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CERAMICS FOR VEHICULAR ENGINES: STATE-OF-THE-ART

R. NATHAN KATZ
CERAMICS RESEARCH DIVISION

June 1980

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ABSTRACT

↙ The use of silicon-based, high performance ceramics offers significant potential for energy conservation in heat engine technology. In particular, three families of silicon-based engineering ceramics, the alumino silicates, the silicon nitrides, and the silicon carbides, possess unique combinations of properties which make these materials very attractive candidates for components in gas turbine, diesel, and Stirling engines. After a brief discussion of the design process with brittle materials, this paper will review some of the relevant properties of various members of each of these three families of engineering materials, as processed by a variety of routes. Some advantages and disadvantages of the major materials/process combinations will be discussed. Selected examples of applications of a variety of materials/process combinations as applied to vehicular engine technology will be presented.

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CONTENTS

	Page
INTRODUCTION.	1
THE FAMILIES OF HEAT ENGINE CERAMICS	
Alumino Silicate-Based Ceramics.	4
Silicon Nitride-Based Ceramics	5
Silicon Carbide-Based Ceramics	11
CERAMICS IN VEHICULAR ENGINE DEMONSTRATION PROGRAMS	13
Ford/DARPA/DOE Vehicular Engine.	14
Detroit Diesel Allison/DOE-404-4 Ceramic Engine Development Program.	15
The AS Regenerator Programs.	15
The Cummins/TARADCOM Adiabatic Turbocompound Diesel.	16
SUMMARY	16
REFERENCES.	17

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INTRODUCTION

The energy problem, environmental concerns, and materials supply considerations are the driving forces behind the current emphasis on ceramic materials technology for vehicular engines. High temperature 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 of managing a cooling fluid, as required in current engine technologies. Examples of the potential gains to be obtained for various generic classes of engines are presented in Table I (ref. 1-4). It is important to note that worthwhile gains can be obtained for several types of engine at temperatures between 2000 and 2300 F, even though automotive gas turbines will probably require a 2500 F capability.

Table I. CERAMIC TECHNOLOGY PAYOFF FOR SOME TYPICAL HEAT ENGINES

Engine Type	H.P. Range	Configuration	$\sim \Delta$ SFC*	$\sim \Delta$ Power	Ref
AUTOMOTIVE GT	$\sim$ 200	REGENERATED, SINGLE-SHAFT, 2500 F, TIT	-27%	0	[1]
TRUCK GT	$\sim$ 350	REGENERATED, 2-SHAFT, 2265 F, TIT	-17%	+30%	[2]
LIGHT SHIP GT	$\sim$ 1000	SIMPLE CYCLE, 3-STAGE, 2500 F, TIT	-10%	+40%	[3]
DIESEL	$\sim$ 500	ADIABATIC - TURBO COMPOUND $T_{max} \approx 2000$ F	-22%	+37%	[4]

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

In order to attain the increased efficiencies shown in Table I, engine designers must learn to design with brittle materials. Design with brittle materials requires a very precise definition of the state of stress at every point in the component. Dukes (ref. 5) has pointed out that the most difficult aspect of high temperature design with brittle materials is the definition of, and subsequent design to minimize, thermal stresses. Thus, a realistic expectation of exploiting the potential of ceramics in heat engines requires the availability of two technologies. These are: an analytic design capability to predict the thermal and stress environments from point to point in a component, and high temperature engineering materials with low thermal expansions in order to minimize thermal stresses. Advances in 2-D and 3-D computerized finite element thermal and stress analysis techniques have provided the former, and the development of alumino silicates, silicon nitrides and

- 1 J.L. Klann, "Advanced Gas Turbine Performance Analysis", 8th Summary Report, Automotive Power Systems Contractors Coordination Meeting, ERDA-64, May 1975, p. 173.
- 2 H.E. Helms and F.A. Rockwood, "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.
- 3 F.B. Wallace, A.J. Stone and R.B. Nelson, "Ceramic Component Design ARPA/Navy Ceramic Engine Program", Chapter 34 in Ceramics for High Performance Applications-II, Eds. J.J. Burke, E.M. Leno and R.N. Katz, Brook Hill, pp. 593-624.
- 4 R. Kamo, "Cycle and Performance Studies for Advanced Diesel Engines", Chapter 47, ibid, pp. 907-922.
- 5 W.H. Dukes, "Handbook of Brittle Material Design Technology", AGARDograph No. 152, North Atlantic Treaty Organization, February 1971, p. 5.

improved silicon carbides have provided the latter. Both of these developments reached a sufficient level of maturity in the late 1960's for various research programs for demonstration of ceramics in gas turbines to be initiated (ref. 6-8). The results of these early programs were sufficiently encouraging for additional efforts to be initiated in both the gas turbine (ref. 9) and diesel (ref. 10) areas. As will be discussed below, and in several other papers at this conference, considerable success in demonstrating the feasibility of ceramics in both gas turbines and diesels has been attained.

While low thermal expansion is necessary for a ceramic engine material, other properties must be considered. All of the materials properties and requirements listed in Table II must be adequate. In particular, the time, temperature, stress, and environmental dependencies of the mechanical properties must be known (or predictable) for the entire duty cycle. One must also know the effects of processing method on these properties. Having such information, the designer is then in a position to perform trade-off studies as in Fig. 1, and specify an optimum design/material/processing combination for a given component in the system under consideration. Once the design/material/process for a given component has been selected, validation by rig and/or engine tests usually requires several passes through the

Table II. GENERAL PROPERTIES REQUIRED OF A CERAMIC
HEAT ENGINE COMPONENT

LEVEL I.	<u>TECHNICAL FEASIBILITY DEMONSTRATION</u>
	• THERMAL SHOCK/THERMAL FATIGUE RESISTANCE • NECESSARY TEMPERATURE CAPABILITY • ADEQUATE STRENGTH AND CREEP BEHAVIOR AS A FUNCTION OF TEMPERATURE • NECESSARY LONGEVITY IN THE ENGINE ENVIRONMENT
LEVEL II	<u>ENGINEERING FEASIBILITY DEMONSTRATION</u>
	• CONSISTENCY OF PROPERTIES IN AS-FABRICATED COMPONENTS • ADEQUATE RELIABILITY ATTAINED IN COMPONENTS • COST-EFFECTIVE POTENTIAL
LEVEL III	<u>TECHNOLOGY IMPLEMENTATION</u>
	• DEMONSTRATED BENEFIT/COST FOR THE TECHNOLOGY MUST BE SUFFICIENTLY HIGH TO WARRANT IMPLEMENTATION VERSUS EXISTING OR OTHER ALTERNATE TECHNOLOGIES

