Note that the grain boundary phase is on the order of 10 to 50 Å in width. Quantitative measurements on such small regions are not currently possible, therefore all GBE developments which have lead to improved fully dense Si_3N_4 were based upon inferential evidence as to phases and reactions in the grain boundary phase. Initial work on improving the high temperature properties of Si_3N_4 were carried out on hot-pressed materials. Figure 6 shows the GBE strategies utilized by various researchers between 1972 and the present.

The reduction of impurities which cause the magnesia-containing grain boundary glass to soften at temperatures lower than would otherwise be the case was investigated by

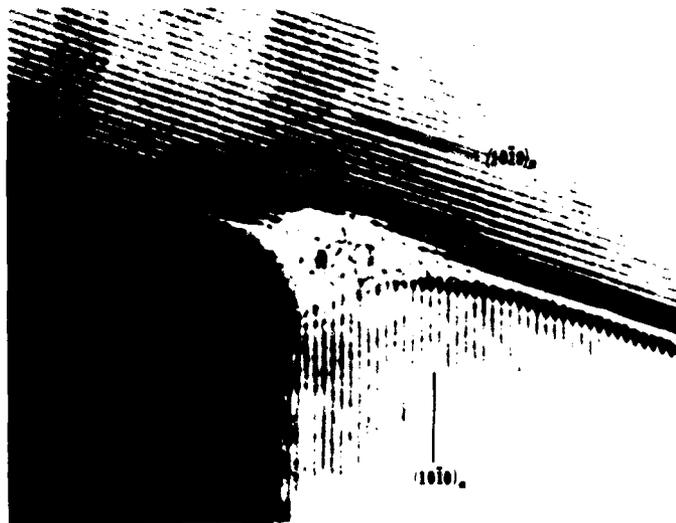


Fig. 5. Grain boundary phase (A) in HPSN with MgO addition.
(after Clarke and Thomas)

33. Katz, R. N., and Gazza, G. E. "Grain Boundary Engineering and Control in Nitrogen Ceramics", *Nitrogen Ceramics*, Noordhoof, Leyden, 1977, p. 417-431.
34. Katz, R. N., and Gazza, G. E. "Grain Boundary Engineering in Non-Oxide Ceramics" in *Processing of Crystalline Ceramics*, H. Palmour, R. F. Davis, and T. M. Hare, ed., Plenum Publishing Co., New York, 1978, p. 547-560.
35. Clarke, D. R., and Thomas, G. "Grain Boundary Phases in a Hot-Pressed MgO Fluxed Si_3N_4 ", *J. Amer. Cer. Soc.*, v. 60, 1977, p. 491-495.

1. Reduce Ca, Na, etc., impurities to make the grain boundary MgSiO₃ glass more refractory.
2. Develop a densification aid to yield a more refractory glass than MgSiO₃.
3. Develop a nonglass grain boundary.
4. Eliminate the grain boundary phase by promoting sintering via volume diffusion.

Fig. 6. Strategies to increase the high-temperature behavior of hot-pressed Si₃N₄.

Lange,³⁶ Kossowsky,³⁷ and Richerson.³⁸ This approach was successful in improving the situation, but two problems remained. First, the levels of calcium, sodium, and other alkali and alkaline earth elements (which were shown to be the main problem) would have to be controlled very tightly in production, hence, cost would increase. Secondly, even pure MgSiO₃ is strength limiting at about 2400°F.

