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WEST MIFFLIN, PENNSYLVANIA
MATERIALS TECHNOLOGY
SPACE PLANT MATERIALS ENGINEERING



Title: Compatibility of Space Nuclear Power Plant Materials in an Inert He/Xe Working Gas Containing Reactive Impurities (U)

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Summary: A major materials selection and qualification issue identified in the Space Materials Plan is the potential for creating materials compatibility problems by combining dissimilar reactor core, Brayton Unit and other power conversion plant materials in a recirculating, inert He/Xe gas loop containing reactive impurity gases. Reported here are results of equilibrium thermochemical analyses that address the compatibility of space nuclear power plant (SNPP) materials in high temperature impure He gas environments. These studies provide early information regarding the constraints that exist for SNPP materials selection and provide guidance for establishing test objectives and environments for SNPP materials qualification testing.

Observations
and
Conclusions:

1. There is consensus in the high temperature gas reactor (HTGR) literature that thermodynamic equilibrium exists neither among the gaseous impurities in the He coolant nor between the impurities and metal alloys in contact with the coolant. However, concepts and considerations of equilibrium thermochemistry are important as they provide a fundamental framework upon which to construct an understanding of the thermodynamic potential for thermochemical reactions. Reaction rate kinetics may be understood in terms of deviations from equilibrium.

2. Equilibrium thermochemical analyses can be used to determine the thermodynamic potential for individual and competitive reactions to occur. Equilibrium thermochemistry is used in this study to synthesize the gas-metal, gas-ceramic and gas-gas reactions that are judged most likely to be rate limiting. These calculations identify the oxidation and carburization reactions that *may and may not occur* but cannot identify the reactions that necessarily *will occur*. Comparison of analysis results to data is necessary to discover the predominant reactants where reactions are kinetically competitive.

3. Whether or not an oxide or carbide forms in a given environment depends on the relative concentrations and reactivities of oxidizing and carburizing constituents in the environment. The principal reactive gases in an HTGR are water vapor (causes oxidation and decarburization), carbon monoxide (causes oxidation and carburization), methane (causes carburization) and hydrogen (causes oxide reduction). Also present are low concentrations of carbon dioxide (a product of carbon monoxide oxidation), oxygen and nitrogen (an essentially non-reactive gas due to the relatively slow kinetics of reaction):

4. The SNPP environment that will develop is not known. The concentrations of residual gases, which cannot be entirely eliminated, will depend on the initial purity of the charging gas, the effectiveness of the system de-gas procedure, the sorption and desorption rates, reactivities, carbon content and relative surface areas of the structural materials that are exposed to the closed environment and unknown concentrations of organic and inorganic impurities from various plant components. An effort is currently underway to gather the SNPP system information, solubilities and kinetic data needed to predict the oxidation and carburization potentials of the SNPP

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environment.

5. In order to proceed without *a priori* knowledge of the SNPP environment, we assume that adsorbed water is the primary reactive impurity in the system at reactor start up and is the source of water vapor and hydrogen. We further assume that carbon present in SiC and the superalloys, which can react with water vapor and hydrogen, is the only source of carbonaceous reactive impurities in the He/Xe working gas and that carbon is present in excess of equilibrium amounts. These assumptions are found to be reasonable when applied to an analysis of the Graphite Core-HTGR (GC-HTGR) for which the typical concentrations of impurity gases are known. The analysis proceeds by calculating the concentrations of reaction products as a function of one independent variable, the assumed water burden present on reactor start up.

6. Use of these analysis assumptions and methods to evaluate candidate SNPP materials shows that alloys of Nb are inherently incompatible with the heat resistant superalloys. Thermochemical analyses show that Nb is both oxidized and carburized when exposed to a 1400 K environment that was established by reaction of 1150 K water vapor with chromia former superalloys having chromium carbides. Alloys of Nb and Ta require environments having very low oxidation and carburization potentials to avoid absorption of embrittling concentrations of oxygen and carbon. Use of Nb and Ta alloys in an oxidizing and carburizing environment will require development of oxidation and carburization resistant coatings.

7. The chromia former superalloys will be decarburized in an environment dominated by Nb alloys due to the inability of the superalloys to form protective chromia surface oxides in this environment. The same result is expected for Ta alloys as Ta has reactivity for oxygen and carbon similar to that of Nb. The candidate Brayton Unit iron-nickel-, cobalt- and nickel-base superalloys require environments having oxidation potentials high enough to form the Cr and Al surface oxides that protect these alloys from embrittlement due to internal oxidation and carburization and loss of creep strength due to excessive decarburization and dealloying by evaporation of low vapor pressure metals like chromium. Due to the much higher oxygen and carbon reactivities of Nb and Ta, protective Cr oxides will be reduced and carbon transferred from the superalloys to Nb and Ta alloys.

8. Analysis of the refractory metal Mo, alloys of which are being considered for core applications, shows that Mo is compatible with Brayton Unit superalloys. Molybdenum alloys are less susceptible to environmental embrittlement by oxygen due to very low solubility for oxygen. Moreover, at oxidation potentials where protective Cr_2O_3 oxides form on superalloys, Mo alloys form neither a surface oxide nor internal carbides.

9. Analysis of SiC, which is a candidate SNPP core material, shows that SiC is thermochemically compatible with the superalloys. SiC forms a protective surface oxide at oxidation potentials required to form protective Cr_2O_3 oxide. However, the analyses reported here show that a rather large water burden, approaching that of a GC-HTGR, is required to stabilize Cr_2O_3 oxide in the presence of SiC. Success of the SiC option will depend on an ability to establish and maintain the oxidation potential of the SNPP at or above this level. A potential disadvantage of SiC is the possibility that free carbon "dust" may form leading to thinning of SiC components and circulation of carbon throughout the system. An issue for use of SiC that is not addressed here is the greater potential for release of fission products and the potential negative impact that these may have when transported around the SNPP system.

10. An advantage of both Mo- and SiC - core HTGRs is that both would require an environment functionally similar to that of a graphite core HTGR (GC-HTGR), which allows use of the extensive GC - HTGR database of superalloy design properties

currently available. Additional testing may be required to extend these superalloy databases to the lower carburization potential of a Mo - core HTGR.

11. Whatever materials are selected, development of passive methods for controlling the oxidation and carburization potentials of the SNPP environment may be necessary to assure satisfactory performance of reactor core, Brayton Unit and other power conversion plant materials.

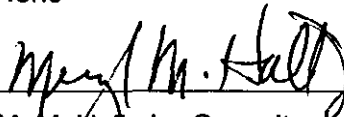
Significance to the NR Program: An equilibrium thermochemical assessment of the candidate SNPP reactor core materials shows that Mo alloys and SiC are compatible with superalloy Brayton Unit components operating in an impure He/Xe working gas environment. Use of Nb and Ta alloys will require development and qualification of protective coatings.

Future Action: No additional effort on this topic is planned.

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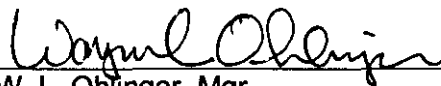
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1. Introduction

1.1. Selection of Space Nuclear Power Plant Materials

The Space Materials Plan (SMP) [1] identifies materials compatibility as a major materials selection and qualification issue for major systems of the space nuclear power plant (SNPP). The reference SNPP design is a helium/xenon-cooled reactor directly coupled to a closed cycle gas Brayton turbine-electric generator. The helium/xenon working gas will contain impurities due to the presence of residual gases at start up, outgassing of adsorbed gases from internal surfaces and reactions with carbon bearing materials that are in contact with the SNPP environment. Because of their high melting temperatures and low vapor pressures, refractory metal alloys of niobium, tantalum and molybdenum and structural ceramics based on SiC are being considered for use as reactor core materials. Because of their high temperature creep strengths, iron-nickel-, cobalt- and nickel-base alloys ("superalloys") are candidate Brayton Unit materials. There is potential for creating materials incompatibility problems by combining dissimilar materials in a recirculating gas loop having low levels of reactive impurities.

1.2. Materials Selection Strategy

Thermochemical reactions among SNPP reactor core, Brayton Unit, other power conversion plant materials and reactive gases in the working gas may degrade SNPP materials unacceptably. Refractory metal alloys perform best in oxide-reducing, decarburizing environments while the superalloys were developed and have been qualified for use in oxidizing, carburizing environments. A purpose of this study is to determine if there is an SNPP working gas chemistry that will satisfy the performance needs of any of the combinations of candidate reactor core and Brayton Unit materials. Materials selection and qualification would be simplified considerably if SNPP core materials could operate acceptably in a moderately oxidizing and carburizing environment similar to that of the graphite-core high temperature reactor (GC-HTGR), for which the superalloys are qualified [2]. There is an extensive GC-HTGR database of superalloy design properties and significant GC-HTGR operational experience currently available. Significant effort would be required to qualify superalloys for applications in an oxide reducing and decarburizing environment that would be more suitable for applications of refractory alloys. There are neither long term data nor operational experience to justify applications of superalloys in a high temperature environment where a protective surface oxide will not form and either carburization or decarburization is likely. Selection of core materials that are compatible with a GC-HTGR environment or an environment that can be shown to be functionally equivalent would allow the available effort to be focused on qualification of SNPP core materials.