- 6 Ceramics for Turbines and Other High Temperature Engineering Applications, Ed. D.J. Godfrey, Proceedings of the British Ceramic Society, 22, (1973).
7 Ceramics for High Performance Applications, Eds. J.J. Burke, A.E. Gorum and R.N. Katz, Brook Hill Publishing Co., Chestnut Hill, MA (1974).
8 A.F. McLean, E.A. Fisher and D.E. Harrison, "Brittle Materials Design/High Temperature Gas Turbine", AMRC CTR 72-3, March 1972.
9 Ceramics for High Performance Applications-II, Eds. J.J. Burke, E.M. Lenoe and R.N. Katz, Brook Hill Publishing Co., Chestnut Hill, MA (1978), Chapters 25, 26, 27, 34, 42, 43, 44, 49 and 50.
10 Ibid, Chapters 45 and 47.

iterative design scheme shown in Fig. 2. It is important to note that there is unlikely to be one optimum material/process 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 processing options.

Several state-of-the-art alumino silicate, silicon nitride, and silicon carbide-based ceramic materials have now progressed through the iterative design process to the point of successful component demonstration in test rigs and/or actual engines. These materials should be considered as representative of the first generation of their respective families of materials. Table III shows some of the members of the current three families of engine ceramics together with some representative properties. The next section of this paper will review the processing, properties, and trade-offs for these three families of engine ceramics in more detail.

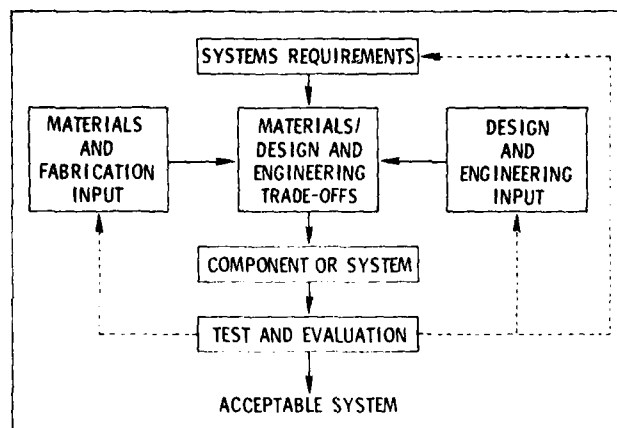


Figure 1. The engineering trade-off logic for introducing a new material or design into a system.

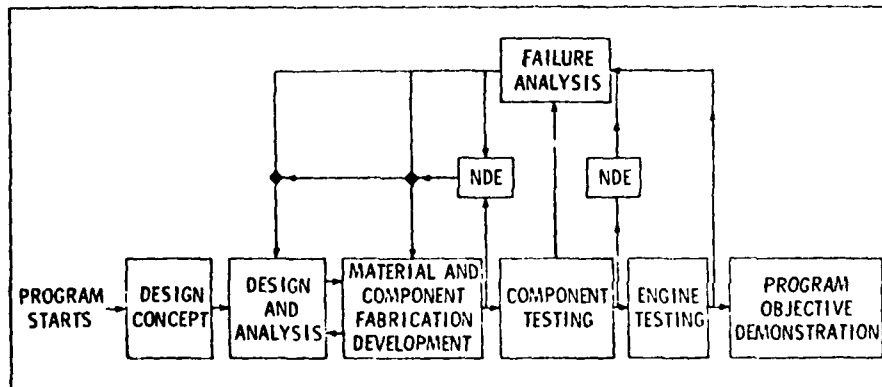


Figure 2. Iterative "brittle materials design" scheme.

Table III. FAMILIES OF HEAT ENGINE CERAMICS AND TYPICAL BULK PROPERTIES

		MOR (RT, psi)	MOR (1000 C, psi)	MOR (1375 C, psi)	RT - ξ (psi/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 NITRIDES	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 (α 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 FAMILIES OF HEAT ENGINE CERAMICS

Alumino Silicate-Based Ceramics

The alumino silicate families of ceramics in the lithium aluminum silicate (LAS), magnesium aluminum silicate (MAS) and aluminum silicate (AS) systems can cover a wide range of compositions and phases. In general, they are all formed as glasses, then crystallized. In some cases the parts are formed as glass, then crystallized preserving component shape (a great advantage of this class of materials). In other cases a crystallized frit is ground into powder which is then sintered (this is sometimes necessary to control microstructure). For all practical purposes the glass-ceramics of interest in engines are LAS as β -Spodumene, MAS as Cordierite and AS as Keatite. MAS glass ceramics have been considered for turbine wheels and stators (ref. 11), but were abandoned due to temperature limitations. LAS (Cervit 140) has been evaluated as an insulator in the Westinghouse/DARPA portion of the DARPA Brittle Materials Design Program (ref. 12). Again, temperature limitations led to abandonment of this approach. Massive LAS or MAS may have some potential in diesel engine technology, but a successful demonstration has yet to be made. Thus, this paper will not consider the use of the alumino silicate glass ceramics as bulk structural components. The area we will focus on, and where these materials have demonstrated considerable success, is in their use as heat exchangers (regenerators and recuperators) for gas turbine and possibly Stirling engine use.