The next development was undertaken by Gazza at AMMRC,³⁹ who reasoned that Y₂O₃·SiO₂ glasses and compounds would be more refractory than MgO·SiO₂ glasses and compounds. Further, it was reasoned that due to a larger ionic radius of the yttrium cation as opposed to magnesium, the yttrium would be prone to remain in the grain boundary and not diffuse away from it as has been observed with magnesium, thus contributing to high temperature stability. This assumption was later confirmed with lattice-imaging TEM by Clarke and Thomas.⁴⁰ It was also thought that it might be possible to devitrify the Y₂O₃·SiO₂ glass phases and form crystalline yttrium silicates in the grain boundary. As it has been subsequently shown by Prof. Jack and his students,⁴¹ yttrium silicates in the grain boundary may form yttrium silicon oxynitrides which can accommodate relatively large amounts of calcium and other impurity cations which are present in commercial purity Si₃N₄ powder. Tsuge and his co-workers have demonstrated the benefits to be gained from the grain boundary crystallization approach using Y₂O₃ + Al₂O₃ additives.⁴² As shown in Fig. 7,⁴³ the predicted improvement using Y₂O₃ has been borne out. Similar improvements in creep behavior at 1260°C have been noted. Much work remains to be done on the Y₂O₃ additive materials and if care is not taken

36. Lange, F. F., and Iskoe, J. L. "High Temperature Strength Behavior of Hot-Pressed Si₃N₄ and SiC" in Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1974 p. 223-235.
37. Kossowsky, R. "The Microstructure of Hot-Pressed Si₃N₄", *J. Materials Science*, v. 8, 1973, p. 1603-1615.
38. Richerson, D. W. "Effect of Impurities on the High Temperature Properties of Hot-Pressed Si₃N₄", *Amer. Cer. Soc. Bull.*, v. 52, 1973, p. 560-562.
39. Gazza, G. E. "Hot-Pressed Si₃N₄", *J. Amer. Cer. Soc.*, v. 56, 1973, p. 662.
40. Clarke, D. R., and Thomas, G. "Microstructure of Y₂O₃ Fluxed Hot-Pressed Si₃N₄", *J. Amer. Cer. Soc.*, v. 61, 1978, p. 114-118.
41. Rae, A. W. J. W., Thompson, D. P., Pipkin, M. J., and Jack, K. H. "The Structure of Yttrium Silicon Oxynitride and its Role in the Hot-Pressing of Si₃N₄ with Yttria" in Special Ceramics, P. Popper, ed., v. 6, BCRA, 1975, p. 347-360.
42. Tsuge, A., and Nishida, K. "High Strength Hot-Pressed Si₃N₄ with Concurrent Y₂O₃ and Al₂O₃ Additions", *Amer. Cer. Soc. Bull.*, v. 57, 1978, p. 424-426.
43. Richerson, D. W., and co-workers. "ARPA/Navy Ceramic Engine Materials and Process Development Summary" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 625-650.

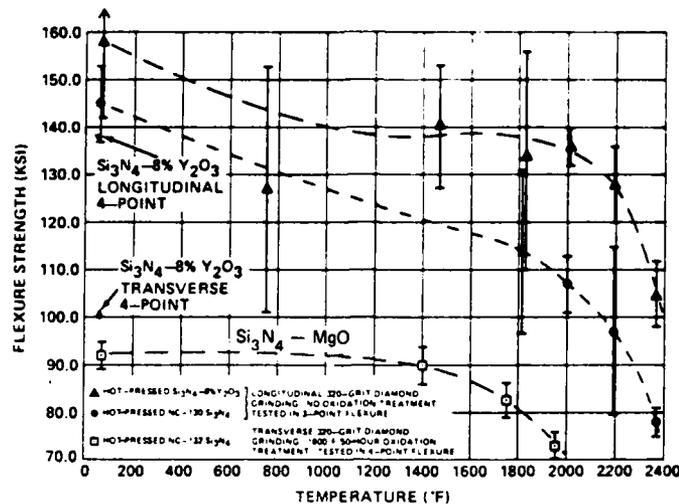


Fig. 7. Flexure strength of hot-pressed silicon nitride versus temperature (from Ref. 43).

to be in the proper area of the Y_2O_3 - SiO_2 - Si_3N_4 phase diagram, a phase instability can occur at $1000^\circ C$.⁴⁴ Post fabrication heat treatment has been shown to significantly reduce if not eliminate this problem.²³

