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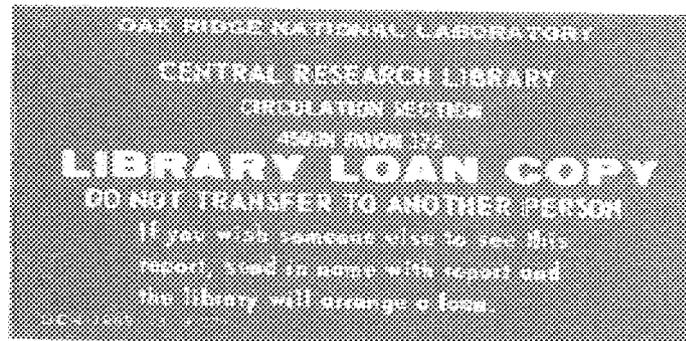


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ORNL/TM-10267

Assessment of Ceramic Composites for Multimegawatt Space Nuclear Power Systems

T. M. Besmann



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ASSESSMENT OF CERAMIC COMPOSITES FOR MULTIMEGAWATT SPACE
NUCLEAR POWER SYSTEMS

T. M. Besmann

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CONTENTS

ABSTRACT	1
INTRODUCTION	1
EQUILIBRIUM THERMODYNAMIC ANALYSIS	2
BRAYTON CYCLE, CONTINUOUS MODE	3
BRAYTON CYCLE, BURST MODE	9
RANKINE CYCLE	11
NONEQUILIBRIUM ASSESSMENT	14
CONCLUSIONS AND RECOMMENDATIONS	16
REFERENCES	18

ASSESSMENT OF CERAMIC COMPOSITES FOR MULTIMEGAWATT SPACE
NUCLEAR POWER SYSTEMS*

T. M. Besmann

ABSTRACT

A calculational thermodynamic equilibrium analysis of the compatibility of ceramic-ceramic composites and a nonequilibrium assessment based on the literature of their use in proposed multimegawatt space nuclear power systems was performed. The five candidate composites included Al_2O_3 -reinforced Al_2O_3 , SiC-whisker-toughened Al_2O_3 , SiC-whisker-toughened Si_3N_4 , SiC-fiber-reinforced ZrC, and carbon-fiber-reinforced ZrC. The reactor concepts included Brayton cycle (continuous and burst mode) and Rankine cycle. It was determined that Al_2O_3 -reinforced Al_2O_3 and carbon-fiber-reinforced ZrC are compatible in the Brayton-cycle continuous-mode system and that Al_2O_3 -reinforced Al_2O_3 , SiC-fiber-reinforced ZrC, and carbon-fiber-reinforced ZrC are compatible in the Rankine-cycle system. None of the candidate ceramic composite systems was likely to be sufficiently stable under Brayton-cycle burst-mode conditions.

INTRODUCTION

Proposed multimegawatt (MMW) space nuclear power systems are designed to operate at high temperatures that are unprecedented and, in some designs, with potentially corrosive working fluids. These temperatures, levels of stress, and hostile environments may be outside the capability of refractory alloys, so designers are interested in the use of ceramic composites as structural materials.

Two MMW space reactor power cycles are under consideration, a Brayton and a Rankine cycle. The Brayton cycle is envisioned as using inert gas as the working fluid during continuous operation with a peak temperature of 1500 K, and hydrogen as the working fluid for short-duration (30-min) high-power operation at a temperature of 2500 K. The Rankine-cycle system uses potassium liquid and vapor as the working fluid with a peak temperature of 1450 K.

*Research sponsored by the Division of Defense Energy Projects, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

The object of this assessment is to determine the applicability of several candidate ceramic composites to the proposed MMW space power systems. Five ceramic composites have been identified as potentially possessing the necessary high-temperature stability and mechanical properties. These include two previously developed materials - SiC-whisker-reinforced Al_2O_3 and SiC-whisker-reinforced Si_3N_4 - and three speculative materials - carbon-fiber-reinforced ZrC, SiC-fiber-reinforced ZrC, and Al_2O_3 -reinforced Al_2O_3 .

Two approaches were taken in assessing the use of the ceramic composites in the proposed power cycles: an equilibrium thermodynamic analysis and a nonequilibrium assessment based on a literature review. In the equilibrium thermodynamic analysis, gas pressures were calculated and the stable liquid and solid phases containing the constituent elements for each ceramic composite-power cycle combination were determined over the appropriate temperature range. For the nonequilibrium assessment, reported results of high-temperature compatibility studies between the ceramic phases and the proposed working fluids were reviewed for indications of detrimental interactions.

EQUILIBRIUM THERMODYNAMIC ANALYSIS

The equilibrium thermodynamic assessment was performed by calculating the equilibrium state for each set of constituents under the appropriate pressure, temperature, and compositional conditions. The computer program SOLGASMIX-PV¹ was used to execute the calculations to determine equilibrium pressures of the gaseous species and the amounts of each of the stable solid and liquid phases. Equilibrium states were computed for sets of conditions for each of the power cycles (Table 1). The species used and

Table 1. Power cycle conditions

Conditions	Cycles		
	Brayton, continuous	Brayton, burst	Rankine
Temperature, K	ΔT , 1100-1500	2500 (once through)	ΔT , 1340-1450
Pressure, MPa	Inert gas, 2.76	Hydrogen, 6.89	Potassium, 1.38

attendant thermodynamic data are listed in Tables 2 and 3. Two separate data sets were used for convenience and to avoid exceeding the capacity of the SOLGASMIX-PV program.

Thermodynamic values were obtained from available data compilations, as indicated in Tables 2 and 3. The values of enthalpy of formation (ΔH_f°) and entropy (S°) at 1800 K were used to determine the Gibbs free energy of formation over the entire temperature range of interest. Although this is an approximation, little error is incurred in the free energy values over a range of several hundred degrees. For some species elevated-temperature values were not available, so 298 K values were used.

For the zirconium silicides, the only values found were 298 K ΔH_f° . Entropy values at 298 K for the zirconium silicides thus had to be estimated, and this was done using the averaged S° values for other transition metal silicides of the same composition.⁶ For phases where data for other metals were not available, (M_6Si_5 and $MSi_{1.62}$) values were determined from a curve of 298 K S° per gram-atom versus metal/silicon ratio that was constructed from the average values for transition metal silicides that do exist (Fig. 1). Liquid phases were assumed to be adequately modeled with a single, ideal solution.