Because glass ceramic materials for heat exchanger use are fabricated as rather thin-walled honeycomb-like matrices, the properties of these materials are presented

11 C.R. Gostelow and J.E. Restall, "Ceramics with Potential for Gas Turbine Application", Reference 6, p. 117.

12 D.G. Miller, C.A. Anderson, S.C. Singhal, F.F. Lange, E.S. Diaz, R. Kossowsky and R.J. Bratton, "Brittle Materials Design, High Temperature Gas Turbine Material Technology", AMMRC CTR 76-32, Vol IV, December 1976.

as properties of the matrix structure. Also, owing to proprietary considerations, exact compositions are not provided. Thus, materials will merely be designated MAS, LAS, or AS with no further identification. The regenerator matrices are fabricated by three principal techniques as shown in Fig. 3. Table IV lists the important properties for candidate regenerator matrices (ref. 13). Most of the properties listed in Table IV need no explanation, however, several terms may not be familiar. Thermal strain difference (or thermal excursion) refers to the entire thermal excursion of the matrix over a given temperature range in parts per million. Because there is a regime of contraction as well as expansion for these materials this term, rather than thermal expansion, is used. Strain tolerance is the modulus of rupture divided by the modulus of elasticity (MOR/MOE) for the matrix. The thermal stress factor is the strain tolerance divided by the thermal excursion. Based on the data of Table IV, AS appears to be the preferred regenerator material. However, MAS materials are considered to be capable of considerable development and thus are still considered attractive candidates for regenerators.

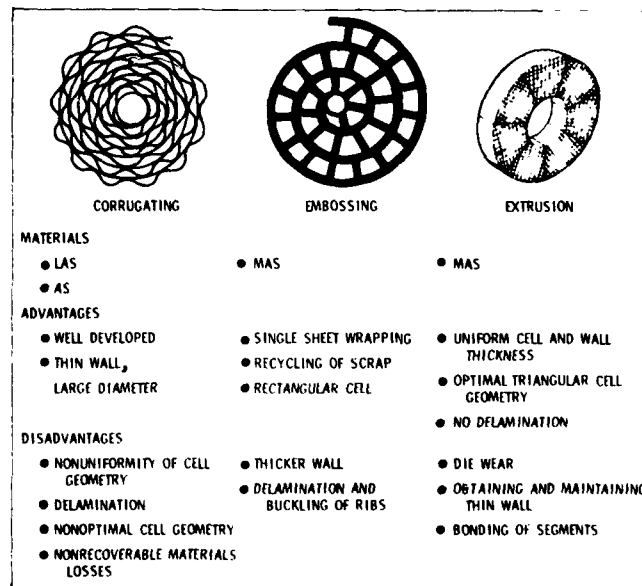


Figure 3. Ceramic regenerator manufacturing techniques.
(After Fucinari, Reference 13)

Silicon Nitride-Based Ceramics

The silicon nitride families of ceramic materials include:

Chemically Vapor-Deposited Silicon Nitride (CVD-SN)

Hot-Pressed Silicon Nitride (HPSN)

Reaction-Bonded Silicon Nitride (RBSN)

Sintered Silicon Nitride (SSN)

B' SiAlON's (SiMON)

13 C.A. Fucinari, Chapter 19 in reference 9.

Table IV. REGENERATOR THERMAL STRESS CAPACITY (Reference 13)

MATRIX NO.	1	2	3
MANUFACTURING PROCESS	CORRUGATING	EMBROSSING	EXTRUSION
MATERIAL COMPOSITION	AS	MAS	MAS
MATRIX WALL POROSITY (%)	9.8	39.6	35.2
TEMPERATURE (°F.)			
H.P. INLET	450	450	450
L.P. INLET	1470	1470	1470
THERMAL STRAIN DIFFERENCE (Δ PPM)	70	800	930
MODULUS OF ELASTICITY (PSI) X 10-6			
TANGENTIAL (E_T)	.65	1.20	.59
RADIAL (E_R)	.04	.90	.63
MODULUS OF RUPTURE (PSI)			
TANGENTIAL (MOR_T)	650	420	340
RADIAL (MOR_R)	140	390	330
STRAIN TOLERANCE (PPM)			
TANGENTIAL	1000	350	575
RADIAL	3500	435	525
THERMAL STRESS FACTOR			
TANGENTIAL (σ_T)	14.3	.44	.62
RADIAL (σ_R)	50.0	.54	.56
MAX. TANGENTIAL TENSILE STRESS (σ_{TMAX})-PSI	45	700	490
THERMAL STRESS SAFETY FACTOR (S.F.)	14.4	.60	.70

CVD-SN in very thin layer form is currently an important electronic material (ref. 14). Building on this thin film technology, some research in the use of CVD-SN coatings is in progress, but bulk CVD-SN has yet to be demonstrated as a structural material. Thus, we will not consider it further. The other four classes of silicon nitrides are each, in themselves, broad families of engineering ceramics.

Hot-Pressed Silicon Nitride can be produced by either uniaxial (ref. 15) or hot isostatic pressing (ref. 16). Most work to date has utilized the conventional uniaxial route. One generally starts with α - Si_3N_4 powder and adds a densification aid such as: MgO , Y_2O_3 , ZrO_2 , CeO_2 , or $SiBeN_2$. Depending on the purity and phase composition of the starting Si_3N_4 powder, the percentage and type of additive, milling and mixing procedures, and hot pressing parameters (T,t), one can obtain a very wide range of strength, strength versus temperature, creep, fracture toughness, or oxidation behaviors. Illustrative of such variety of behavior, the strength versus temperature curves of HPSN's, as a function of the foregoing processing variables, are shown in Fig. 4 (ref. 17). To reiterate an important point, one cannot look at the data in Fig. 4 and make an arbitrary judgment that one material is better than another. The concept of "better" depends on the application requirement. The lower curve may be better for a given application than one of the higher curves.

- 14 J.T. Milek, Silicon Nitride for Microelectronic Applications - Handbook of Electronic Materials, Vol 6, IFI-Plenum, New York (1972).
- 15 T. Vasilos, "Densification of Nitrides by Hot Pressing", Nitrogen Ceramics, Noordhoff, Leyden, p. 367 (1977).
- 16 H. Larker, J. Adlerborn and H. Bohman, "Fabricating of Dense Si_3N_4 Parts by Hot Isostatic Pressing", SAE Paper 770335, March 1977.
- 17 A.F. McLean, A.E. Gorum, E.M. Lenoe and R.N. Katz, "Status of Vehicular Turbine Engine Ceramics," Proceedings of the 3rd Conference on Gas Turbines in a Marine Environment, U. Bath (1976).