Thus far, additions used for enhancing the sinterability of Si_3N_4 have resulted in the formation of separate boundary phases which have controlled properties. A further approach to sintering, successfully used by Prochazka⁴⁵ with SiC , would be to use additions which promote greater degrees of volume diffusion, remove inhibiting species from the system by volatilization, form solid solutions, and produce "clean" grain boundaries. While various teams are pursuing research on Si_3N_4 using this concept, no one has yet achieved the goal. The achievement, however, may result in differences in microstructural morphology and fracture mode of Si_3N_4 which may benefit some properties and be detrimental to others.

The gains that have been attained over the past eight years in the fast fracture strength (MOR) of HPSN are significant, as shown in Fig. 8, and validate the GBE concept. Present work is aimed at combining the GBE with more traditional microstructural development approaches and considerable progress along these lines has been achieved.⁴⁶

CASE STUDY OF A SUCCESSFUL CERAMIC DESIGN - THE DARPA/DOE/FORD STATOR

We will now turn our attention from the engineering of ceramics to the engineering of a ceramic structure, by use of the iterative Brittle Materials Design approach. The best documented utilization of the iterative Brittle Materials Design approach is

44. Lange, F. F. "Dense Si_3N_4 Ceramics: Fabrication and Interrelations with Properties" in Processing of Crystalline Ceramics, H. Palmour, R. F. Davis, and T. M. Hare, ed., Plenum Publishing Co., New York, 1978, p. 597-614.
45. Prochazka, S. "Sintering of SiC " in Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1974, p. 239-252.
46. Knoch, H., Gazza, G. E., and Katz, R. N. "The Influence of Processing Parameters on Development of Microstructure in HPSN and its Correlation with Mechanical Properties", Presented at the 4th CIMTEC, St. Vincent, Italy, May 1979 (to be published).

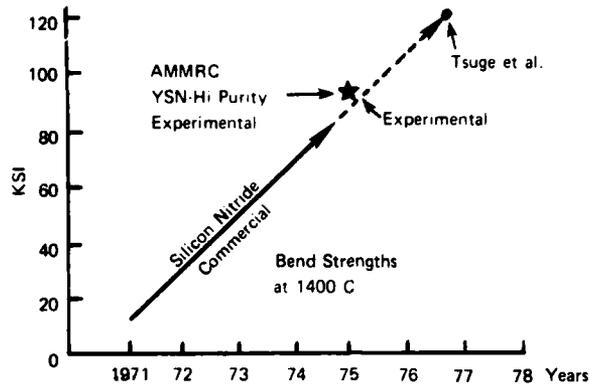


Fig. 8. Progress in the development of high temperature strength of HPSN since the inception of the ARPA Brittle Materials Design Program.

provided by the DARPA/DOE/Ford Program's stator development.⁴⁷ The initial goal of the program was to be a demonstration of an uncooled RBSN stator in an all-ceramic engine. A representative duty cycle was to be used with turbine inlet temperature (TIT) varying between 1930°F and 2500°F for an aggregate time of 200 hours. When it became apparent that lack of a 200-hour ceramic rotor capability would preclude engine demonstration within the existing program, the goal was redefined to demonstrate the stator over a representative engine duty cycle comprising 175 hours at 1930°F and 25 hours at 2500°F in a test rig (an engine minus rotors) shown in Fig. 9. Short duration engine tests (10 to 100 hr) were to be conducted with metallic and, later, ceramic rotors to validate the overall engine feasibility.