1.3. Role of Equilibrium Thermochemical Analyses in Materials Selection

There is consensus in the HTGR literature that thermodynamic equilibrium exists neither among the gaseous impurities in the He coolant nor between the impurities and metal alloys in contact with the coolant [3,4]. However, concepts and considerations of equilibrium thermochemistry are important as they provide a fundamental framework upon which to construct an understanding of the nonequilibrium thermodynamics and reaction rate kinetics of thermochemical reactions as deviations from equilibrium. Equilibrium thermochemical analyses can be used to determine the thermodynamic potential for individual and competitive reactions to occur. Equilibrium thermochemistry calculations are used here to determine the relative amounts of reaction products at equilibrium in a complex heterogeneous isothermal and isobaric system. Gas-metal, gas-ceramic and gas-gas reactions that are judged most likely to contribute are included in the calculations. These calculations identify the oxidation and carburization reactions that *may and may not occur* but cannot identify the reactions that necessarily *will occur*.

Equilibrium thermochemical analyses must be benchmarked using HTGR operating experience and laboratory data. The analyses must be interpreted with caution as thermodynamic equilibrium will not be attained due to short residence times in the vicinity of SNPP components having dissimilar materials and very large differences in temperature. However, equilibrium analyses provide early information regarding the constraints that may exist for SNPP materials selection. They also provide guidance for establishing test objectives and environments for the SNPP candidate materials testing to be conducted in the future.

A major effort is required to develop a fully kinetic "burden" model capability to predict the SNPP operating environment and to experimentally qualify candidate materials in this environment. However, equilibrium thermochemical analyses and the existing materials performance data can play a significant role in materials selection decisions.

1.4. Previous and Current Equilibrium Thermochemical Analyses of Relevance to the SNPP

The HTGR literature is large and contains numerous papers on equilibrium and kinetic analyses relating the corrosion of HTGR materials to the concentration of reactive impurities in the He coolant [see for example 3,4]. A major future effort is required to thoroughly review and evaluate the HTGR corrosion literature for relevance of models and data to SNPP development. Pending completion of this effort, the approach taken for this study is to use simplified analysis assumptions and methods that are at least partially validated, as discussed below, by their ability to account for the oxidation and carburization potentials of typical graphite core - HTGR (GC-HTGR) environments.

In two previous NR program reports [5,6], equilibrium thermochemical analyses were used to establish the expected environment for operation of a SiC-core HTGR (SiC-HTGR). Important conclusions drawn from these analyses are that a SiC-HTGR environment would have impurity levels similar to those of a GC-HTR and that deliberately maintaining comparatively high concentrations of oxidizing impurities might be advantageous for promoting SiC passivity and minimizing carburization of structural alloys in the coolant circuit. The oxidation potential should be high enough to passivate both SiC and the power conversion unit materials (superalloys). These previous analyses also addressed a number of materials issues of interest to the SNPP including compatibility with Cr and Al oxide-forming heat resistant turbine alloys, Si and C transport around the system and radionuclide transport following a fuel element failure.

Reported here are results of equilibrium thermochemical analyses to determine oxidation potentials of environments in which refractory metal alloys and SiC, used as core materials, can be considered compatible (either passivate or not react) and in which the non-refractory superalloy Brayton Unit materials also may passivate, forming protective surface oxides. Other power conversion plant materials, such as those used in the gas cooler are not evaluated.

2. Discussion

2.1. Thermochemical Reactivity of Metals; Graphical Representations

SNPP reactor core and Brayton Unit metal alloys of construction will be subject to gas-metal or gas-ceramic reactions, some beneficial and some potentially detrimental to performance, depending on the specific gas and metal alloy combinations and the oxidation and carburization potentials (OCP) of the SNPP environment. In addition to the gas-metal and gas-ceramic reactions, there are gas-gas reactions that play an important role in establishing the OCP of the SNPP working gas. There are in the literature several graphical analysis methods that can be used to portray the relative thermochemical reactivities of SNPP metal alloys and ceramics and to synthesize the multitude of complex gas-metal and gas-ceramic and gas-gas corrosion reactions. Two that have been most extensively developed and used to evaluate GC-HTGR environments and corrosion data are the Ternary Environment Attack (TEA) diagram introduced by Brenner and Graham [3] and the "modified stability" diagram developed by Quadackers and Schuster [4,7]. A third analysis method, which is used in this study to obtain an understanding of the factors determining the environment of the GC-HTGR,

was discussed by Ebberink, Krompholz and te Heesen [8] and mentioned and illustrated without discussion by Nickel, Schubert and Schuster [9].

2.1.1. Ellingham Diagrams

The Ellingham Diagram is a graphical method that is customarily used to portray thermochemical reactivities of metals. These diagrams cannot be used for the analysis of corrosion in a He environment containing multiple reactive impurities but may be used to get a first estimate of the thermochemical reactions most likely to occur. In the case of oxidation and carburization, these are diagrams versus temperature of the standard Gibbs free-energies required to react 1 mole of O_2 to form metal oxides or 1 mole of C to form metal carbides, respectively. Figures 1 and 2 show the standard Gibbs free energies released, ΔG^o , (a measure of "reactivity") on formation of the oxides and carbides of interest.

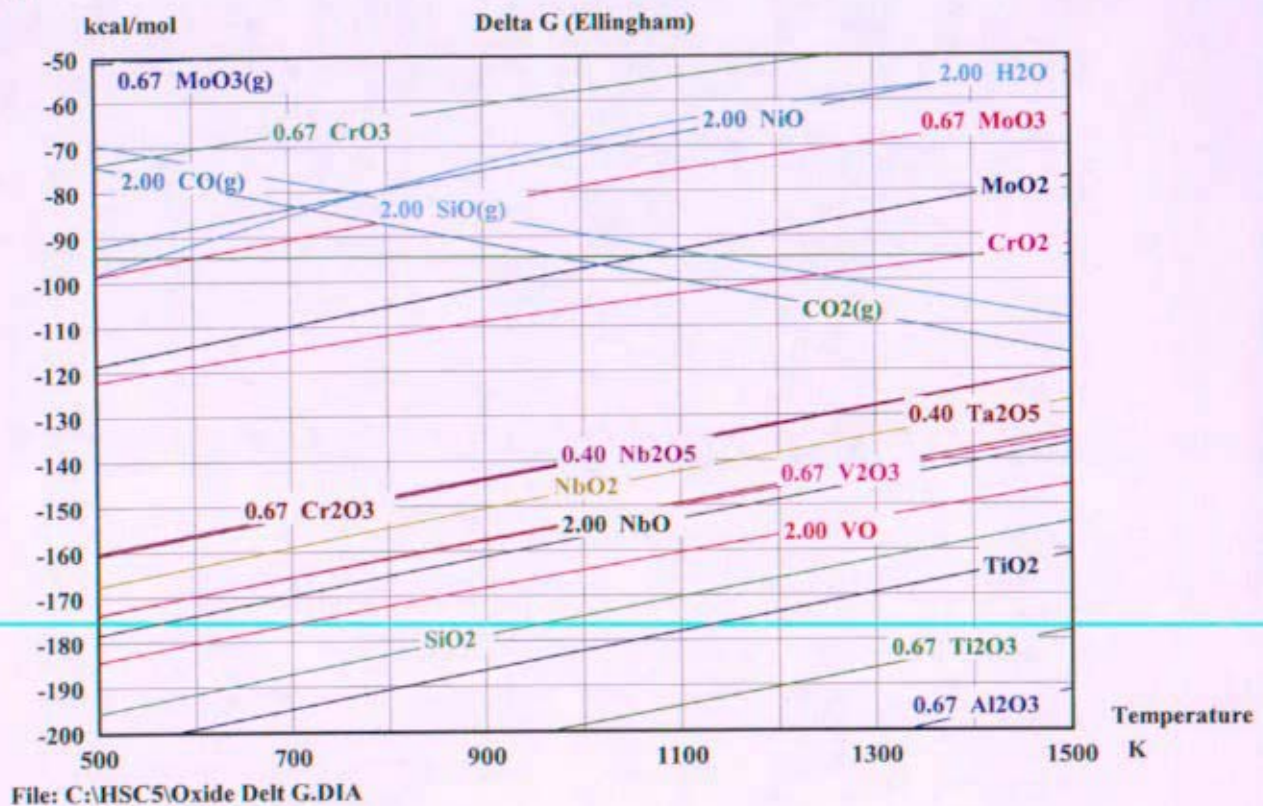


Figure 1. Gibbs Free Energy Release on Reaction of Metals with 1 Mole of O_2 to form Metal Oxides. Curves for the more reactive metals lie lower (at more negative Gibbs free energy of formation) on the chart.