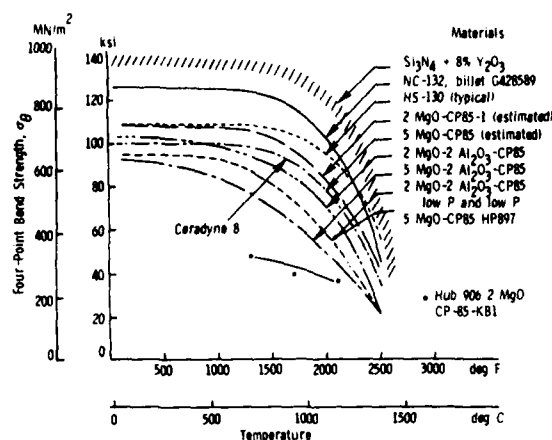


Figure 4. Flexural strength of various types of HPSN (Reference 17).

Almost all HPSN's exhibit MOR values of 100,000 psi (690 MPa) or greater at RT and retain these values to 1000 or 1100 C (dependent on amount and type of additive), have high K_{IC} (~ 5 to $8 \text{ MN/m}^{3/2}$), and excellent thermal shock and erosion resistance.

Hot-pressed silicon nitride with MgO as an additive, typified by NC-132 material, most nearly approaches a mature engineering material. HPSN with Y₂O₃ additives typically has higher strengths at both room and elevated temperatures (see Fig. 4), as well as better oxidation resistance and K_{IC} and less slow crack growth than MgO containing HPSN. However, this material has been plagued with an intermediate temperature (~ 1000 C) oxidation problem. While it appears that the post-fabrication nitridation treatment demonstrated by Gazza, Knoch, and Quinn (ref. 18) can overcome this problem, more work needs to be carried out before HPSN with Y₂O₃ is as developed as the MgO-containing varieties. Very high strengths have been attained by Tsuge, et al., (ref. 19), with Y₂O₃ + Al₂O₃ as additives using the grain boundary crystallization approach. The high cost associated with diamond machining of HPSN from simple billets to complex geometry components has led to research in near net shape hot pressing in several laboratories. Significant progress in hot pressing Si₃N₄ to near net shape has been demonstrated by Ford (ref. 20), AMMRC (ref. 21), and Annawerk (ref. 22) for rotor hubs; and AMMRC for diesel piston caps. Such work has demonstrated the utility of hot pressing in engine development programs, even though it is recognized that conventional or near net shape hot pressing is not likely to be applicable to automotive mass production (ref. 23).

- 18 G.E. Gazza, H. Knoch and G.D. Quinn, "Hot Pressed Si₃N₄ with Improved Thermal Stability", Am. Ceram. Soc. Bulletin 57, [11], (1978) pp. 1059-60.
- 19 K. Komeya, A. Tsuge, H. Hashimoto, T. Kubo and T. Ochiai, "Silicon Nitride Ceramics for Gas Turbine Engines", Gas Turbine Society of Japan, Paper No. 65, Tokyo Joint Gas Turbine Conference, May 1977.
- 20 See Chapters 1, 11 and 15 in Reference 9.
- 21 A.F. McLean and E.A. Fisher, "Brittle Materials Design, High Temperature Gas Turbine", AMMRC CTR 77-20, August 1977, p. 45.
- 22 See Chapter 28 in Reference 9.
- 23 R.N. Katz and E.M. Lenoe, "Ceramic Rotors for Small Automotive Gas Turbine Engines - Technology Assessment", 8th Summary Report, Automotive Power Systems, Contractors Coordination Meeting, ERDA-64, May 1975, p. 181 ff.

Reaction-Bonded Silicon Nitride has been developed largely to obtain a readily formable (little machining required), low-cost material. In contrast to HPSN, RBSN maintains its strength to temperatures beyond 1400 C, as shown in Fig. 5 (ref. 24). Some RBSN can exhibit creep rates significantly below those of HPSN (ref. 25). As a consequence of the reaction bonding process, RBSN is of necessity at least 10% porous which makes it less oxidation resistant than HPSN at intermediate temperatures (ref. 26), and limits its strength to less than 60 ksi (415 MPa).

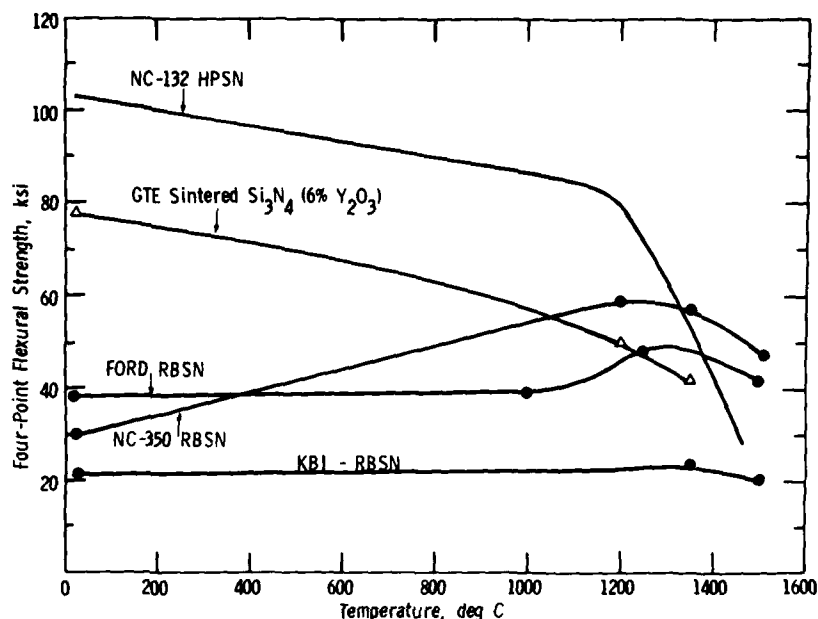


Figure 5. Flexural strength of silicon nitride materials versus temperature (Reference 24).