The results of the thermodynamic calculations are shown as plots of gas and vapor pressures versus temperature for each of the power cycle-ceramic composite systems. Due to the large number of species for which pressures were calculated, each is not shown explicitly on the plots. Rather, the sums of the partial pressures of species having the same elemental constituents are shown. Also indicated on these plots are the stable liquid and solid phases in the equilibrium system at the temperatures shown.

BRAYTON CYCLE, CONTINUOUS MODE

Alumina is thermodynamically stable to 1800 K based on the pressures indicated in Fig. 2, which show no species pressures in excess of 10^{-5} Pa.

Table 2. Thermodynamic data at 1800 K for the H-K-Al-Si-C-O system

Species	ΔH_f° (kJ/mol)	S° (J·mol ⁻¹ ·K ⁻¹)	Ref.	Species	ΔH_f° (kJ/mol)	S° (J·mol ⁻¹ ·K ⁻¹)	Ref.
Gaseous				Gaseous (continued)			
H ₂	0.0	184.7	2	O	254.9	198.9	4
O ₂	0.0	264.7	2	SiO	-162.8	272.7	2
Al	304.7	202.1	2	SiO ₂	-361.1	326.8	2
Al ₂	307.9	436.3	2	O ₃	145.4	330.3	2
AlH	234.2	247.5	2	Si	393.2	206.2	2
AlOH	-212.3	297.0	2	SiH ₄	-25.7	335.0	3
AlO ₂ H	-486.0	373.4	2	Si ₂	594.1 ^a	229.8 ^a	6
AlO	43.1	283.1	3	Si ₂ H ₆	79.9 ^a	274.3 ^a	6
AlO ₂	-206.8	348.9	3	Si ₃	615.1 ^a	253.7 ^a	6
Al ₂ O	-179.2	362.4	3	Liquid			
Al ₂ O ₂	-481.0	414.1	3	K	-69.7	128.3	2
AlC	660.1	287.9	2	Al	0.0	92.3	2
CH	590.5	240.0	2	Si	0.0	93.4	2
CH ₂	378.0	270.2	4	Al ₂ O ₃	-1601.5	290.2	3
CH ₃	133.7	290.0	2	K ₂ CO ₃	-1197.8	478.1	2
CH ₄	-92.7	295.9	2	K ₂ SiO ₃	-1650.5	475.9	5
CO	-117.3	254.8	2	SiO ₂	-939.5	163.8	2
C	717.4	195.4	2	Crystalline phases			
CO ₂	-397.4	302.9	2	Si	-49.9	63.7	2
SiC	661.7	288.3	2	C	0.0	38.2	2
Si ₂ C	413.0	342.2	2	α-Al ₂ O ₃	-1683.4	256.7	5
C ₂	832.5	266.6	2	β-SiC	-122.9	94.2	2
C ₂ H	468.6	288.2	2	Al ₂ SiO ₅	-2634.5	416.1	2
C ₂ H ₂	220.8	312.7	2	Al ₆ O ₁₃ Si	-6925.1	1116.7	2
C ₂ H ₄	35.3	362.4	2	SiO ₂	-946.8	160.2	2
C ₂ H ₄ O	-66.6	416.1	2	K ₂ CO ₃	-1209.5	464.5	2
C ₂ O	284.4	328.9	2	AlH ₃	-11.5 ^a	29.3 ^a	6
SiC ₂	550.0	334.7	2	Al ₄ C ₃	-265.7	380.6	2
C ₃	795.6	313.3	2	K ₂ O	-456.0	287.0	2
C ₃ O ₂	-93.9	436.6	2	K ₂ O ₂	-564.1	357.2	2
C ₄	960.9	353.7	2	K ₂ SiO ₃	-1693.9	440.0	5
H	226.1	152.0	2	Al ₂ O ₃ ·H ₂ O	-2004.2 ^a	28.6 ^a	6
HO	37.0	238.6	2	Al ₂ O ₃ ·3H ₂ O	-2586.6 ^a	140.2 ^a	6
HO ₂	-2.1	308.3	2	Al ₄ C ₃	-215.7 ^a	88.7 ^a	6
SiH	315.0	256.6	2	K ₂ Si ₂ O ₅	-299.2 ^a	182.0 ^a	6
H ₂ K ₂ O ₂	-830.2	512.7	2	KH	-57.7 ^a	51.5 ^a	6
H ₂ O	-251.1	259.3	2				
H ₂ O ₂	-141.1	324.4	2				
K	0.0	197.6	2				
KO	-17.9	305.7	2				
K ₂	-54.3	319.9	2				

^aValues at 298 K.