These figures and the ones that follow were obtained using the equilibrium thermodynamic code HSC [10]. These figures show that, in both oxidizing and carburizing environments, Nb and Ta are more reactive than Cr and may then be expected to preferentially form oxides and carbides when thermodynamically coupled with Cr and C bearing superalloys. Al is more reactive than both Nb and Ta in an oxidizing environment and may preferentially form oxides when thermodynamically coupled to Nb and Ta alloys. However, Nb and Ta are more reactive than Al in a carburizing environment and may preferentially absorb carbon and form carbides when thermodynamically coupled with a superalloy having Al and C. Figure 1 shows that at a given temperature Mo is significantly less reactive with O_2 than is Cr (or Al). Figure 2 shows that Mo, Cr and Al have reactivities with C that are comparable at a core temperature of 1400 K (Mo) and a turbine temperature of 1150 K (Cr and Al). We conclude that Mo may not preferentially absorb oxygen and form oxides but potentially may form carbides when thermodynamically coupled with Cr, Al and C bearing superalloys.

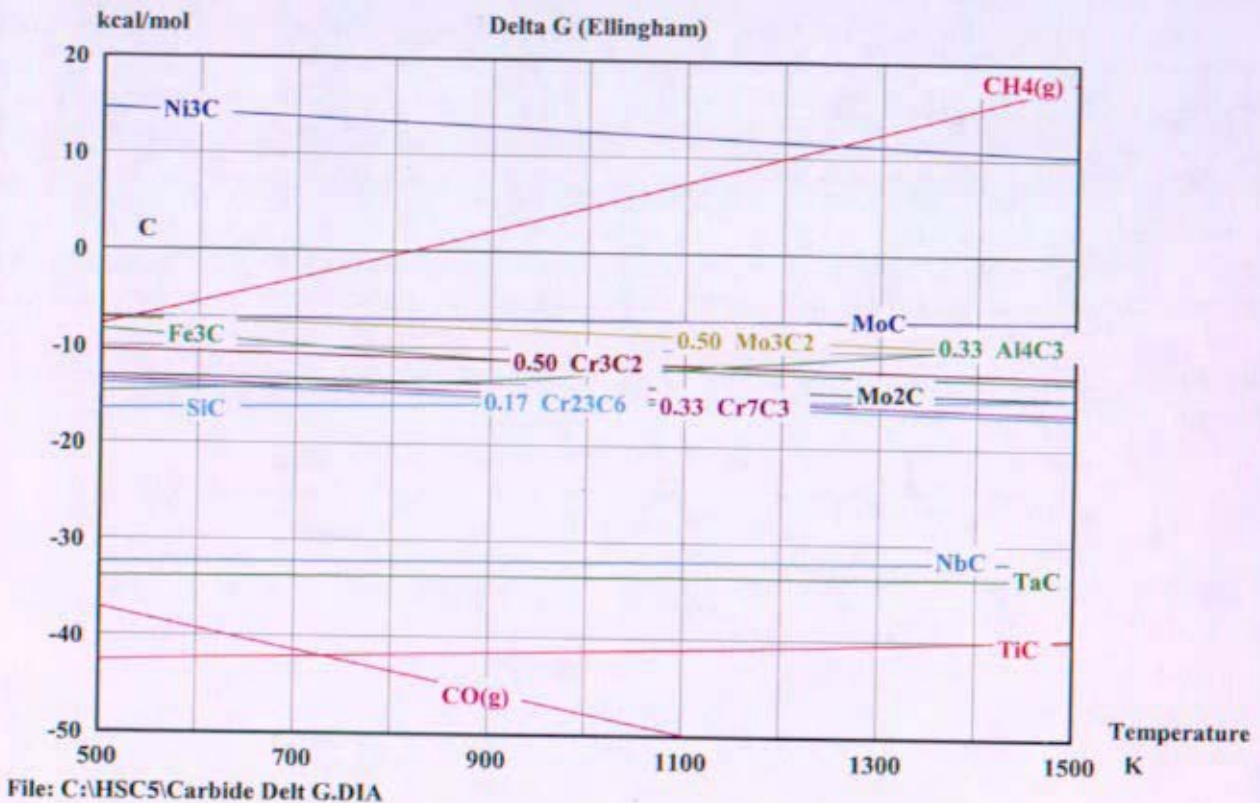


Figure 2. Gibbs Free Energy Release on Reaction of Metals with 1 Mole of C to form Metal Carbides. Curves for the more reactive metals lie lower (at more negative Gibbs free energy of formation) on the chart.

2.1.2. Phase Stability and Predominance Area Diagrams

While informative, Ellingham diagrams are not sufficient to determine whether or not a metal will oxidize or carburize in an environment that contains both oxidizing and carburizing constituents. Formation of oxides and carbides depends on the relative concentrations and reactivities of oxidizing and carburizing constituents in the environment. The principal reactive gases in an HTGR are water vapor, H₂O(g), (causes oxidation and decarburization), carbon monoxide, CO(g), (causes oxidation and carburization), methane CH₄(g) (causes carburization) and hydrogen, H₂(g), (causes oxide reduction). Present at low concentrations are carbon dioxide, CO₂(g) (a product of carbon monoxide oxidation), oxygen, O₂(g), and C(g), which are usually treated as dependent variables and assumed to have equilibrium concentrations determined by gas-gas and gas-carbon reactions. The working gas of the SNPP also will contain oxygen present as gaseous metal oxides. Low concentrations of nitrogen, N₂(g), may be present but are usually neglected in considerations of HTGR impurity effects as N₂(g) is an essentially non-reactive gas due to the relatively slow kinetics of reaction. Formation of nitrides in HTGR environments has not been observed.

Phase stability and predominance area diagrams allow competitive thermochemical reactions to be taken into account. Figure 3 is an example of an isothermal phase predominance diagram for the Cr-C-O system^a. The independent variables are taken as the partial pressures of O₂(g) and C(g), measures of the oxidation and carburization potentials of the environment, respectively.

To the right of the vertical and intersecting diagonal lines in Figure 3 metals carburize with the carburization potential increasing with increasing distance to the right of the line. To the left of these

^a Figure 3 is essentially that developed by Quadackers and Schuster [4,7] with the axes interchanged and the regions differently labeled to better suit the development here.

lines metal carbides are thermodynamically unstable so metals containing carbon will decarburize with the decarburization potential increasing with increasing distance to the left of the line.

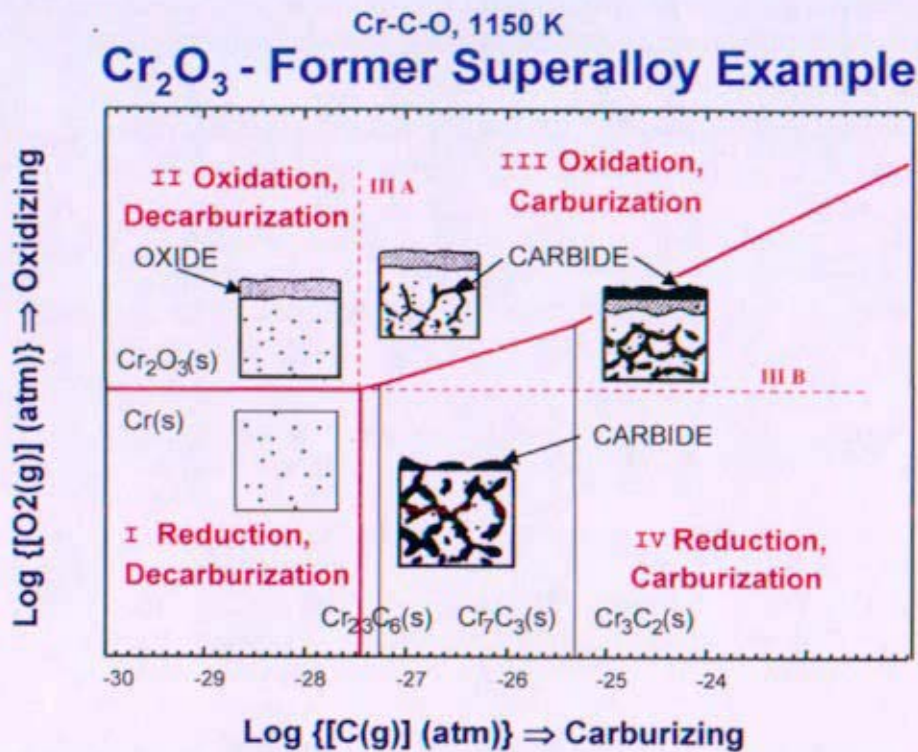


Figure 3. Phase Predominance Diagram for Chromium Showing Regions of Oxidation and Carburization. The Group V refractory elements Nb, Ta and V are embrittled in all but oxide-reducing and decarburizing environments (Region I of their respective diagrams). The heat-resistant superalloys are qualified for applications in Regions II and IIIA of their diagrams.