Figure 10 lists some of the systems considerations and requirements which were subjected to the trade-off analysis illustrated in Fig. 1. Injection-molded reaction-bonded silicon nitride (RBSN) and reaction-sintered silicon carbide were chosen to go through the iterative design/materials/process refinement procedure as illustrated in Fig. 2. This section will focus on the development of the RBSN stator, as it provides a more complete example of how various design procedures and qualification

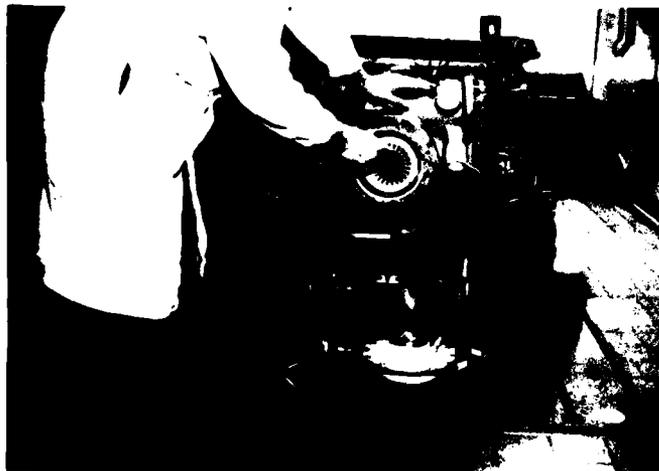


Fig. 9. Test rig.

47. Hartsock, D. L., and co-workers. "Test and Development of Ceramic Combustors, Stators, Nose Cones, and Rotor Tip Shrouds" in Ceramics for High Performance Applications-II, J. J. Burke, E. M. Lenoe, and R. N. Katz, ed., Brook Hill Publishing Co., Chestnut Hill, MA, 1978, p. 293-315.

STATOR FOR TYPICAL SMALL AXIAL AUTOMOTIVE G.T. (150-250 HP, ~100 KW)			
SELECTION CRITERIA: ● HIGH TEMPERATURE CAPABILITY			
● THERMAL SHOCK AND THERMAL FATIGUE RESISTANCE			
● CORROSION/EROSION/OXIDATION RESISTANCE			
● FABRICABILITY			
● STRENGTH			
MOST IMPORTANT SELECTION CRITERIA:			
● COST			
Duty Cycle	LIFE OR TBO	STEADY STATE, σ_{MAX}	TRANSIENT, σ_{MAX}
VERY SEVERE, MANY TRANSIENTS, 2500 F (TIT)	~3500 HR	~1-3,000 PSI	12,000 PSI
POSSIBLE ENGINEERING CERAMICS: INJ MOLD RBSN, SLIP CAST RBSN, R.S. SiC			
FAVORED CERAMIC/FABRICATION ROUTE: INJ MOLD RBSN 2.7 g/cc or R.S. SiC			

Fig. 10. Materials/design considerations.

tests were developed. The iterative development of this stator proceeded over a period of seven years during which five major iterations (plus many more subiterations) were carried out. These included four iterations of design and three iterations of materials, summarized in Fig. 11.

The original design, A in Fig. 11, included an integral first-stage rotor shroud and did not include an inner shroud. Individual vane segments were injection molded and bonded together with a silicon metal slip in the green state to form the full first-stage stator. The vane segments attained a nitrided density of 2.2 g/cc while the bond material had a somewhat lower density. During tests of this stator in the static engine rig the bonding material in the outer shroud often failed. More significantly, vane root thermal cracks were initiated during thermal transients. These lead to loss of vanes on light off or shut down. Two-dimensional finite element stress analysis was used to attempt to correct the thermal shock problems. This resulted in elimination of the integral rotor shroud and a thinning down of the outer stator shroud channel. Aerodynamic considerations lead to the introduction of inner vane shrouds. This first iteration, Design B, is shown in Fig. 11. Again vanes were injection molded as individual segments, bonded together, and nitrided to 2.2 g/cc. Design B lead to significantly longer test rig life than Design A. An important factor in the improved performance was the better shroud-to-vane mass balance in Design B. The vane cracking, which was not predicted by the 2-D analysis, moved to the mid-span of the vane, and outer shroud cracking continued as before. The use of a laboratory thermal shock rig (Fig. 12) allowed empirical evaluation of several modified vane profiles. Based on this empirical design input a second iteration, Design C, was defined in which the vanes had their trailing edges cut back. Stators were still assembled from individual segments and nitrided to 2.2 g/cc. These design changes with material being held constant increased maximum stator life time from ~10 hours to ~100 hours. It is significant to note that the Design C stator was used in the first major engine demonstration of 50 hours at up to 87% design speed and 1930°F. (Full speed occurs at 2500°F and therefore required a ceramic rotor, which was unavailable.)