The fabrication of RBSN components begins with a silicon metal powder preform made by slip casting, dry pressing, flame spraying, injection molding, or other techniques. The preform is then nitrided in an atmosphere of pure N_2 or N_2+H_2 at either a preselected temperature schedule (ref. 27) or now more usually using a nitrogen demand control cycle (ref. 28). The process of nitriding such a Si preform is a remarkable, if still somewhat imperfectly understood event. For $3Si_{(s)} + 2N_{2(g)} \rightarrow Si_3N_{4(s)}$, there is a 23% expansion in the solid volume compared to Si; yet, when this reaction is carried out on a preform there is essentially no change in dimensions ($\sim 0.1\%$). The reason for this appears to be that the Si_3N_4 forms in the void space of the Si preform

24 D.C. Larson and C. Walther, "Property Screening and Evaluation of Ceramic Vane Materials", Interim Report No. 4, ITT Research Institute on AFML Contract F 33615-75-C-5196, October 1977.

25 D.C. Larson, S.A. Bortz, R. Ruh and N.M. Tallan, Chapter 36 in Reference 9.

26 A.F. McLean, Chapter 1 in Reference 9.

27 D.R. Messier and P. Wong, Chapter 8 in Reference 7.

28 P. Wong and D.R. Messier, "Procedure for Fabrication of Si_3N_4 by Rate Controlled Reaction Sintering", Am. Ceram. Soc. Bulletin 57 [5], 1978, p. 525-6.

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Watertown, Massachusetts 02172
CERAMICS FOR VEHICULAR ENGINES:
STATE-OF-THE-ART - R. Nathan Katz
Monograph Series AMMRC MS 80-2, June 1980,
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via vapor phase or surface diffusion reaction processes. The net result is that what appears to be a rather complex series of processing steps, and what is a complex series of chemical reactions, yields a product of great technological import--an engineering ceramic which can be mass produced to tight dimensional tolerances with little or no machining and at low cost.

Reaction-bonded silicon nitride has improved considerably over the past few years, as shown in Fig. 6. Nevertheless, where high strength, more oxidation resistant material is required, it would be desirable to have a readily fabricable, fully dense silicon nitride. This has been the impetus for the development of sintered silicon nitride.

REACTION-BONDED SILICON NITRIDES		
RECENT ADVANCES:		
● INCREASING COMPONENT DENSITIES 2.2 g/cc → 2.7 g/cc		
● RATE-CONTROLLED REACTION BONDING → IMPROVED MATERIALS UNIFORMITY		
Property	1972	1978
ρ (g/cc)	2.2	2.7
4-Pt MOR-RT (ksi)	~17	~36
Stress Rupture at 2200 F, 30 ksi	Failure	>300 Hour Suspension
Oxidation - Weight Gain in 200 Hours at 1900 F (Worst Case)	~6%	~0.75%

Figure 6. Recent advances in reaction-bonded silicon nitrides.

Sintered Silicon Nitrides are a rather recent development. Although Si_3N_4 was sintered prior to 1976 (ref. 29), the balance between dissociation of the Si_3N_4 and densification under the conditions that were used was such that densities of only 90% of theoretical were obtainable. Indeed, the possibility of sintering fully dense silicon nitride at reasonable pressures was still an open question as late as 1976. Since then several groups have succeeded in producing sintered Si_3N_4 of at least 95% density, and a few groups have obtained greater than 99% density. Gazza (ref. 30) has reviewed the status of SSN and it is summarized in Table V. Figure 5 shows SSN material with strengths between HPSN and RBSN. Some more recent experimental SSN materials have approached the properties of HPSN. SSN has been formed by injection molding and, provided isotropic shrinkage can be obtained, components will require little machining. It is also possible that sintered Si_3N_4 bodies of over 95% T.D. may be used as preforms for cladless "hot isostatic pressing". Such a development would be a major breakthrough toward attaining mass producible, affordable, high performance components such as rotors.

29 G.R. Terwilliger and F.F. Lange, "Pressureless Sintering of Si_3N_4 ", J. Mat. Sci. 10 [7], 1975, p. 1169-1174.

30 G. Gazza, Chapter 53 in Reference 9.

Table V. STUDIES OF SINTERING OF Si_3N_4
(See Reference 30 for Individual References in this Table)

Investigator	Starting Si_3N_4 Powder	Additives	Temp/Pressure Regime	Properties
Terwilliger Lange (7)	AME (CP-85)	5 w/o MgO	1300-1700 C 0.1 MPa N_2	85-90% dense product
Mitomo (8)	Denki Kagaku Co. Ltd (>95% β phase)	5 w/o MgO; La_2O_3 Y_2O_3 ; Y_2O_3 - Al_2O_3 ; Al_2O_3 ; Ga_2O_3 - Al_2O_3 ; AlN - Al_2O_3	1450-1900 C 1.0 MPa N_2	90-95% dense with MgO; 70-85% dense with others
Priest, Giazza (10)	GTE Sylvania (SN-502)	20 w/o CeO_2 ; Y_2O_3	1650-1950 C 2.0 MPa N_2	90-95% dense; 345-448 MPa MOR at RT; 379 MPa MOR at 1300 C
Rowcliffe, Jorgensen (13)	GTE Sylvania (SN-402) KBI-AME (CP-85)	4-17 w/o Y_2O_3	1560-1750 C 0.2 MPa N_2	90-99% dense; 527-542 MPa MOR at RT
Oda, Kaneno, Yamamoto (14)	AME (CP-85)	1.25 w/o BeO -3.75 w/o MgO 5.0 w/o CeO_2	1600-1800 C 0.1 MPa N_2	814 MPa MOR at RT
Buljan, Kleiner (15)	GTE Sylvania (SN-402) (SN-502) (amorphous and high α -phase)	rare earth based additions	1600-1750 C 0.1 MPa N_2	>97% dense product; 690 MPa MOR at RT
Masaki, Kamigaito (9)	β - Si_3N_4 (80% β) or Si_3N_4 and Spinel (85% α -10% Spinel)	5-15 w/o MgO; Al_2O_3 ; MgAl_2O_4	1650-1900 C 0.1 MPa N_2	>96% dense; 690 MPa MOR at RT
Greskovich, Prochazka, Rosolowski (3)	G.E.-In-House (amorphous)	1-5 w/o Be_3N_2 ; Mg_2Si ; Mg_3N_2	1800-2000 C 5-8 MPa N_2	98% dense products