To the left of the vertical line and below the horizontal line the metal is stable, preexisting oxides will be reduced. Above this line and the intersecting diagonal lines the metal oxide may form with the oxidation potential increasing with increasing distance above this line.

2.1.3. Oxidation-Carburization Potential Regions, Microstructure and Performance of Alloys

The lines in Figure 3 divide the phase predominance diagram (PPD) into four regions of oxidation-carburization potential (OCP). In Region I, the OCPs are low and metal is the thermodynamically stable phase. This is the preferred region of the relevant PPDs for applications of Group V refractory metals such as Nb and Ta. They have large thermochemical reactivities in environments containing oxygen, carbon and nitrogen and are embrittled due to absorption of low ppm concentrations of these interstitial elements [11] so that Regions II, III and IV of their PPDs should be avoided. Group V metals have very low vapor pressures so that evaporation of bare metal is not a concern for applications in Region I.

The Group VI refractory elements, which include Mo, also perform well in Region I. However, Mo may not be limited to Region I applications as Group VI elements have much lower solubilities for oxygen, carbon and nitrogen. Mo alloys form surface films of the stable dioxide MoO₂ without severe interstitial element embrittlement. Moreover, Mo alloys having up to 0.2 w/o C having good ductility have been developed [12]. This provides potential for applications of Mo in Regions II and perhaps IIIA. Additional literature search and potentially experimental effort is required to determine if MoO₂ is

sufficiently protective to prevent excessive carburization in Region IIIA. Application of Mo in Regions II and III must be done with full knowledge of the potential for forming the volatile trioxide, MoO_3 . Application of Mo in Region IV results in unacceptably severe carburization and should be avoided.

Region I should of course be avoided for applications of SiC. Region II and perhaps Region III are preferred as SiC forms a protective oxide, which greatly reduces the rate of SiC corrosion that may occur for applications of SiC in Regions I and V of the PPD [13].

Region I of the PPD should be avoided in applications of the superalloys as protective surface oxides of Cr and Al will be reduced and internal carbides will be destabilized and depleted to a depth that depends on temperature, time, solubility and diffusivity of carbon. Without protective surface oxides, elements such as Mn, Fe, Cr and Ni, which have low vapor pressures at high temperatures, will vaporize, depleting surfaces layers of these beneficial alloy constituents [14]. Decarburization will result in loss of carbides and reduction of the creep strength of superalloys.

The preferred PPD regions for applications of superalloys are Regions II and IIIA and for carburization potentials within these regions that lie close to the diagonal that separates carburizing from decarburizing regions. Metal oxides are stable in Regions II and III and carbides are stable in Region III. Nearer the diagonal line the kinetics of carburization-decarburization may be sufficiently slow relative to the oxidation kinetics such that protective oxide films may grow and prevent carburization-decarburization altogether or limit carbon reactions to a shallow region near the surface. However, for larger carburization potentials, the growth of a carbide film may interfere with formation of the oxide film allowing deep carburization of the alloy. Low carburization potentials may allow preexisting carbides to be destabilized prior to formation of a protective oxide, leading to carbide depletion to a depth depending on the kinetics of the carbide dissolution and permeation of carbon to the surface. In Region IV metal carbide is the stable phase, protective oxides will not form and carburization may be extensive at moderate to high carburization potentials. This is the PPD region that generally is most detrimental to the performance of both refractory and non-refractory metals. Miller [5] summarized the situation for superalloys having Cr for oxidation-carburization protection: "...the range of gas mixtures which give good behavior for Cr-containing alloys and are still compatible with a graphite core is quite small, and gets smaller as temperature increases. If the mixture is "well balanced", a comparatively protective oxide forms, and the alloys are neither severely carburized nor decarburized. If there is not enough H_2O relative to H_2 , CH_4 , and CO , the alloys can be severely carburized. If there is too much H_2O , an oxide still forms, but it is not as protective, and the alloys can be severely decarburized, which leads to degradation of mechanical properties and creep strength."

2.2. Use of Equilibrium Thermochemical Calculations to Anticipate the SNPP Environment

The impurity gases that will exist in the SNPP and the OCP of the environment that they establish are not known. Effort is currently being applied to develop a "burden model" that will provide estimates of the environments that can develop on start up and operation of the SNPP. Development of the SNPP Burden Model, when completed, will provide a method for predicting the environments that may develop for a given pre-startup environment assumption and a selected set of SNPP core and power conversion plant materials. In the meantime, equilibrium thermochemical calculations provide an ability to determine the thermodynamic potential for environments of interest to develop. Moreover, operating experience with GC-HTGRs and results of laboratory experiments provide environmental data that may be used to assess the relevance of these calculations.

2.2.1 Principal Contributors to the SNPP Environment

It is expected that adsorbed water vapor, present at the time the system is sealed prior to startup, will be a major contributor to the impurity gases that develop during operation. The amount of water vapor that is present on startup will depend on the total surface area for water adsorption and the extent of outgassing that can be accomplished. In addition to the metal surfaces in contact with the working gas, heat insulator materials such as ceramic wool, which have very large surface areas, can significantly contribute to the water burden. It is not known if the SNPP can be heated to temperatures in the 400°

C to 600° C range while drawing the high vacuum needed to obtain adequate outgassing. Moreover, as discussed below, it may be necessary to use passive methods to control water vapor pressures within limits in order to establish and control the oxidizing potential of the SNPP environment to assure acceptable performance of SiC and superalloys.

With or without use of special measures, the SNPP environment will develop over time as a result of thermodynamic and kinetic competition among the core and power conversion plant materials that are in contact with the gases circulating through the system, which includes metal alloys containing significant carbon, core materials containing silicon and carbon in the case of a SiC core, porous ceramics, potting materials, electrical and heat insulators, lubricants and environmental control materials, if used. The environment that develops will depend on the initial purity of the charging gas, the effectiveness of the system de-gas procedure, the compositions, reactivities and surface areas of the materials that are exposed to the closed environment. Outgassing of occluded volumes, desorption of adsorbed and absorbed gases and diffusion limited dissolution of carbides and desorption of carbon may act as "virtual leaks", contributing impurities to the system long after reactor start up. During SNPP operation impurities in the circulating gas will approach steady state values that are determined by the kinetics of virtual leaks, equilibrium thermodynamics and kinetics of formation of metal oxides and carbides and the relative masses of materials that contribute to and take from the circulating gas impurities.

2.2.2 GC-HTGR Environments

The oxidizing and carburizing environments developed in the operation of GC-HTGRs are relevant to SNPP in that a functionally similar SNPP environment will provide an environment that is favorable for SiC and superalloy performance and will minimize the effort required to qualify SNPP Brayton Unit materials. Moreover, Miller has concluded [5] that, without special measures to control the oxidation potential of the SiC-HTGR, "...an HTGR with a silicon carbide core would have impurity levels similar to those in a graphite-core HTGR." A "typical" GC-HTGR environment has been chosen for the initial SNPP materials testing:

Table I provides impurity gas concentrations that may be considered to be representative of a GC-HTGR. Typical operating GC-HTGR impurity gas concentrations were derived from a review of experience in operating GC-HTGRs [15]. Impurity concentrations are given for a simulated GC-HTGR environment test, which was conducted by ORNL for the NR program [16], and for a similar test conducted by General Electric [see reference 16]. Finally, there are relevant data in the literature that have been obtained in a "Prototype Nuclear Process Heat (PNP) environment [8].

Table I. Impurity Gas Concentrations Used in Simulated GC-HTGR Environmental Testing ($\mu\text{atm.}$)

Environment	H ₂ (g)	H ₂ O(g)	CO(g)	CO ₂ (g)	CH ₄ (g)	N ₂ (g)	He(g)	$\frac{[\text{H}_2\text{O}]}{[\text{H}_2]}$	$\frac{[\text{CO}][\text{CH}_4]}{[\text{H}_2]^2}$
Typical GC-HTGR	450	1 - 3	45	0.1	45	10	bal.	0.0044	0.0100
ORNL	400	1	40	0.2	20	<10	bal.	0.0025	0.0050
GE	400	2	40	0.2	20	<20	bal.	0.005	0.005
PNP	500	1.5	15	---	20	< 5	bal.	0.003	0.0012

2.2.3 Oxidation and Carburization Potentials for Reaction of Water Vapor with Graphite and Relevance to the GC- HTGR Environment

The partial pressures O₂(g) and C(g) used as measures of oxidation and carburization potentials in Figure 3 are not included in Table I as they are too small to be measured. Therefore, we need

alternate oxidation and carburization potential variables that may be defined in terms of the measured reactive gases present in HTGR systems.