Table 3. Thermodynamic data at 1800 K for the H-K-Zr-Si-C-N system

Species	ΔH_f° (kJ/mol)	S° (J·mol ⁻¹ ·K ⁻¹)	Ref.	Species	ΔH_f° (kJ/mol)	S° (J·mol ⁻¹ ·K ⁻¹)	Ref.
Gaseous				Gaseous (continued)			
H ₂	0.0	184.7	2	N ₃	423.3	320.0	5
K	0.0	197.6	2	Si	393.2	206.2	2
CH ₂	378.0	270.2	4	Zr	1310.1	233.5	2
CKN	-9.9	353.3	2	CH	590.5	240.0	2
CN	430.2	260.2	2	CH ₃	133.7	290.0	2
NCN	477.4	321.9	5	CH ₄	-92.7	295.9	2
C	717.4	195.4	2	Si ₂	594.0 ^a	229.8 ^a	6
SiC	661.7	288.3	2	Si ₂ H ₆	79.9 ^a	274.3 ^a	6
Si ₂ C	413.0	342.2	2	Si ₃	615.0 ^a	253.7 ^a	6
C ₂	832.5	266.6	2	Liquid			
C ₂ H	468.4	292.4	2	Si	0.0	93.4	2
C ₂ H ₂	220.8	312.7	2	Zr	18.4	101.8	2
C ₂ H ₄	35.3	362.4	2	K	-69.7	128.3	2
C ₂ K ₂ N ₂	-174.6	588.7	2	CKN	-166.4	269.7	2
C ₂ N	558.0	330.9	2	Crystalline phases			
C ₂ N ₂	311.6	369.2	2	β-SiC	-122.9	94.2	2
SiC ₂	550.0	334.7	2	ZrC	-199.8	123.1	2
C ₃	795.6	313.3	2	C	0.0	38.2	2
C ₄	961.1	353.7	2	Si ₃ N ₄	-878.5	367.6	2
C ₄ N ₂	538.5	489.0	2	Si	-49.9	63.7	2
C ₅	969.4	401.0	2	Zr	0.0	93.2	2
H	226.1	152.0	2	CKN	-188.8	247.3	2
HN	377.2	236.4	5	KH	-57.7 ^a	-51.5 ^a	6
HSi	315.0	256.6	5	ZrN	-368.2 ^a	-38.9 ^a	6
HZr	494.8	277.1	2	ZrH ₂	-169.5 ^a	-35.1 ^a	6
N ₂ H ₂	208.5	316.8	2	Zr ₂ Si	-338.9 ^b	-105.4 ^b	2
N ₂ H	183.9	266.6	2	Zr ₅ Si ₃	-613.8 ^b	-225.9 ^b	2
NH ₃	-55.4	283.9	2	Zr ₆ Si ₅	-850.6 ^b	-268.8 ^b	2
N ₂ H ₄	94.9	390.4	2	ZrSi	-147.7 ^b	-46.9 ^b	2
SiH ₄	-25.7	335.0	2	ZrSi _{1.62}	-149.8 ^b	58.4 ^b	2
K ₂	-54.3	319.9	3				
N	479.4	190.6	2				
SiN	312.9	279.3	2				
Si ₂ N	283.5	360.4	2				
N ₂	0.0	248.2	2				

^aValues are at 298 K.

^bEnthalpy values are at 298 K from Kubaschewski and Alcock,⁶ and entropy values are estimated for 298 K as described in the text.

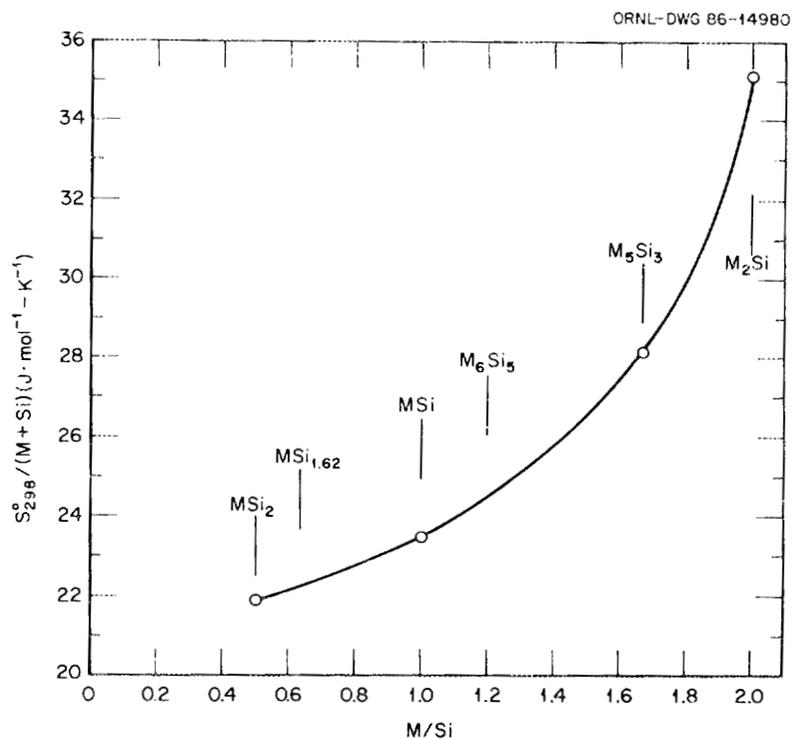


Fig. 1. Entropy per gram-atom at 298 K of transition metal (M) silicides versus M/Si ratio. *Source of entropy data:* O. Kubaschewski and C. B. Alcock, *Metallurgical Thermochemistry*, 5th ed., Pergamon Press, Ltd., Oxford, U.K., 1979, pp. 268-323.

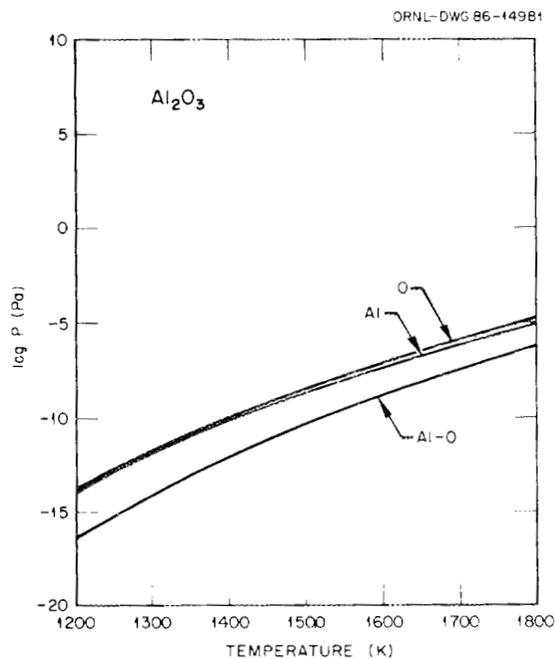


Fig. 2. Equilibrium partial pressures over Al_2O_3 in 2.76 MPa of inert gas.

SiC-whisker-toughened Al_2O_3 , however, is significantly less stable, with C-O, Si-O, and aluminum species exhibiting pressures well above 100 Pa at 1800 K (Fig. 3).