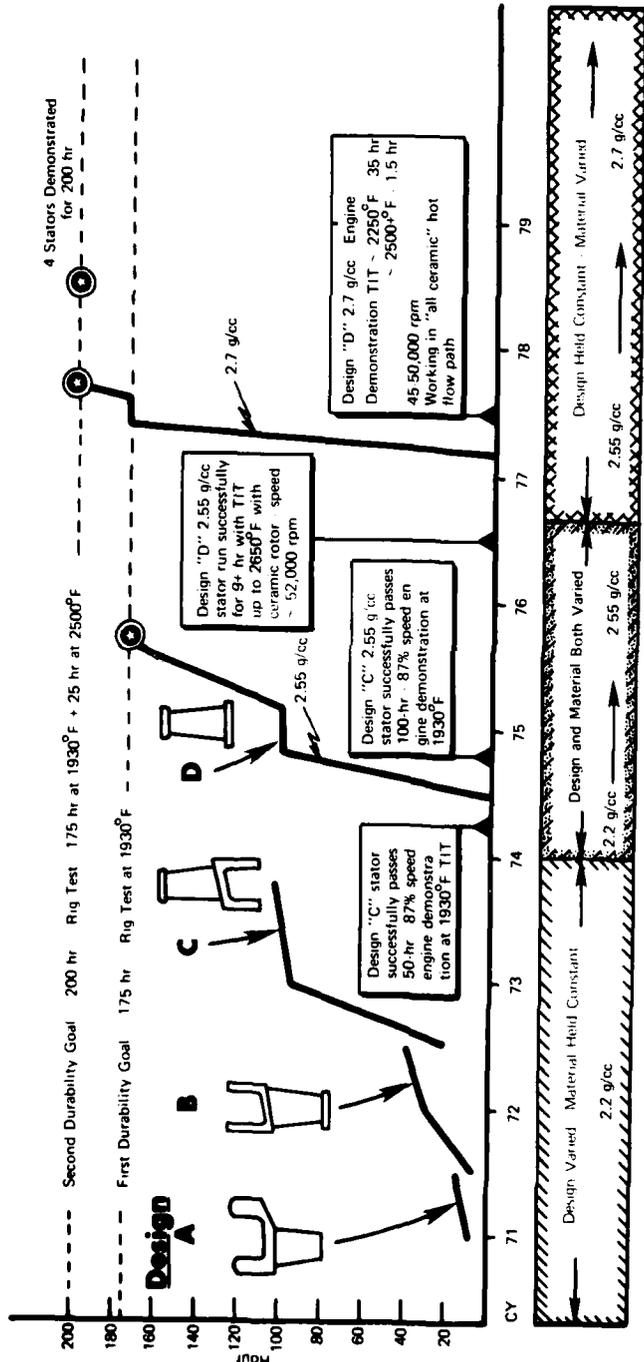


Fig. 11. Engineering development history - first stage RBSN stator ARPA/Ford engine - a case study of iterative "Brittle Materials Design".

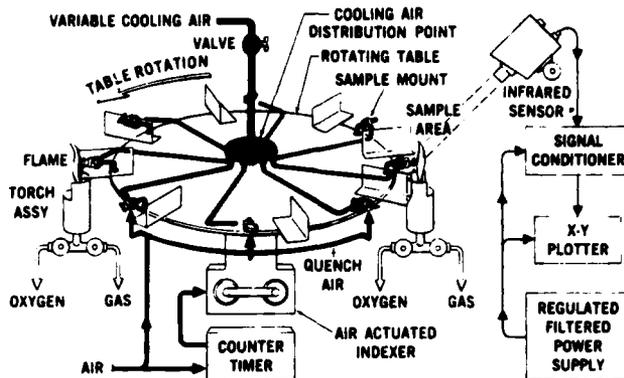


Fig. 12. Stator "laboratory" thermal shock test rig.