Assuming that the gas-gas reactions are in equilibrium,



where the equilibrium constant is

$$K_1 = \frac{[H_2O]^2}{[H_2]^2[O_2]}, \quad \text{then} \quad [O_2] = \frac{1}{K_1} \left\{ \frac{[H_2O]}{[H_2]} \right\}^2. \quad (2)$$

The hydrogen and methane will be in equilibrium with free carbon^b:

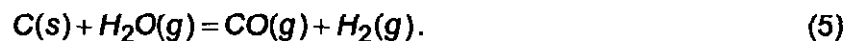


where the equilibrium constant is

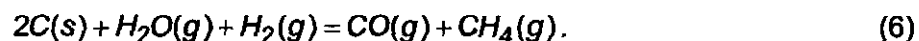
$$K_3 = \frac{[CH_4]}{[C][H_2]^2}, \quad \text{then} \quad [C] = \frac{[CH_4]}{K_3[H_2]^2}. \quad (4)$$

The second of Equations (2) shows that the oxidation potential of the environment, $[O_2]$, is proportional to $\{[H_2O]/[H_2]\}^2$ and the second of Equations (4) shows that the carburization potential $[C(s)]$ is proportional to $[CH_4]/[H_2]^2$.

The operating GC-HTGR environment has been reported to be in large part determined by the reaction of gaseous impurities with the hot graphite core [17]. The porous graphite core provides a large reservoir for adsorbed water that will react with graphite to form carbon monoxide, hydrogen and methane on start up of the reactor. The relevant reactions are Equation (3) and



Combining Equations (3) and (5), we obtain the reaction equation relating equilibrium among C(s) and the principal reactive impurity gases:



The equilibrium constant is

$$K_6 = \frac{[CO][CH_4]}{[C]^2[H_2O][H_2]}. \quad (7)$$

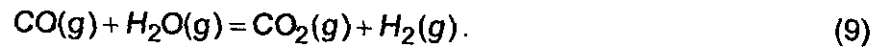
Rearranging, the relationship between the alternative oxidation and carburization potentials, Equations (2) and (4), when water vapor is reacted with graphite, is given by

$$\frac{[H_2O]}{[H_2]} = \frac{[CO]}{K_6} * \frac{[CH_4]}{[H_2]^2}, \quad (8)$$

where we have assumed that for the GC-HTGR carbon is in excess of that needed to attain equilibrium with the reactive impurities, so that the carbon activity can be taken to be $[C(s)] = 1$. The meaning of this equation is that the oxidation and carburization potentials of the GC-HTGR are not independent if the GC-HTGR environment is dominated by the reaction of the water "burden" with the hot graphite core.

^b Note that this equilibrium could be written for gaseous carbon C(g) instead of solid carbon C(s) using the equilibrium constant appropriate to this alternate reaction.

There are six species; $H_2O(g)$, $CO(g)$, $CO_2(g)$, $CH_4(g)$, $H_2(g)$ and $C(s)$, and three components; C, O and H. Therefore a total of three independent equilibria are all that is needed to define the state of the system. The equilibrium among the oxidized species of C(s), that is, $CO(g)$ and $CO_2(g)$, is given by



The equilibrium constant for this reaction is

$$K_9 = \frac{[CO_2][H_2]}{[CO][H_2O]}. \quad (10)$$

Therefore, the oxidation potential of the water vapor - graphite system, in terms of our alternate variable, is related to the partial pressures of $CO(g)$ and $CO_2(g)$ by

$$\frac{[H_2O]}{[H_2]} = \frac{[CO_2]}{K_9[CO]}. \quad (11)$$

Equations (8), diagonal line, and (11), horizontal line, are plotted in Figure 4 using the HSC database to obtain the equilibrium constants. Equation (11) is plotted using the $CO(g)$ and $CO_2(g)$ concentrations for the "typical" GC-HTGR environment given in Table I. Also shown is the location of the GC-HTGR typical and test environments.

Note that the OCP of the typical operating GC-HTGR lies at the intersection of the $C(s)$ stability line, Equation (8), and the $CO(g) - CO_2(g)$ equilibrium oxidation potential line, Equation (11). The goodness of this coincidence is of course fortuitous given a graphite temperature of 1400 K is a guess at best. However, sensitivity of the intersection point to temperature is not great.

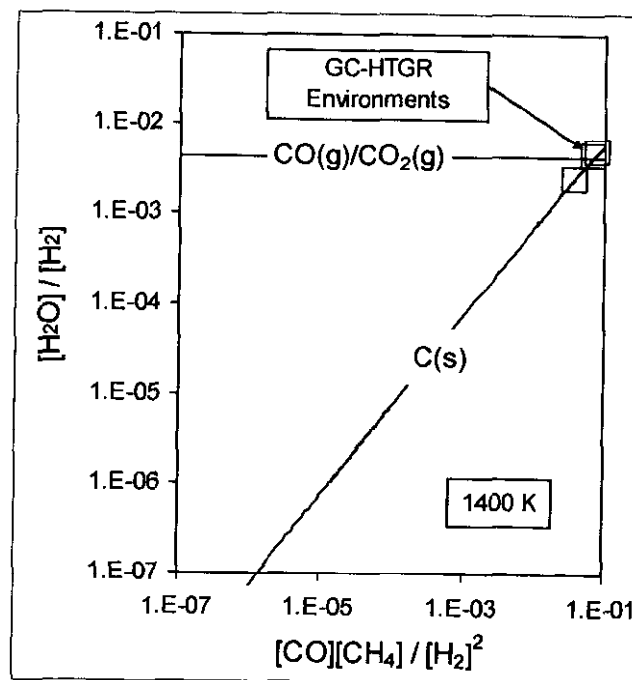
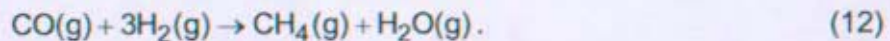


Figure 4. OCP diagram for carbon showing the location of a typical GC-HTGR relative to the carbon stability line and carbon monoxide oxidation potential. The implication of this figure is that the reactive impurities of a GC-HTGR are in thermodynamic equilibrium with the graphite core.

It then appears that at least the ratios of the major reactive impurities that determine the oxidation and carburization potentials of the typical GC-HTGR environment may be influenced significantly by a tendency toward equilibrium with the graphite core. However, Quadackers and Schuster [4] have pointed out that the methane levels of the typical GC-HTGR environment are larger (~10X) than expected based on equilibrium thermochemistry. Not discussed by these authors is that the carbon

monoxide concentration of the typical GC-HTGR environment is about one-tenth the expected equilibrium amount. Then this could explain why the typical GC-HTGR environment may, as in Figure 4, fall on the graphite C(s) stability line as the product $[CO][CH_4]$ remains unchanged by the observed departures from their expected equilibrium values. This may be rationalized as radiolysis is known to significantly enhance the production of $CH_4(g)$ in GC-HTGRs [17] and the equilibrium constant of the following reaction is increased by a factor of 10 due to radiolysis:



Radiolysis is known not to influence the ratios of impurity gas concentrations, Equation (11), that determine the oxidation potentials of the GC-HTGR environment [17].

2.2.4 Estimation of the GC- HTGR Water Burden

The discussion above shows that, given independent measurements of impurity concentrations and the OCPs derived from them, equilibrium thermochemistry calculations can be used to locate the GC-HTGR environment OCP on the graphite stability line. The result is that the reactive impurities appear to be in equilibrium with the graphite core, when we use Equation (8) defining the relationship between the oxidation and carburization potentials of the GC-HTGR environment. With this understanding, we proceed to apply equilibrium thermochemistry to an analysis of the GC-HTGR and SNPP environments.

As adsorbed water vapor is expected to be a major impurity present on startup of the GC-HTGR and the SNPP, we have an interest in estimating the water burden of the GC-HTGR to provide a basis for comparing the SNPP environment with GC-HTGR experience. We do this by calculating the oxidation potential of the environment formed when the water burden reacts with graphite as a function of the magnitude of the water burden. Figure 5 shows the results of these calculations, which were performed using the Equilibrium Module of HSC Chemistry, which calculates multi-component equilibrium compositions in a heterogeneous system at isothermal and isobaric conditions. These results were used to calculate the oxidation potential, as expressed by the ratio $[H_2O]/[H_2]$, as a function of the water burden, the results of which are shown in Figure 6. Also shown in Figure 6 is the range of oxidation potential for GC-HTGR typical and test environments. Then the GC-HTGR water burden can be estimated as 0.037 - 0.080 mole fraction.

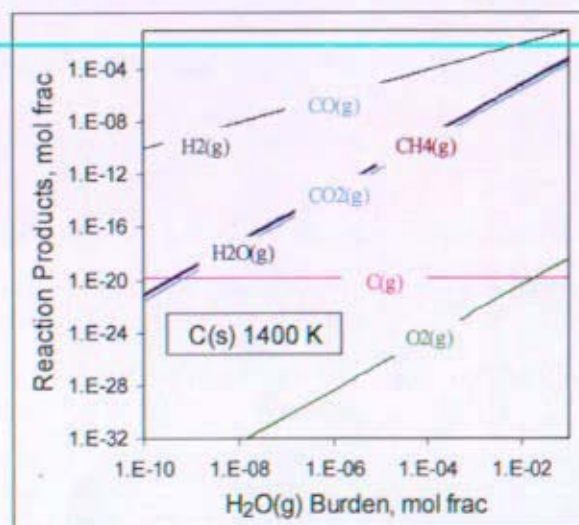


Figure 5. Reaction products formed on reacting graphite with water in a closed system at 1400 K and containing 1 kmol helium at a system pressure of 20 bars.