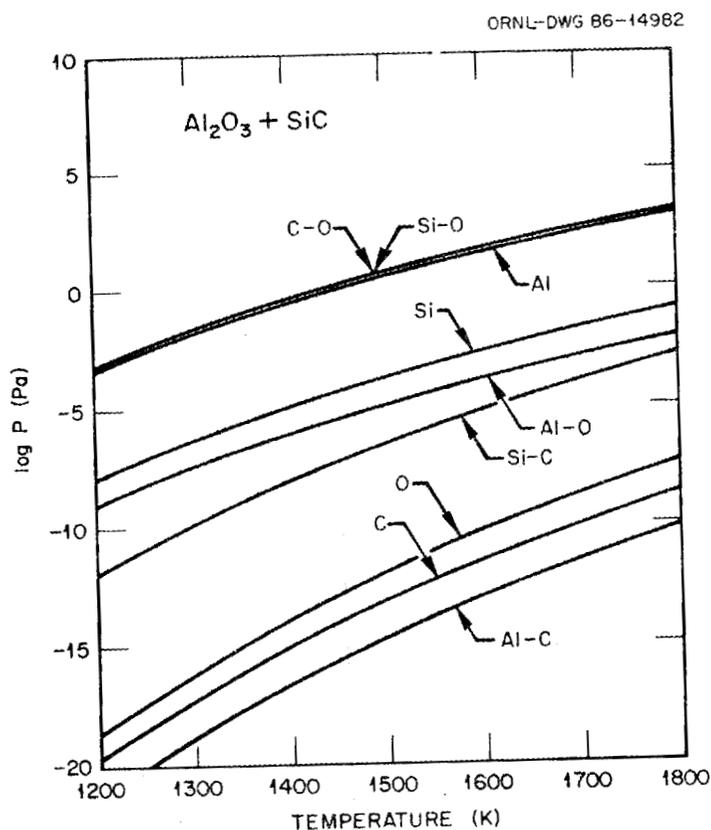


Fig. 3. Equilibrium partial pressures over Al_2O_3 and SiC in 2.76 MPa of inert gas.

Carbon-fiber-reinforced ZrC has extremely low vapor pressures to 1800 K with only carbon species exhibiting any significant pressure ($<10^{-7}$ Pa), as shown in Fig. 4. Silicon carbide-fiber-reinforced ZrC, however, is calculated to have moderate silicon species pressures equalling 10^{-2} Pa at 1800 K (Fig. 5).

Silicon carbide-whisker-toughened Si_3N_4 suffers from high nitrogen species pressures over the temperature range, with a value of almost 1 kPa at 1800 K (Fig. 6). Silicon-containing species also have substantial pressures in this system.

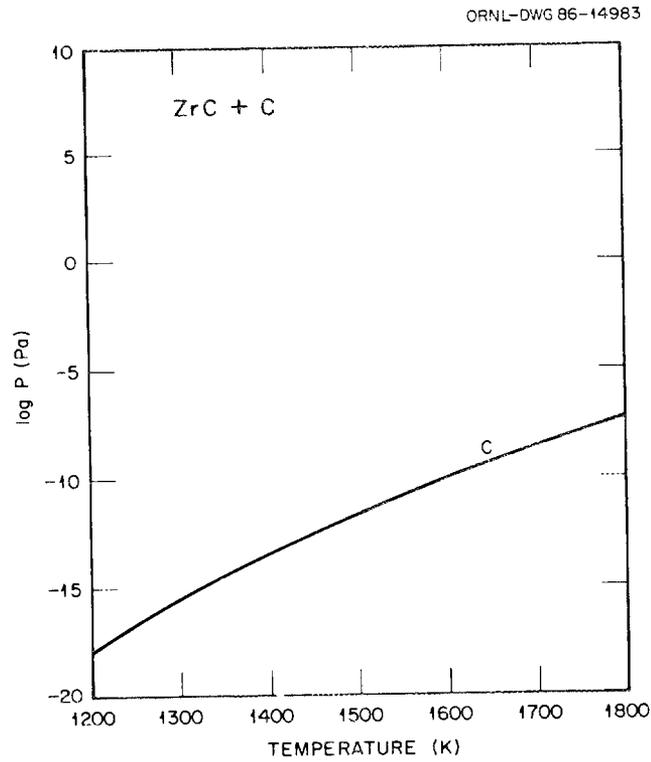


Fig. 4. Equilibrium partial pressures over ZrC and C in 2.76 MPa of inert gas.

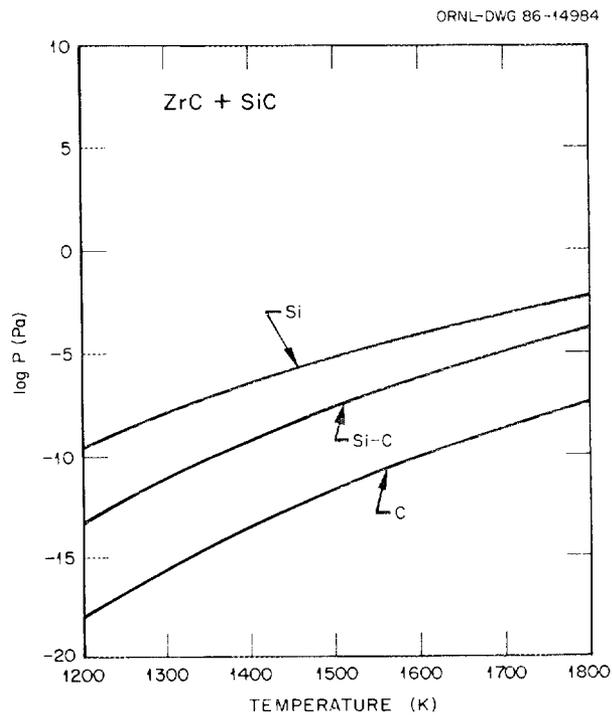


Fig. 5. Equilibrium partial pressures over ZrC and SiC in 2.76 MPa of inert gas.

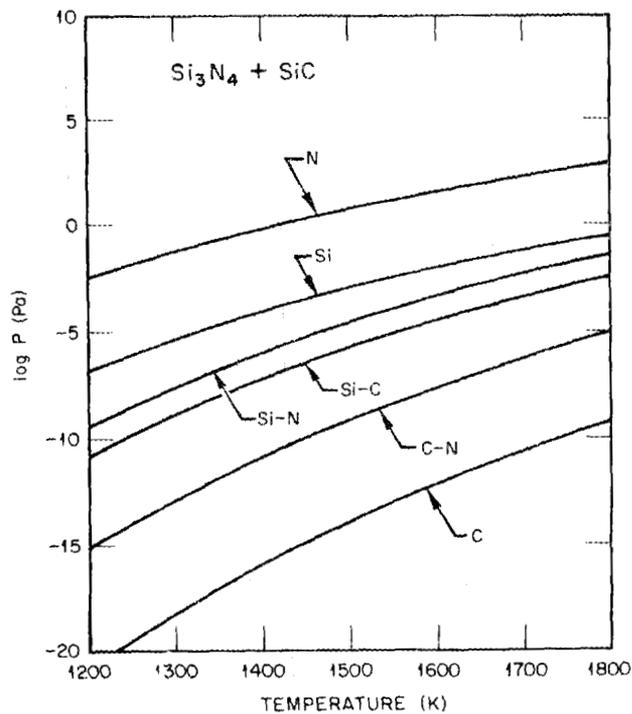


Fig. 6. Equilibrium partial pressures over Si_3N_4 and SiC in 2.76 MPa of inert gas.