At this point failures in the bond joint between the individual vane segments were recognized as being due to oxidation. Factoring in the difference in thermal expansion of oxidized bond material and oxidized vane material (the two materials had different densities and thus oxidation rates) into a detailed 3-D analysis confirmed that in order to meet the program goals a one-piece stator would be required. Tooling for a one-piece stator was procured in the third design iteration D. This design had the same vane geometry as Design C but the outer shroud channels were removed and the gaps in the inner shroud were precisely defined. Simultaneously with the introduction of Design D, the first materials iteration, 2.55 g/cc RBSN, was incorporated. The combined effect of this improvement in design configuration and material enabled the program to achieve two important program goals: a 175-hour rig durability demonstration, and a 100-hour engine test demonstration. These are noted on Fig. 11.

Outer shroud cracking was still a problem with the Design D 2.55 g/cc materials, possibly related to oxidation of the RBSN. At this point it was considered that the elimination of the outer shroud cracking could be most directly addressed via materials improvement, thus design of the stator was frozen. The final iteration was the introduction of 2.7 g/cc material. This last iteration carried the stator to a 200-hour program demonstration as well as several engine tests with ceramic rotors at temperatures of up to 2650°F. Thus, the demonstrated durability of the first-stage stator increased from ~10 hours in 1971 to 200 hours, reproducibly demonstrated in 1977.

Between 1976 and 1978 several empirically derived proof tests were developed which allowed some degree of prediction as to which stators would survive the demonstration test; these included stator vane bend tests, Fig. 13, stator outer shroud pressure tests, Fig. 14; and an oxidative gain proof test, Fig. 15.

It is important to note that these predictive proof "tests," especially the one utilizing oxidative weight gain in a several-hour exposure, resulted from experience gained with working with the materials and components and could not have been anticipated in the advance of that experience. In the course of the iterative stator development, significant engine demonstrations were made which showed the stators would perform for 35 to 100 hours in a real engine environment, Fig. 11. It was also demonstrated that the Design D configuration stator made of reaction-sintered SiC was capable of attaining 200 hours of life. The U.S. Department of Energy is currently supporting further testing on both RBSN and reaction-sintered SiC stators in a more rigorous automotive duty cycle. Lives of over 250 hours incorporating over 15,000 severe thermal transient cycles have been demonstrated on RBSN stators in the DOE Program.²⁴

The DARPA/DOE/Ford stator is a case study in successful design of a ceramic structure. The next section will briefly review the status of several other ceramic engine programs where similar progress is being achieved.

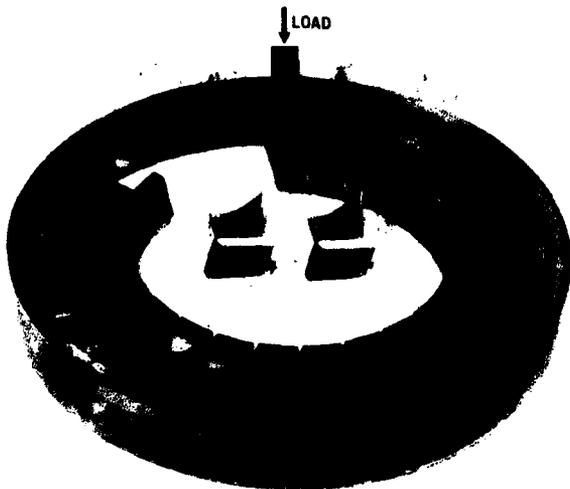


Fig. 13. Stator vane bend test.