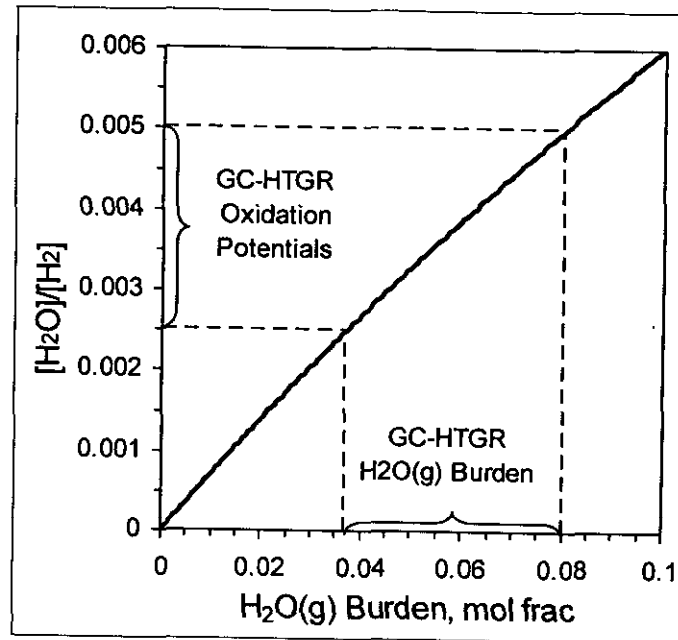


Figure 6. GC-HTGR water burdens determined by using the Figure 5 relationship between oxidation potential and water burden the range of oxidation potentials for GC-HTGR typical and test environments found in Table I.

3. Compatibility of SiC, Nb and Mo Alloys with Chromia Former Superalloys

3.1 Analysis Assumptions and Methods

In order to proceed while not having knowledge of the sources and concentrations of impurities in the working gas of an operating SNPP, we assume that adsorbed water vapor is the principal impurity available at startup of the SNPP reactor and that reactions of the water vapor burden with the metals Nb, Mo, Cr and with carbon, available as SiC and chromium carbides, are the only sources of the reactive impurities $H_2O(g)$, $CO(g)$, $CH_4(g)$ and $H_2(g)$. It is further assumed that all the reactants, except for the water burden, are available in excess of their equilibrium amounts. Reaction products amounts are calculated using HSC as a function of one independent variable, the water vapor burden.

Figure 7 illustrates the assumptions of the analysis relative to temperatures and the sequence of analysis steps taken to accommodate limitations of the HSC code. As HSC cannot calculate reactions between reactor and Brayton Unit materials at different temperatures, the following analysis steps are used:

For the SiC Cores

1. React 1 kmol 20 bar He plus variable $H_2O(g)$ with SiC at 1400 K.
2. Computationally isolate and cool the gases formed in contact with SiC to 1150 K.
3. React this gas with chrome carbide (Cr_3C_2 chosen as representative) at 1150 K (Brayton Unit inlet) to 920 K (outlet)

The working gas chemistry will of course be modified by the reactions in Step 3 and this gas will be further modified by passing through the reactor core again, and so on until a steady condition is obtained. However, only steps 1 - 3 are calculated, without iteration, in the analysis conducted here.

For the Nb and Mo alloy cores

1. React 1 kmol 20 bar He plus variable $H_2O(g)$ with Cr_3C_2 at 1150 K.
2. Computationally isolate and heat the gases formed in contact with Cr_3C_2 to 1400 K.
3. React this gas with Nb and Mo

Again, these steps are not iterated.

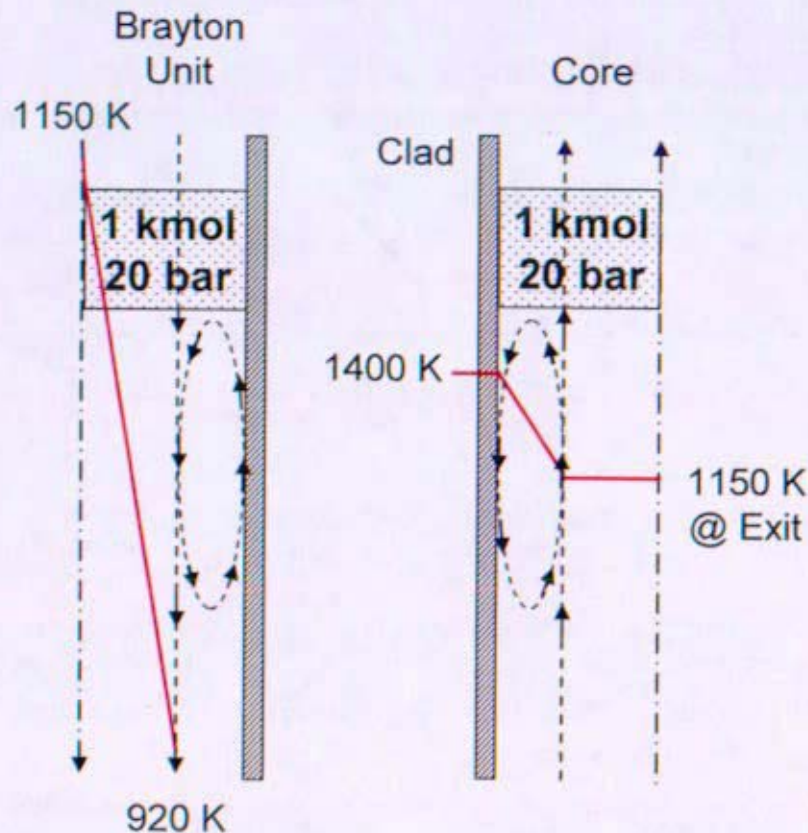


Figure 7. Illustrating analysis assumptions relative to temperatures and the sequence of analysis steps taken to accommodate limitations of the HSC code.

3.2 Thermochemical Stability of SiC, Cr₃C₂, Nb and Mo in the Presence of Water Vapor

Figure 8 shows the reaction products due to reaction of water vapor with SiC at 1400 K. This figure shows that SiC reacts to form the volatile oxide phase SiO(g) and silicon vapor Si(g) for a water burden of less than about 2.6E-6 mol frac H₂O(g) and that the more stable SiO₂ is stable above this water burden. Also shown is that free carbon "dust" may potentially form for a water burden greater than about 6.0E-4 mol frac.

Figure 9 shows the reaction products due to reaction of the water vapor with Cr₃C₂, which is taken as representative of the three carbides of Cr, at 1150 K. This figure shows that below a water burden of about 2.8E-8 mol frac, Cr₃C₂ is unstable, forming Cr(s) and the volatile Cr(g). In this case decarburization and dealloying of the chromia former superalloys will occur. Above this water burden, chromia Cr₂O₃ is stable so that a protective surface oxide may form, preventing either excessive carburization or decarburization. There is potential for the formation of free carbon dust for a water burden above about 2.1E-4 mol frac.

Figure 10 provides a summary of the oxidation reactions for SiC, Cr₃C₂, Nb and Mo in the presence of water vapor. This figure shows that a water burden of less than about 1.0E-6 mol frac is required to prevent the formation of NbO. Embrittling concentrations of oxygen are soluble in Nb at even lower oxidation potentials. Given the expected difficulty of lowering the water burden in the SNPP to these levels, it can be concluded that use of Nb would require use of a protective coating.

At the water burden needed to form a protective Cr₂O₃ oxide, Mo oxides will not form. Moreover, the water burden needed to stabilize superalloys in a Mo-core SNPP design is at least 1/100 X that needed for a SiC-core design.

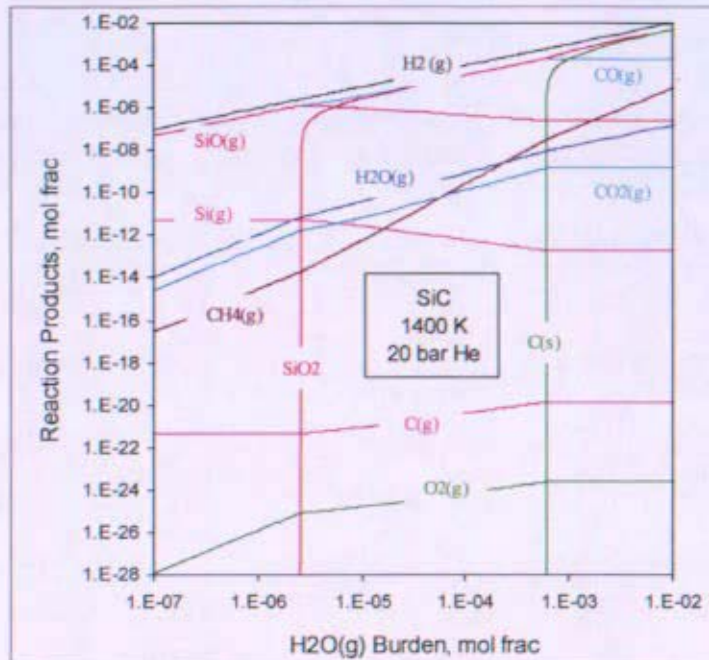


Figure 8. Reaction products due to reaction of SiC with water vapor at 1400 K. A minimum water burden of about 2.6E-6 mole fraction is needed to passivate SiC by the formation of SiO₂.