BRAYTON CYCLE, BURST MODE

The alumina-based composites are unstable in hydrogen at the high operating temperatures, forming a liquid phase and generating H-O and aluminum species pressures near 100 kPa at 2500 K (Fig. 7). Silicon carbide-whisker-toughened Al_2O_3 also exhibits undesirable equilibrium properties with species pressures in excess of 1 MPa and the formation of a liquid phase above 1500 K (Fig. 8).

Carbon-fiber-reinforced ZrC reacts with hydrogen to generate high pressures of hydrocarbons, of the order of 1 MPa, over the entire temperature range of the calculations (1300-2500 K). Less than 1 Pa of other species such as carbon and Zr-H are also generated (Fig. 9).

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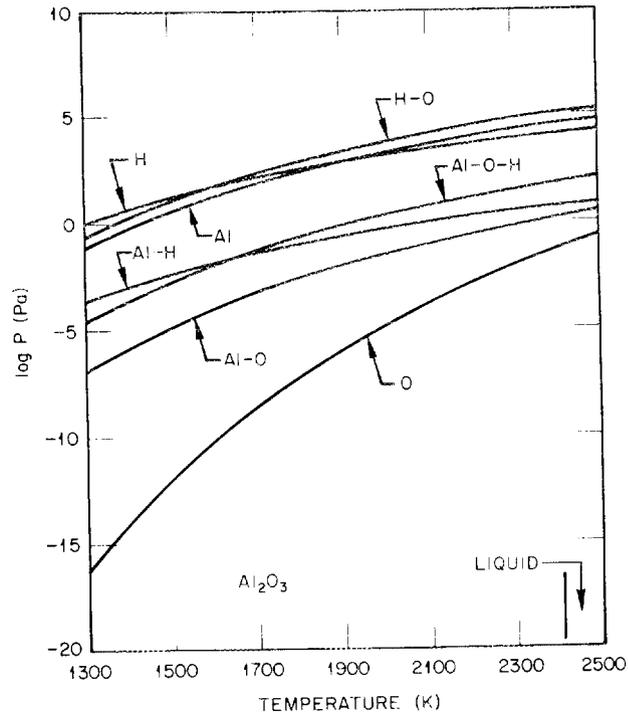


Fig. 7. Equilibrium partial pressures over Al_2O_3 in 6.89 MPa of hydrogen.

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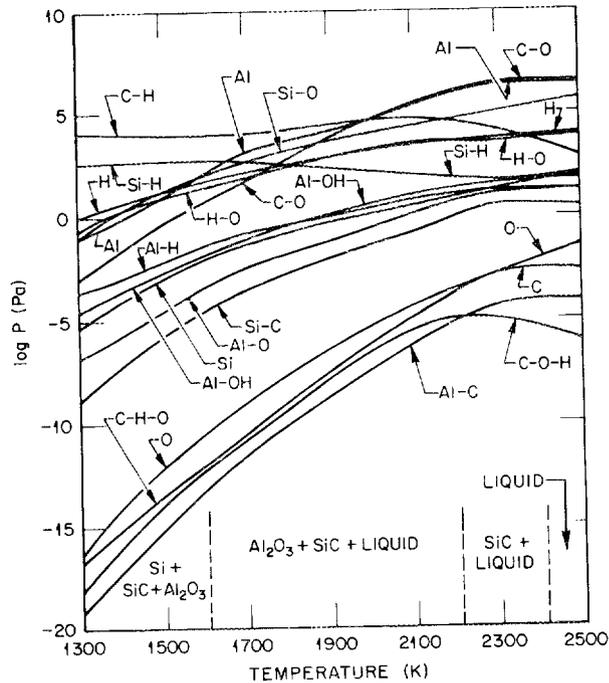


Fig. 8. Equilibrium partial pressures over Al_2O_3 and SiC in 6.89 MPa of hydrogen. The liquid phase is Al and Si containing dissolved Al_2O_3 and SiC .

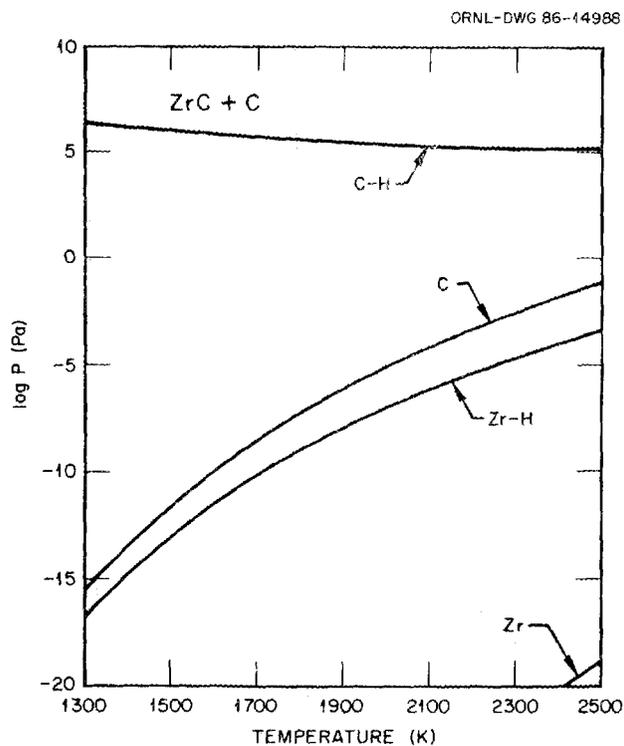


Fig. 9. Equilibrium partial pressures over ZrC and C in 6.89 MPa of hydrogen.