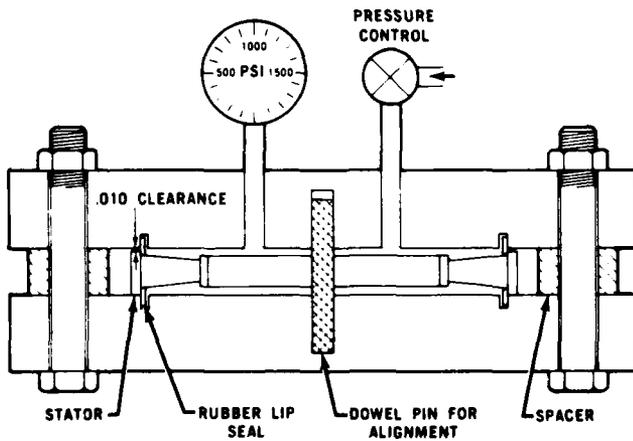


Fig. 14. Stator outer shroud pressure test.

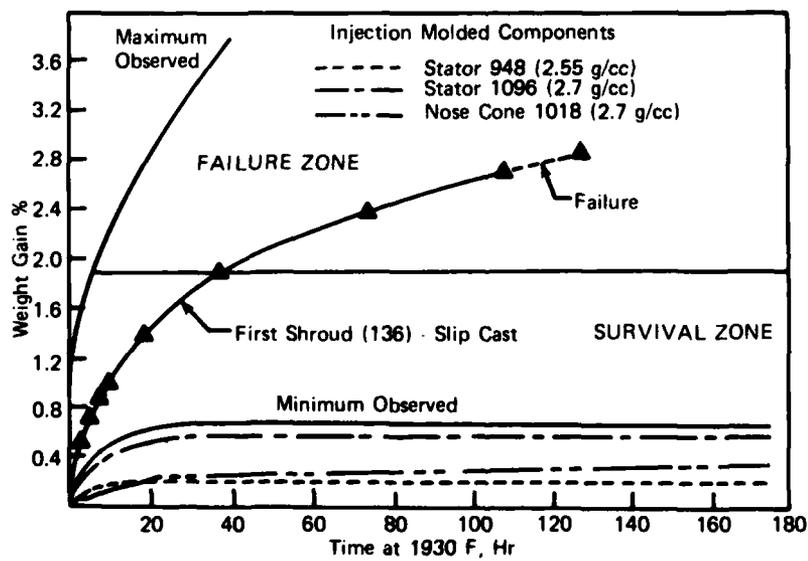


Fig. 15. Oxidative weight gain used to predict stator quality and survival.

RECENT PROGRESS ON CERAMICS FOR HEAT ENGINES

In the introduction to this paper the high payoff to be gained from the use of ceramics in heat engines was presented. We reviewed some of the difficulties in both the design and materials areas which are being addressed by the ceramic engine community. We next went through a case study to show that these difficulties, while formidable, could be surmounted. In this section I should like to report some rather impressive achievements on several ceramic engine demonstration programs currently underway in the United States. These are listed in Table 4. Of particular note, though not listed in the table, is the current interest in the use of ceramics in conventional gasoline and diesel engines. Phoenix⁴⁸ has recently reviewed the use of sintered SiC in such applications including diesel pre-chambers, valve lifters, and turbochargers.