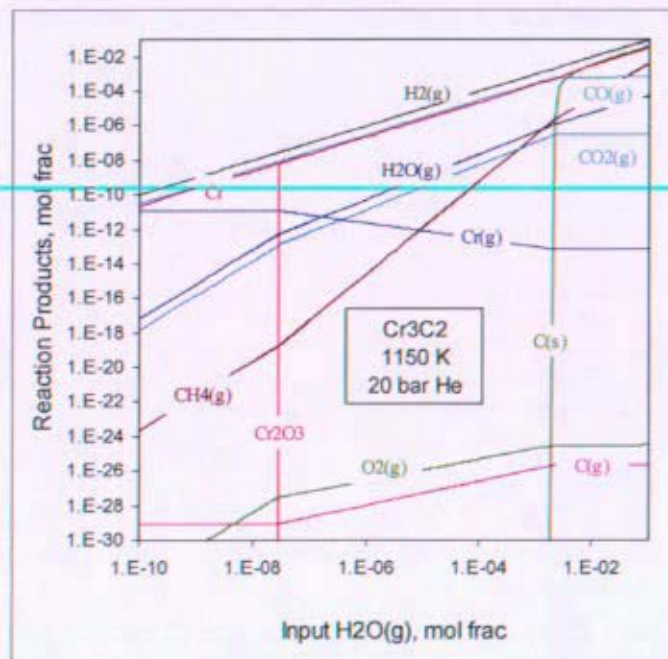


Figure 9. Reaction products due to reaction Cr₃C₂ with water vapor at 1150 K. A minimum water burden of about 2.8E-8 mole fraction is needed to passivate chromia former alloys having Cr₃C₂ carbides.

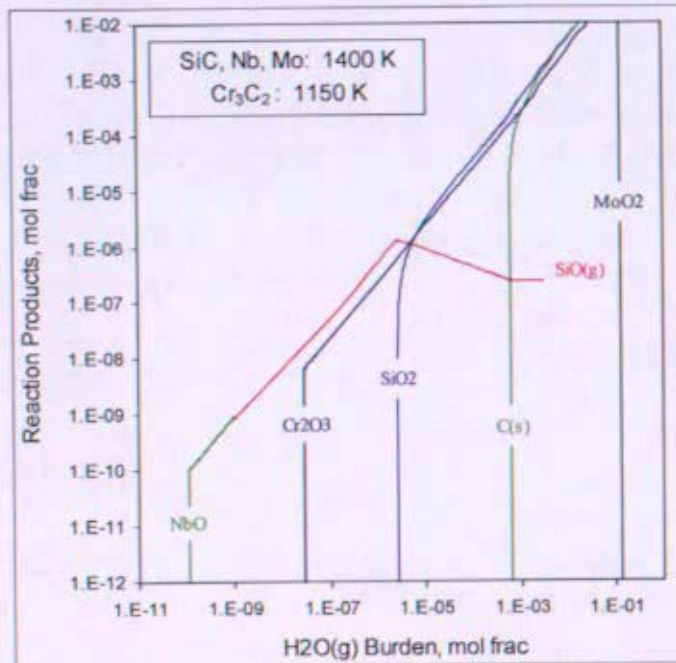


Figure 10. Water burden required to form desirable surface oxides for SiC and chromia former superalloys and to avoid oxidation of Nb and Mo.

3.3 Passivation of Chromia Former Superalloys at 920 K - 1150 K in Thermodynamic Communication with SiC at 1400 K

Heat resistant superalloys have been developed and qualified to operate in oxidizing and carburizing environments. Satisfactory performance of iron-nickel-base superalloys depends on formation of protective chromia oxide (Cr₂O₃) films and the cobalt- and nickel-base superalloys depend on formation of chromia, alumina oxide (Al₂O₃) and mixed chromia/alumina films. Formation of protective oxides is essential for the prevention of excessive carburization and decarburization and depletion of alloying elements due to evaporation. As alumina is far more stable than chromia, we consider passivation of chromia former alloys as limiting.

A ceramic fuel system based on SiC requires an oxidizing environment to form SiO₂. Figure 11 shows the effect of reducing the temperature of the gas formed by interaction of water vapor with SiC at 1400 K, for a water burden (oxidation potential) high enough to form SiO₂ but low enough to prevent formation of free carbon (6E-4 mole fraction), so-called carbon "dusting". The figure shows that reducing temperature results in a reduction in both the oxidation (O₂(g)) and carburization (C(g)) potentials of the environment. Figure 12 shows the reaction products formed by exposure of chromium carbide to this environment. The carbide Cr₃C₂ was chosen for the analysis as this carbide forms at the highest carbon potential of the three major Cr carbides and is therefore expected to form first at the superalloy surface.

Figure 12 shows that chromia former superalloys may not form a protective Cr₂O₃ oxide at temperatures greater than about 1100 K and that water burdens greater than about (6E-4 mole fraction) may be necessary to passivate superalloys to higher temperatures. We conclude that SiC used as a core material is compatible with superalloy Brayton Unit materials but that the required oxidation potential may exceed the potential at which free carbon may form.

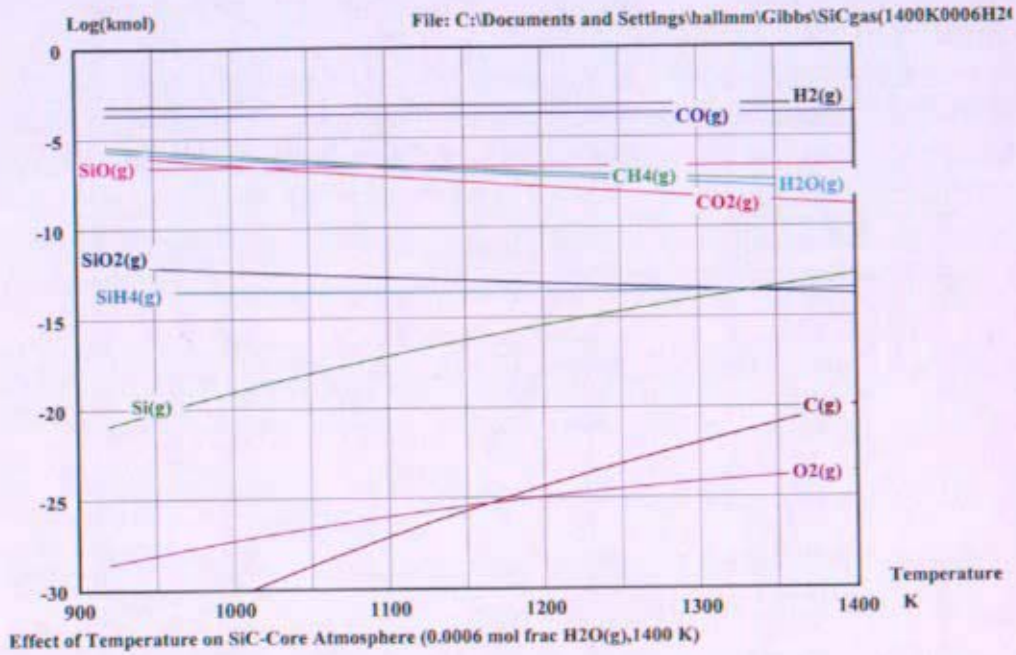


Figure 11. Effect of reducing temperature of the gas formed by interaction of SiC with water vapor at 1400 K.