Silicon carbon-fiber-reinforced ZrC not only also exhibited substantial hydrocarbon pressures under these conditions, but indicated the formation of a liquid phase above 1500 K as well (Fig. 10).

The SiC-whisker-toughened Si_3N_4 material generates significant nitrogen pressures at these high temperatures, more than 1 MPa at 2500 K. In addition, a number of species such as the hydrocarbons, H-N, and Si-H also maintain significant pressures down to lower temperatures (1300 K) (Fig. 11).

RANKINE CYCLE

The alumina ceramic composite is calculated to be relatively stable in the presence of potassium with K-O species pressures less than 10^{-3} Pa at 1550 K (Fig. 12). Silicon carbide-whisker-toughened Al_2O_3 , however, has significant aluminum, C-O, and Si-O pressures under these conditions (Fig. 13).

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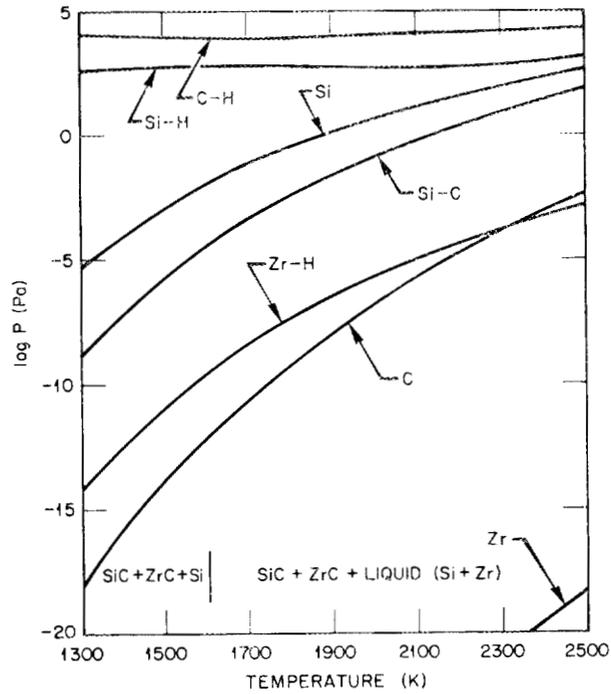


Fig. 10. Equilibrium partial pressures over ZrC and SiC in 6.89 MPa of hydrogen.

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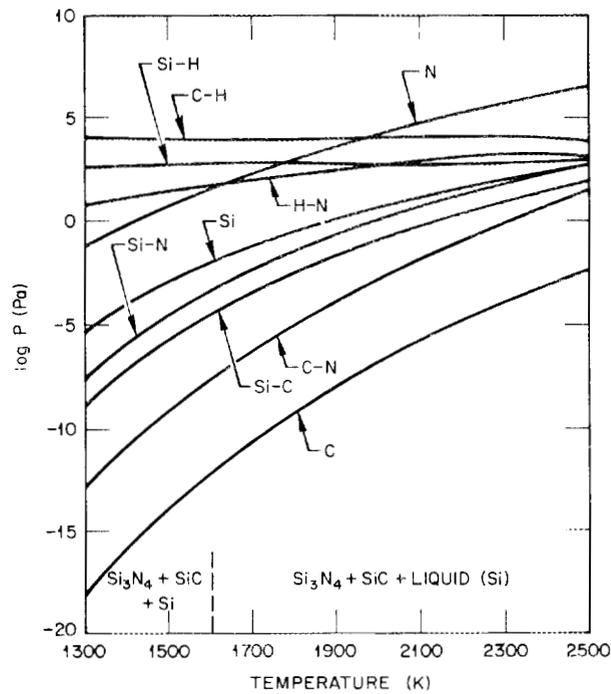


Fig. 11. Equilibrium partial pressures over Si₃N₄ and SiC in 6.89 MPa of hydrogen.

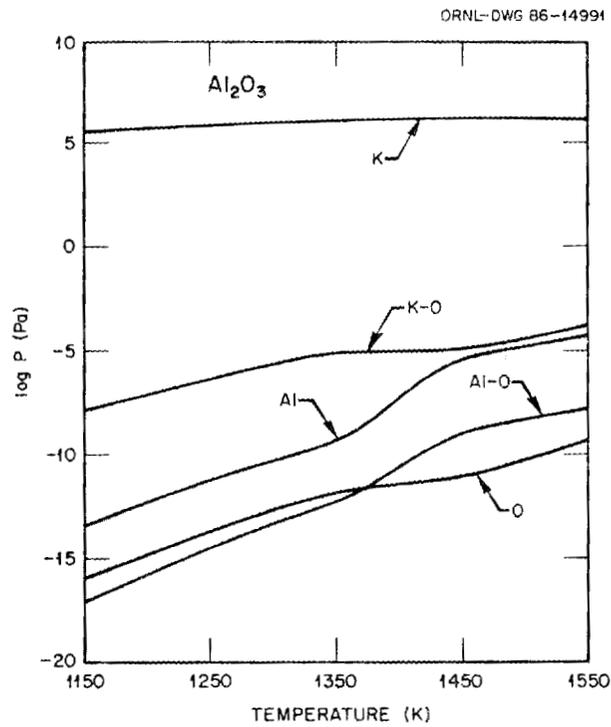


Fig. 12. Equilibrium partial pressures over Al_2O_3 in 1.38 MPa of potassium vapor and liquid.

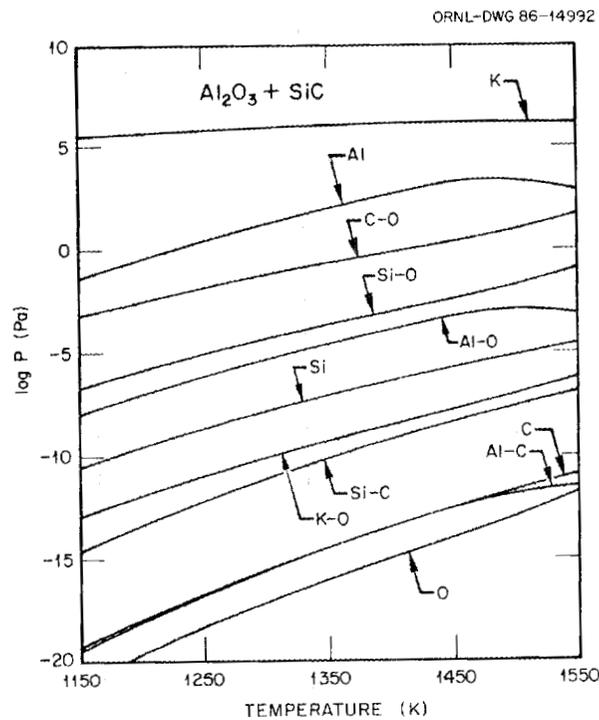


Fig. 13. Equilibrium partial pressures over Al_2O_3 and SiC in 1.38 MPa of potassium vapor and liquid.