SiALON's represent an important new class of ceramic materials which are solid solutions of metal oxides in the β - Si_3N_4 lattice. These solid solutions distort the lattice to β' - Si_3N_4 lattice (hence, β' -SiALON's). SiALON's were originally developed from Al_2O_3 solid solution, but additions of MgO, BeO, Y_2O_3 , etc., to Si_3N_4 have all been found to yield β' solid solutions, as well as a variety of other phases. SiALON's have been developed with a view toward application in heat engines (ref. 31 and 32). However, these materials are still in early development and have not been used in engine demonstration programs to date. It is likely that they will play a role in future engine programs. SiMON phases are of major importance in determining and controlling the grain boundary phases which in turn control the behavior of HPSN and SSN. Therefore, understanding phase relationships

31 W.J. Arrol, Chapter 34 in Reference 7.

32 R.J. Lumby, B. North and A.J. Taylor, Chapter 46 in Reference 9.

in these systems is of current importance. These phase relationships are discussed in the papers of Prof. K. H. Jack, in this conference, and elsewhere (ref. 33-36).

Silicon Carbide-Based Ceramics

The silicon carbide-based families of ceramic materials include:

- Hot-Pressed SiC (HP-SiC)
- Reaction-Sintered (or Bonded) SiC's (RS-SiC)
- Sintered SiC's (S-SiC)
- Silicon Carbide/Silicon Composites
- Chemically Vapor-Deposited Silicon Carbide (CVD-SiC)

Hot-Pressed SiC can be formed with various densification aids, but only HP-SiC utilizing Al_2O_3 additions has been considered for high performance engine applications (ref. 37). Properties of NC-203, the most mature example of this material, are shown in Table III, Figure 7, and given in some detail in reference 12. While HP-SiC is not as strong as HPSN at low temperatures, it retains useful strength out to ~ 1400 C. Thus, it might be an attractive material where high strength is required at

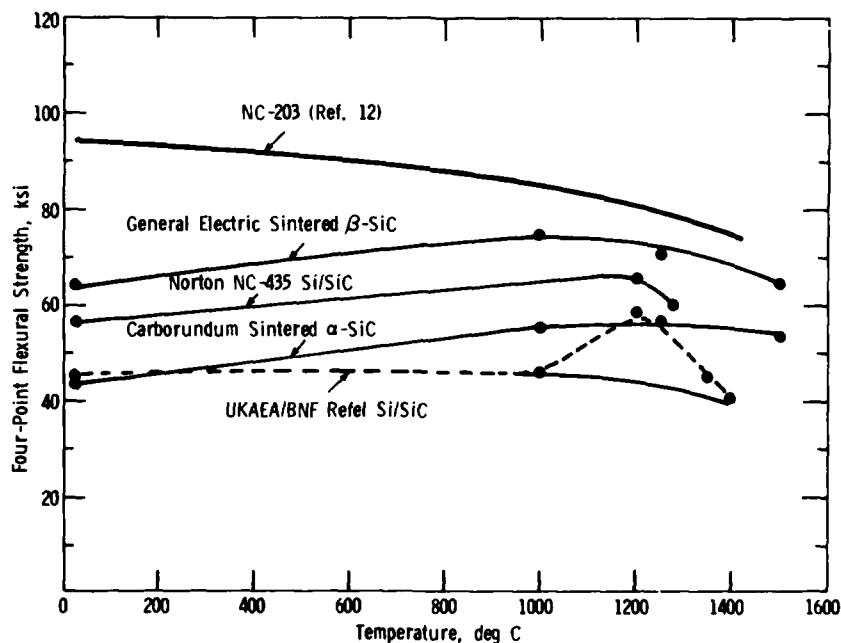


Figure 7. Flexural strength of silicon carbide materials versus temperature (Reference 24).

33 K.H. Jack, See Contribution to this Volume.

34 K.H. Jack, Chapter 14 in Reference 7.

35 K.H. Jack, Chapter 56 in Reference 9.

36 K.H. Jack, "SiAlONs and Related Nitrogen Ceramics", J. Mat. Sci. 11 (1976), pp. 1135-1158.

37 F.F. Lange and J.L. Iskoe, Chapter 11 in Reference 7.

1400 C or beyond. The major drawback of this material, particularly from the vehicular engine viewpoint, is the need to diamond-machine parts from a hot-pressed billet. Accordingly, it is not envisioned that HP-SiC will be used in vehicular engines.

Reaction-Sintered (or Bonded) Silicon Carbides cover a wide range of compositions and manufacturing processes (ref. 38). Typical examples of these materials include; UKAEA/BNF Refel, KT, NC-430, NC-435, and Ford Siliconized Silicon Carbide. While each material is formed by a unique proprietary process, in general a plastic body is formed of SiC powder, graphite, and plasticizer. In some variants of the process SiC powder plus a char-forming plastic binder are used (ref. 39). The plastic body is pressed, extruded, injection molded or otherwise formed into a green body. Plasticizers are burned off or converted to a porous char by pyrolysis. Silicon metal as a liquid or vapor is intruded into the body and reacts with the graphite powder or char to form SiC in situ, which reaction sinters (or bonds) the component. Excess silicon is usually left to fill any voids, thus yielding a nonporous body. Such materials exhibit reasonable strengths which are retained to the melting point of silicon (~ 1400 C) or beyond, depending upon the amount of free silicon retained. A variety of experimental gas turbine components, such as combustors or stators, have been made by this materials/process route. Because these materials/process combinations lend themselves to mass production techniques coupled with good shape retention, there is a strong incentive to use them for automotive components.

Sintered Silicon Carbide is a recent innovation. The benefit of having a fully dense silicon carbide with no silicon, and not requiring expensive machining (as with HP-SiC), was recognized very early. However, pressureless sintering of SiC to full density was thought unattainable until Prochazka demonstrated that by using B and C additions, β -SiC could be sintered to near full density at ~ 2000 C. (ref. 40). Prochazka also demonstrated that sintered β -SiC could be formed into useful shapes by slip casting, die pressing and extrusion. Distribution of the carbon additive and exaggerated grain growth of α -SiC were found to interfere with densification. Coppola and McMurtry (ref. 41) developed sintered α -SiC partly in an effort to eliminate the problems associated with the β - α transformation. Sintered α -SiC has been demonstrated in several engine applications. These will be discussed in these proceedings in the paper by Phoenix and Long (ref. 42). Strength versus temperature for both α and β sintered SiC are shown in Fig. 7. Table III provides some additional properties for sintered α -SiC.