TABLE 4. Significant Achievements in Current Ceramic Heat Engine Programs

Program [Reference]	Type of Engine	Major Achievements
DARPA/DOE/Ford (22)	~200-hp Regenerated, Axial Single Shaft, 2500° F TIT	<ul style="list-style-type: none"> • All stationary ceramic components in reaction-bonded Si₃N₄ demonstrated 200 hours life in engine rig to 2500° F • Duo-density Si₃N₄ rotor demonstrated 200 hours at ~2200° F TIT, 50,000 rpm • First-stage ceramic hardware run in an engine 36.5 hours, 2100-2500° F, 40,000-50,000 rpm
MERADCOM/Solar (49)	~10-kW Turbogenerator, Radial, ≈1700° F TIT	<ul style="list-style-type: none"> • Run ~50 hours with ceramic vanes, producing ~10 kW • Run 100+ hours with all-ceramic nozzle stage, producing ~10 kW
DARPA/AiResearch (50)	~1000-hp Simple Cycle 3-Stage, 2200° F TIT	<ul style="list-style-type: none"> • Integration of over 100 separate ceramic components in a high performance engine. Run at full speed and temperature in excess of 2 hours, including 2 shutdowns and starts. Ceramics demonstrated 200+ hp increase over base engine
DDA/DOE (51)	~350-hp Regenerated 2-Shaft, 2265° F TIT	<ul style="list-style-type: none"> • Ceramic stators run in excess of 1900 hours at 1900° F TIT. Ceramic vanes in vehicle on the road for over 100 hours, including shock and vibration testing
Cummins/TARADCOM (52)	Single-Cylinder, Turbocharged Diesel Engine	<ul style="list-style-type: none"> • Ceramic piston cap completed scheduled 250 hours full performance engine test. Meets all program goals

48. Phoenix, R. C. "Design and Performance Experience of SiC Piston Engine Components", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
49. Metcalfe, A. G. "Ceramics for Small Gas Turbine Power Generators", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
50. Harper, J. "Overview of DARPA/NAVAIR Ceramic Engine Demonstration Program", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
51. Helms, H. E. "Ceramics for Truck and Bus Gas Turbine Engines", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).
52. Kamo, R. "Ceramics for the Adiabatic Turbocompound Engine", Presented at the 6th Army Technology Conference: Ceramics for High Performance Applications-III, Orcas Island, WA, July 1979 (to be published).

CONCLUDING REMARKS

When, on behalf of the Conference organizing committee, Dr. Roger Davidge invited me to give this talk, he suggested that I might want to give a balanced perspective of where we stand today with respect to ceramics for engines. Briefly, we have come a long way and for most applications we still have a long way to go. At the onset of the ARPA Brittle Materials Program, Arthur McLean and I presented a paper on "RBSN as a Small Gas Turbine Nozzle Material"⁵³ at a meeting of the British Ceramic Society, here at Cambridge University. At that time we had barely attained 25 hours in a test rig; now as discussed above that stator has demonstrated its 200-hour goal. Moreover, today ceramics are operating in several demonstration engines on the highway, on the racetrack, and producing electrical power.^{48,49,51} That's a long way in seven years! However, we must bear in mind that these are still one-of-a-kind demonstration engines. For most applications, ceramic components are not yet ready for introduction into commercial engines.

Much progress has been and is continuing to be made in improved ceramic materials. Similarly, with the exception of life prediction, design is proceeding well. The problems are the areas of ceramic attachment, and production reliability and reproducibility for components. Also, these reliable materials and designs must eventually be made affordable.

There is a wide spectrum of possible applications of ceramics to a wide variety of engines. Some applications such as valve lifters, exhaust port liners or diesel pre-chambers can be viewed as fairly near term. Reliable and affordable ceramic rotors for 2500°F and above are probably still a long-term development. Given the rate of progress demonstrated over the past seven years and given adequate resources, such a 2500°F turbine rotor is certainly attainable.

Perhaps the best summation of where we are with ceramic engine technology was given by Arthur McLean at last month's "Ceramics for High Performance Applications - Reliability" meeting at Orcas Island, Washington: "Considerable R&D is needed to take us from the stage of *ceramics can work* to the stage of *ceramics won't fail*." That is the present challenge for all of us in the ceramic engine area.

ACKNOWLEDGMENTS

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53. Katz, R. N., and McLean, A. F. "RBSN as a Small Gas-Turbine Nozzle Material" in Proceedings Brit. Cer. Soc. v. 22, 1973, p. 409-427.

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