Carbon-fiber-reinforced ZrC is thermodynamically stable in potassium under these conditions with only a minor carbon species pressure calculated (Fig. 14). Silicon carbide-fiber-reinforced ZrC also appears to be relatively stable, with the largest species pressure being that of silicon at less than 10^{-4} Pa at 1550 K (Fig. 15).

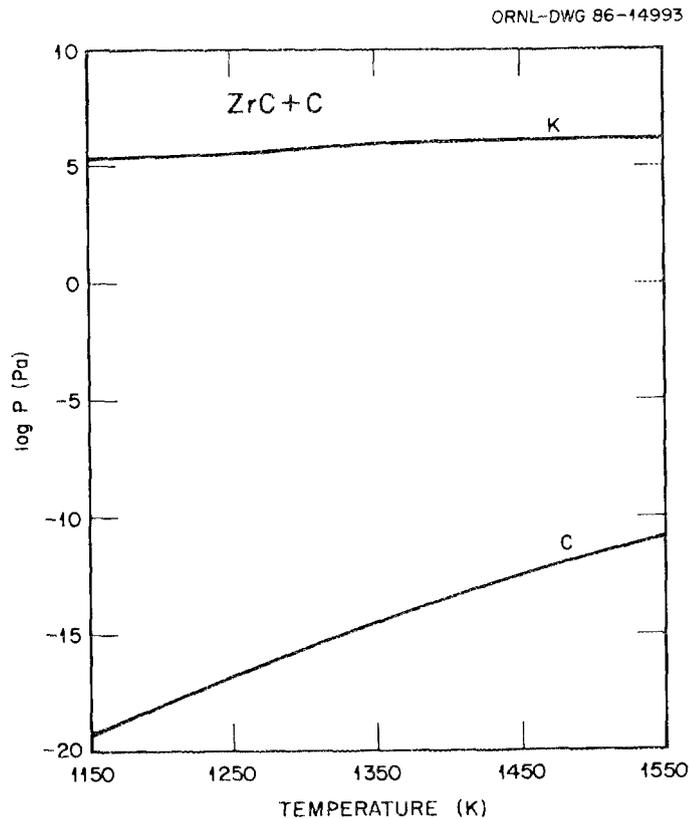


Fig. 14. Equilibrium partial pressures over ZrC and C in 1.38 MPa of potassium vapor and liquid.

The SiC-whisker-toughened Si_3N_4 shows less stability than do the other systems, with nitrogen having an equilibrium pressure greater than 10 Pa (Fig. 16).

NONEQUILIBRIUM ASSESSMENT

The nonequilibrium assessment of the candidate ceramic composites is based on a review of the literature with regard to observed interactions between the composite components and the environment of the proposed power

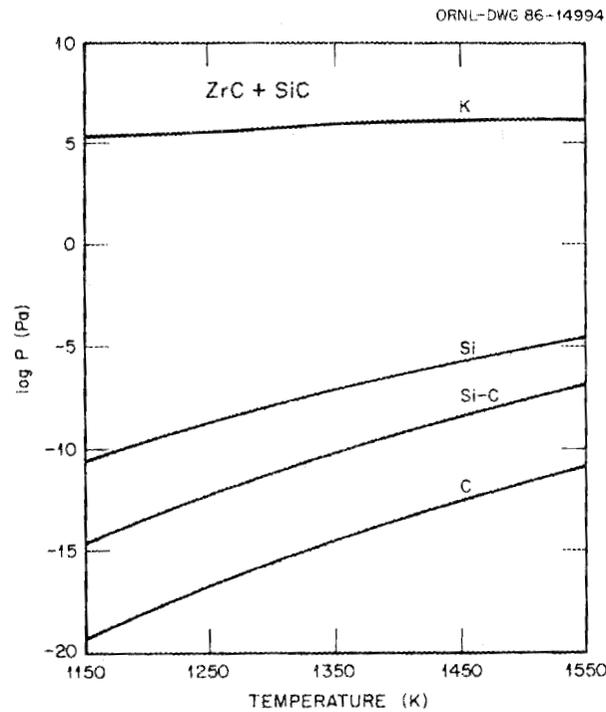


Fig. 15. Equilibrium partial pressures over ZrC and SiC in 1.38 MPa of potassium vapor and liquid.

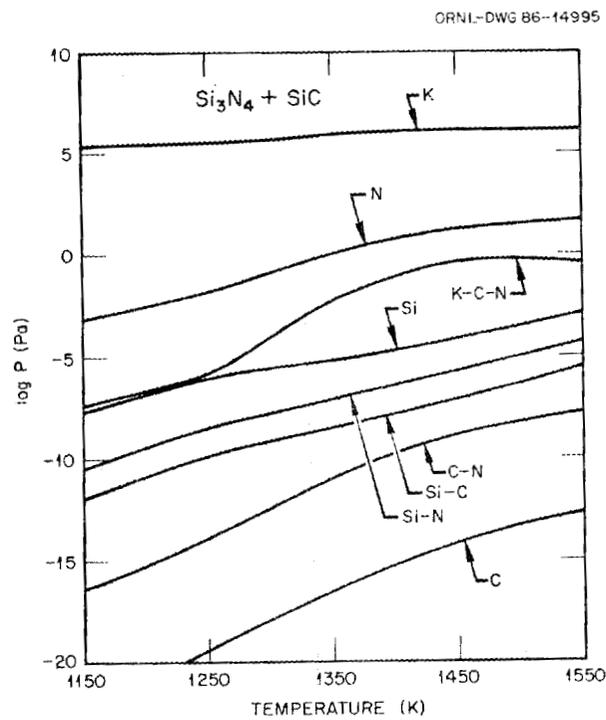


Fig. 16. Equilibrium partial pressures over Si₃N₄ and SiC in 1.38 MPa of potassium vapor and liquid.