38 R.A. Alliegro, Chapter 13 in Reference 7.

39 T.J. Whalen, J.E. Noakes and L.L. Turner, Chapter 9 in Reference 9.

40 S. Prochazka, Chapter 12 in Reference 7.

41 J.A. Coppola and C.H. McMurtry, "Substitution of Ceramics for Ductile Materials in Design", presented at National Symposium on Ceramics in the Service of Man, June 1976, Carnegie Institution, Washington, D.C.

42 R. Phoenix and W. Long, this Proceedings.

Reaction-Formed SiC/Si Composites, developed by Hillig (ref. 43), are the first engineered composite ceramic/ceramic materials which offer the possibility of low cost for structural components. The process consists of starting with a graphite (or carbon) cloth, tow, felt, chopped fiber array, or any other possible precursor; forming a precursor preform by any one of a variety of routes; and infiltrating liquid Si into the graphitic precursor. The molten Si reacts with the graphite materials to form polycrystalline SiC fibers (which may or may not have graphite fiber cores, depending upon process parameters) in a Si matrix. The result is a fully dense oxidation resistant body with about 30 to 50 vol % Si reinforced respectively by 70 to 50 vol % SiC. (This high percentage of Si and the fibrous SiC morphology makes this material quite different from Refel-type materials.) While the material shows promise of ease and versatility of fabrication, it is not yet clear what the costs may be. The material offers the ability to design a composite component optimized for mechanical and thermal requirements. The material's strength is at the 50,000 to 60,000 psi level. Preliminary stress rupture data indicate that the strength of this material is independent of time below 1300 C, unlike most high-temperature ceramics (ref. 44). Perhaps the most intriguing advantage is that damaged areas have been removed and the components repaired with improved properties! The presence of free silicon would be thought to limit a use to temperatures of ~2400 F. In spite of this, the material has been used in a combustion liner application at temperatures above 2600 F (ref. 43). Although the material is still in a very early stage of development, the fact that it is an "engineered" composite material makes it a very exciting new development.

CVD - Silicon Carbide, in contrast to CVD-Si₃N₄, has been produced in bulk. Gas turbine component fabrication capability, including radial and axial rotors, has been demonstrated (ref. 45). The strength properties shown in Table III represent the lower end of the scatter band. Scatter of strength in CVD-SiC is still a major problem due to columnar grains and residual deposition stresses. A variant of conventional CVD methodology, controlled nucleation thermochemical deposition (CNTD) shows promise of both yielding an equiaxed grain structure and reducing residual stresses, thus yielding higher more uniform strength. This new material will be discussed in detail by Holz1, Stiglich and Zealer in their paper at this conference (ref. 46). CVD-SiC has demonstrated outstanding creep and oxidation resistance. The major drawback from an automotive application point of view is likely to be cost. The process may be useful in coating SiC ceramics formed by more conventional routes.

CERAMICS IN VEHICULAR ENGINE DEMONSTRATION PROGRAMS

Several materials/process combinations from each of the three principal families of heat engine ceramics discussed above have progressed to the point of successful

43 W.B. Hillig, Chapter 52 in Reference 9.

44 G.D. Quinn, unpublished research.

45 R.E. Engdahl, "Progress Toward Ceramic Turbine Rotors by CVD", Proceedings Workshop on Ceramics for Advanced Heat Engines, ERDA-CONF-77-0110.

46 R.A. Holz1, J.J. Stiglich and B.G. Zealer, this Proceedings.

component demonstration in a variety of test rigs or engines. Selected examples of such successful demonstrations from four programs sponsored by the US Departments of Defense (DOD) and Energy (DOE) are presented.

Ford/DARPA/DOE Vehicular Engine

The objective of the AMMRC-monitored Ford/DARPA Vehicular Engine Program was to demonstrate capability in brittle materials design and encourage the wider engineering use of structural ceramics. As the results of this program were encouraging and the program goals congruent with their mission, the DOE Division of Transportation Energy Conservation joined with DARPA in 1976 to jointly support this activity. The original program goals were to demonstrate ceramic hot flow path components for 200 hours of uncooled operation at temperatures up to 2500 F (1371 C) in an engine environment. These goals have been substantially met. Table VI shows the results of engine rig* tests on stationary components. All stationary components have demonstrated 200-hour durability. In the case of the stator this has been demonstrated with both a RBSN and a RB-SiC stator.

Table VI. FORD/DARPA PROGRAM -
STATIONARY COMPONENT DURABILITY DEMONSTRATION

Material - Component	Hours at 1930 F		Hours at 2500 F		Total Hours*
	Actual	Goal	Actual	Goal	
REACTION-SINTERED SiC - COMBUSTOR	175	175	26	25	201
RBSN - NOSE CONE	175	175	26	25	201
RBSN - STATORS	175	175	26	25	201
REACTION-SINTERED SiC - STATOR	175	175	29	25	204
RBSN - SHROUDS	175	175	26	25	201

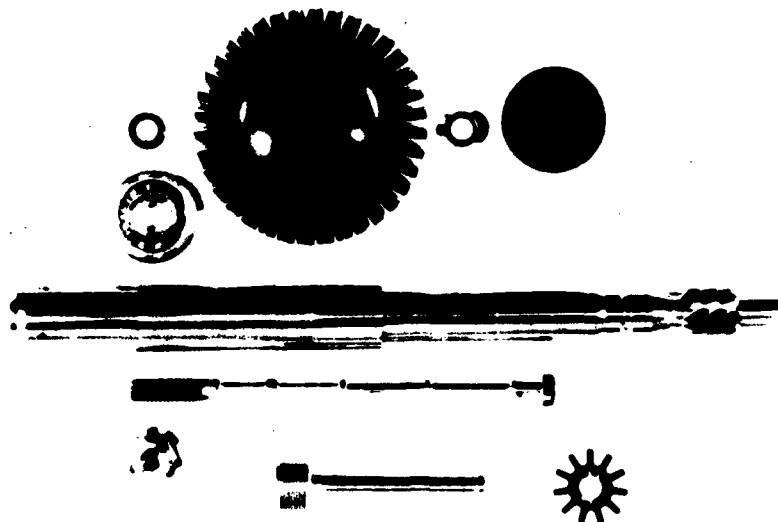
*Survived Intact

Recently a successful 200-hour, hot spin rig test of a duodensity (RBSN blade/HPSN hub) rotor was demonstrated. The conditions were 2200 F TIT, 50,000 rpm for 25 hours and speeds between 50,000 and 27,500 rpm (according to DARPA Program Duty Cycle) for the other 175 hours.