cycles. Unfortunately, there have been few studies of these interactions, and those that have been reported are typically for temperatures well below those of the system designs. McKee and Chatterji⁷ studied the corrosion of SiC in N₂ and H₂ at 1173 K and found no material loss or reaction. Horn et al.⁸ observed significant erosion of SiC beginning at 1400 K in steam-hydrogen mixtures, but they attributed the reaction to the effect of the steam. Alumina was also evaluated in steam-hydrogen, but it displayed little erosion to 1550 K, the maximum temperature used in the study. Sheehan⁹ examined the effect of helium containing H₂, H₂O, CO, and CH₄ impurities on alumina and Si₃N₄ to 1200 K and found the alumina to be unaffected and the Si₃N₄ to undergo passive oxidation. Strength tests following 5000-h exposures showed no strength degradation.

Cook¹⁰ did a scoping study of the corrosion resistance of various ceramics and cermets to liquid metals. Although potassium was not included as a corrodent, sodium was, and it is expected that the response of the ceramics to the two alkali metals will be similar. It was found that ZrC exposed to sodium for 100 h at 1089 K showed zero depth of attack, zero percentage weight change, and zero dimensional change. Alumina, SiC, and Si₃N₄ were described as having fair to good resistance to corrosion, which was defined as less than 25- μ m depth of attack, less than 2% weight change, and less than 1% dimensional change. Blanc et al.¹¹ studied the inclusion of potassium in alumina at 2073 K and determined that there was little solubility for the alkali metal (5-10 ppm) and therefore little effect of exposure to potassium.

CONCLUSIONS AND RECOMMENDATIONS

The compatibility of the ceramic composites determined from the equilibrium calculations may be conservative since equilibrium may never be reached because of mass transport or chemical kinetic constraints. The conclusions of the equilibrium assessment, based on calculated partial pressures, are summarized in Fig. 17. To illustrate the effect of significant species pressures on mass transport in a system, consider the

example of the calculated 10^{-3} Pa partial pressure of silicon over SiC-whisker-toughened Si_3N_4 in inert gas at 1500 K. Assuming the current design criteria of 241 kg/s gas flow (72% He, 28% Xe) at 2.76 MPa, and equilibrium partial pressures maintained in the hot (1500 K) and cold (1100 K) regions of the system, 16.7 kg/year of silicon will be vaporized from the hotter region and condensed in the lower-temperature regions. It is likely that this rate of mass transport would be unacceptable.

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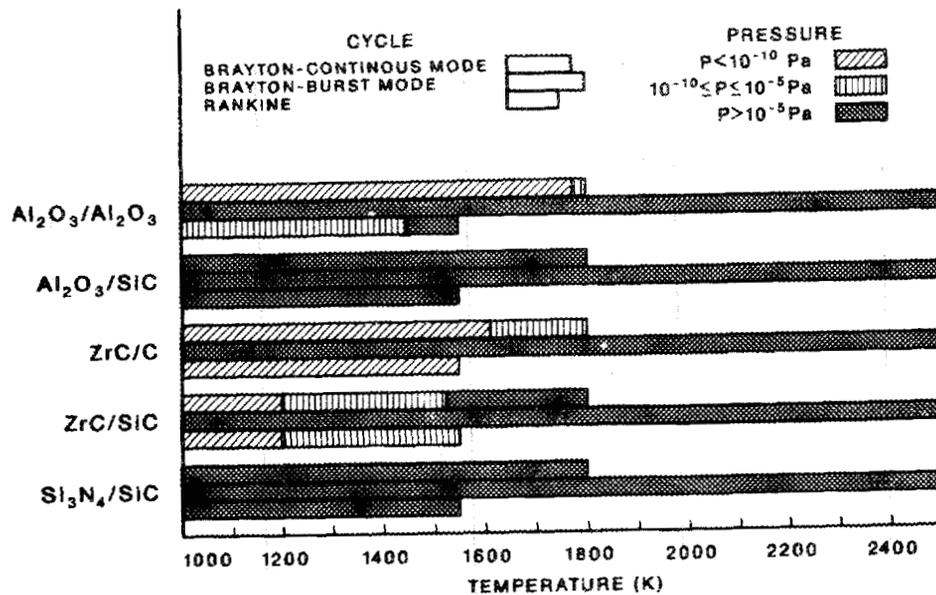


Fig. 17. Summary of the conclusions of the thermodynamic equilibrium assessment. The stabilities of the ceramic-ceramic composites are related to the maximum species pressures, which are shown in the figure. Equilibrium pressures less than 10^{-10} Pa indicate that the system is stable; pressures between 10^{-10} and 10^{-5} Pa indicate less, but possibly acceptable, stability; and pressures greater than 10^{-5} Pa indicate unstable systems.

Using the criteria for the Brayton-cycle continuous-mode power system, the composites containing Al_2O_3 , ZrC, or carbon appear to be thermodynamically stable. The SiC-containing systems, however, are likely to have unacceptably high silicon partial pressures.

The combination of hydrogen and the extreme temperature of the Brayton-cycle burst-mode system eliminates all of the ceramic composites considered. Should the system need to operate for a very short period of time it may be possible to tolerate material vaporization, although such material loss would have to be accommodated in the design. Experimental observations at lower temperatures (1173-1550 K) of interactions with hydrogen, however, indicate little degradation of alumina and Si_3N_4 and little likely degradation of SiC.

For the Rankine-cycle systems the Al_2O_3 composite, SiC-fiber-reinforced ZrC, and carbon-fiber-reinforced ZrC appear to have acceptable thermodynamic equilibrium stabilities. These systems also appear to have fair to good compatibility based on experimental determinations.

Based on the above considerations, it is recommended that Al_2O_3 -reinforced Al_2O_3 and carbon-fiber-reinforced ZrC be considered for the Brayton-cycle continuous-mode system and that Al_2O_3 -reinforced Al_2O_3 , SiC-fiber-reinforced ZrC, and carbon-fiber-reinforced ZrC be considered for the Rankine-cycle system. None of the candidate ceramic composite systems are likely to be sufficiently stable under Brayton-cycle burst-mode conditions.

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