Perhaps most dramatic was the first successful engine demonstration (June 1977) of the integration of static and dynamic ceramic hardware in an operational engine environment (ref. 47). This test was the first demonstration of an aerodynamically functioning ceramic rotor. The duodensity rotor, shown with its attachment hardware in Fig. 8, ran in the engine for 36-1/2 hours at temperatures ranging from 2200 F to over 2500 F and speeds of 40,000 to 50,000 rpm. This run also included a ceramic (RBSN) nose cone, ceramic (RBSN) stator, ceramic (RBSN) shrouds, and ceramic (HPSN) spacers. This was the first demonstration of ceramics operating uncooled in an engine at temperatures in excess of those possible with uncooled superalloys.

*An engine with full air flow and temperature with rotors removed.

47 A.F. McLean and R.R. Baker, "Brittle Materials Design, High Temperature Gas Turbine", AMMRC TR 78-14.



CERAMIC TURBINE ROTOR 1195, ATTACHMENT HARDWARE AND MAIN SHAFT
 ROTOR 1195 - DESIGN D' 2.7 g/cc RBSN BLADES, HPSN HUB DUODENSITY CONFIGURATION
 TEST RESULTS:

Run	Deg F	RPM	Hours
1	2200	45,000	10
2	2250	50,000	25
	2530	50,000	1.5
TOTAL TIME			36.5

Figure 8. Ford/DARPA Program - engine rig test of rotor 1195.

Detroit Diesel Allison/DOE-404-4 Ceramic Engine Development Program

Under DOE sponsorship, Detroit Diesel Allison Division of General Motors is demonstrating the application of ceramic components in their 404/505 engines for truck and bus applications (ref. 48). They are currently operating a modified 404 engine at a 1900 F TIT. This engine is configured with AS regenerators, KT-SiC first-stage vanes, and both KT-SiC and sintered silicon nitride shrouds. The KT vanes have accumulated over 1200 hours of engine test time to date without failure. Of this 1200 hours, approximately 1000 have been at 1900 F. This is the first time that aerodynamically functioning ceramic hot flow path components have operated in an engine for over 1000 hours. The RB-SiC and SSN shrouds have been run for 560 hours and 102 hours, respectively, without incident.

The AS Regenerator Programs

Two programs to demonstrate AS regenerator technology, sponsored by US DOE, have shown impressive results. The first is a regenerator durability demonstration program at Ford Motor Co., and the second is the DDA Ceramic Engine Demonstration Program discussed above.

One objective of the Ford program is to demonstrate a B_{10} regenerator life of 10,000 hours at 800 C. AS was chosen for the demonstration because it appeared to withstand the Na and S attack to which LAS regenerators were susceptible. Engine

48 H.E. Helms and F.A. Rockwood, Presentation at the DOE, Highway Vehicle Systems, Contractor's Coordination Meeting, Dearborn, MI, October 1978.

test results for 18 thin- and thick-walled AS cores have been reported (ref. 49). To date no regenerator has failed. Four have reached 10,000 hours, five are beyond 5000 hours and testing was continuing. There was no sign of serious chemical attack to any of the regenerator cores. In contrast, the LAS regenerators tested in 1973 had average lives of 1600 hours and a B_{10} life of less than 600 hours.

The satisfactory performance of AS regenerator cores is also being confirmed in the DDA Programs (ref. 48). This program has corroborated the satisfactory performance of thin-wall AS regenerator cores in the 404 engine. Disk wear has been negligible, and only a small amount of coning (0.0018 coning per 1000 hours) has been observed. It is noteworthy that the only incident with the thin-wall AS regenerator was that it sustained some foreign object damage (FOD) when metal fragments from an upstream component failure scored the regenerator face. While damaged, the disk still performed satisfactorily and was not replaced. This incident confirms an early prediction by the author that the ceramic regenerator core could act as a final filter and protect the hot flow path of the engine from FOD (ref. 50).

The Cummins/TARADCOM Adiabatic Turbocompound Diesel

This program jointly funded by the US Army Tank and Automotive Research and Development Command and Cummins Engine Co., has demonstrated a ceramic (HPSN) piston cap in a full 250-hour engine performance test. Further details of this engine and its testing will be presented by Kamo, Woods and Geary in their presentation (ref. 51).

The net result of all the engine and rig testing described above has been to convince the engine design community that ceramics are credible candidate materials for engine application. What is now required is to demonstrate that these components can be manufactured reliably and cost effectively, so that such components can proceed from demonstration to utilization.

SUMMARY

This paper has reviewed the brittle materials design process, the three main families of engine ceramics, and results of various successful rig and engine tests of ceramic components. From this review the following major conclusions may be drawn:

1. There are several classes of heat engines that can make use of ceramics that do not necessarily require a 2500 F capacity.
2. There is no one ceramic material or process which is best for all cases. Rather there are many materials/process combinations, each of which may represent the optimum trade-off for some component.
3. The existing "first generation" of ceramic engineering materials have shown that they can work in a variety of engine environments. Further development of these materials aimed at enhanced reliability and affordability, as well as research and development of advanced "second generation" ceramic materials, will be required for future ceramic engine developments.

49 C. Rahnke, Presentation at the DOE, Highway Vehicle Systems, Contractor's Coordination Meeting, Dearborn, MI, October 1978.

50 R.N. Katz and A.F. McLean, Reference 6, pp. 409-427.

51 R. Kamo, M.E. Woods and W.C. Geary, this Proceedings.

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