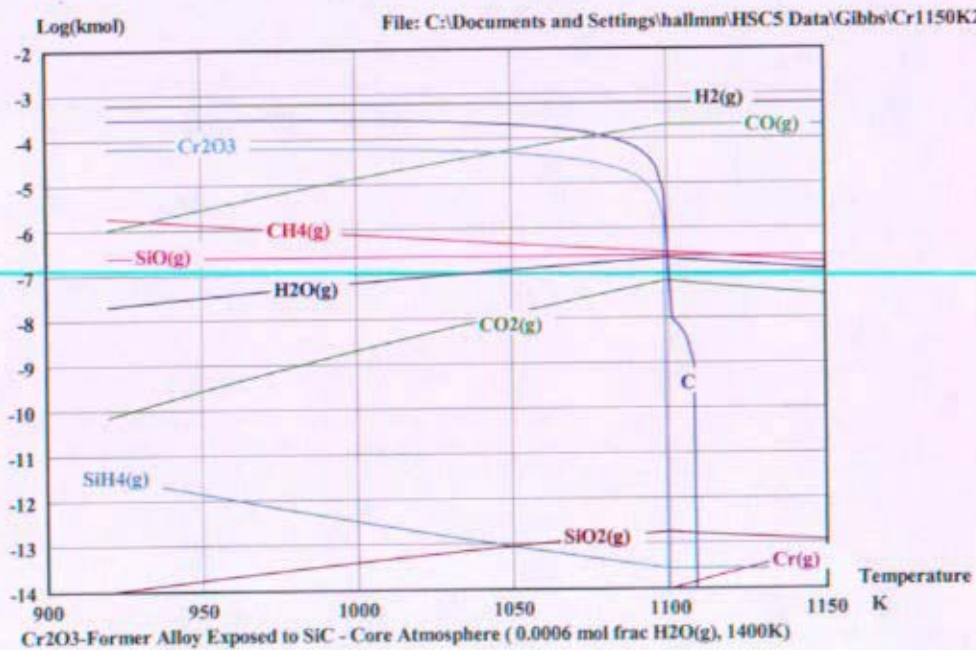


Figure 12. Reaction products formed by exposure of 1150 K Cr₃C₂ to an environment formed by reaction of SiC with the maximum water burden allowed to prevent formation of free carbon at 1400 K.

3.4 Oxidation and Carburization of Nb and Mo Refractory Metal Alloys

When using either Nb or Mo alloys as core materials the objective is to prevent oxidation and carburization of these alloys while at the same time providing an oxidizing environment for chromia former superalloys. Figure 10 shows that this is not possible for Nb alloys but appears to be possible for Mo alloys. Use of Nb alloys will require development of a protective coating. While Mo alloys may

not oxidize, the question for their use is whether these alloys will be excessively carburized in an environment suited for chromia former superalloys. Figure 13 shows the effect of increasing temperature on the environment formed by reaction of Cr_3C_2 with the minimum water burden required to passivate chromia former superalloys at 1150 K. This figure shows an order of magnitude increase in both the oxidation and carburization potentials on increasing the temperature to 1400 K.

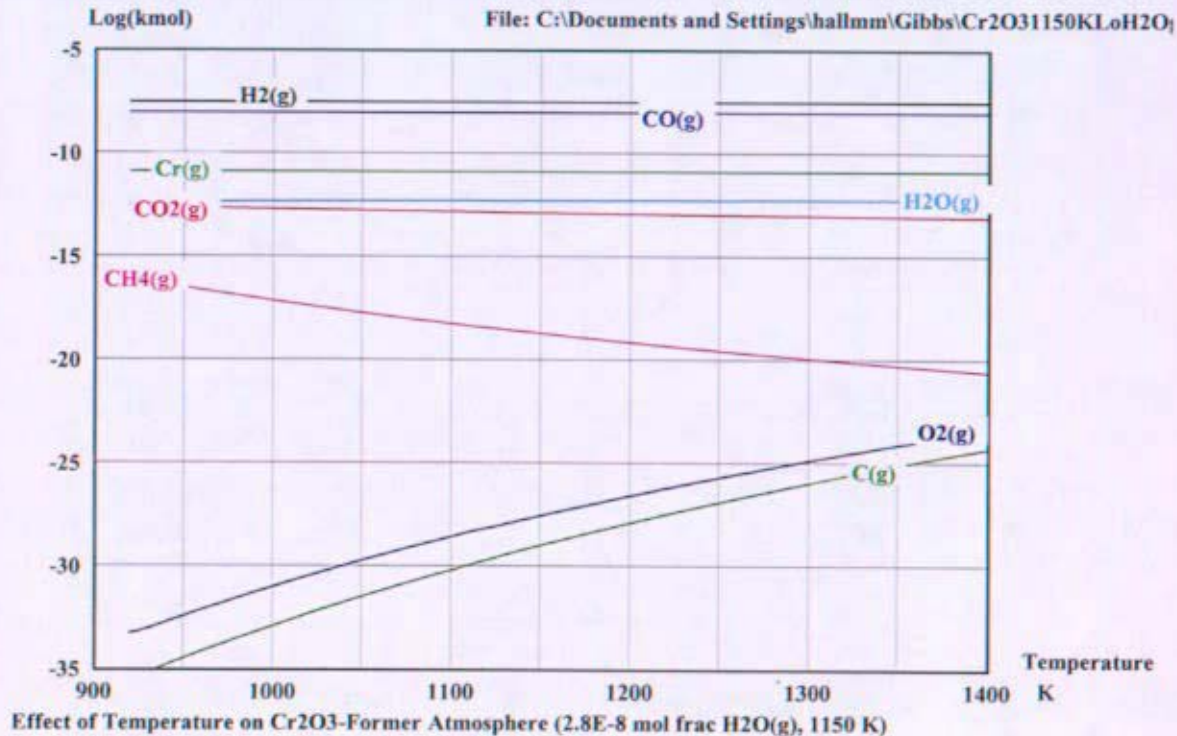


Figure 13. Effect of increasing temperature on the environment formed by reaction of Cr_3C_2 with the minimum water burden required to passivate chromia former superalloys at 1150 K.

Figures 14 and 15 show the reaction products due to exposure of 1400 K Mo to environments formed by reaction of Cr_3C_2 at 1150 K with the minimum water burden required to passivate chromia former superalloys ($2.8\text{E}-8$ mole fraction) and the maximum allowed water burden to prevent formation of free carbon ($2.1\text{E}-4$ mole fraction), respectively. Figure 14 shows that neither oxides nor carbides of Mo are formed at the lower water burden and that Mo carbide forms at the higher water burden. These figures show that upper and lower limits on the oxidation potential of the Mo-core SNPP environment may be necessary if the chromia former alloys are to form protective surface oxides and carburization of Mo must be avoided.

4. Conclusions

1. There is consensus in the high temperature gas reactor (HTGR) literature that thermodynamic equilibrium exists neither among the gaseous impurities in the He coolant nor between the impurities and metal alloys in contact with the coolant. However, concepts and considerations of equilibrium thermochemistry are important as they provide a fundamental framework upon which to construct an understanding of the thermodynamic potential for thermochemical reactions. Reaction rate kinetics may be understood in terms of deviations from equilibrium.
2. Equilibrium thermochemical analyses can be used to determine the thermodynamic potential for individual and competitive reactions to occur. Equilibrium thermochemistry is used in this study to synthesize the gas-metal, gas-ceramic and gas-gas reactions that are judged most likely to be rate limiting. These calculations identify the oxidation and carburization reactions that *may and may not occur* but cannot identify the reactions that necessarily *will occur*. Comparison of

analysis results to data is necessary to discover the predominant reactants where reactions are kinetically competitive.

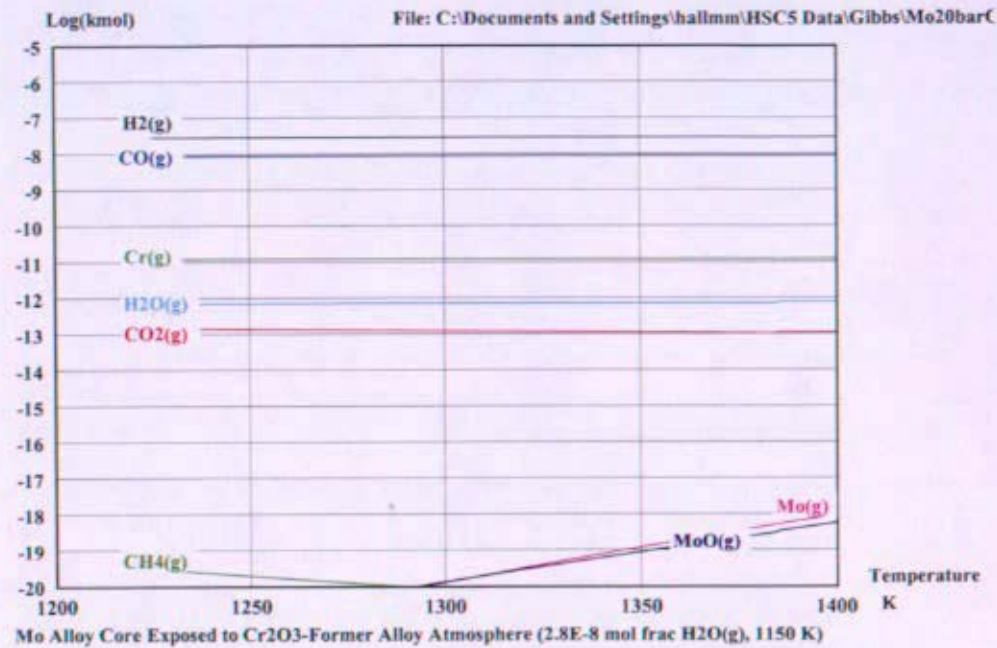


Figure 14. Reaction products formed by exposure of 1400 K Mo to an environment formed by reaction of Cr_3C_2 with the minimum water burden required to passivate chromia former superalloys at 1150 K.

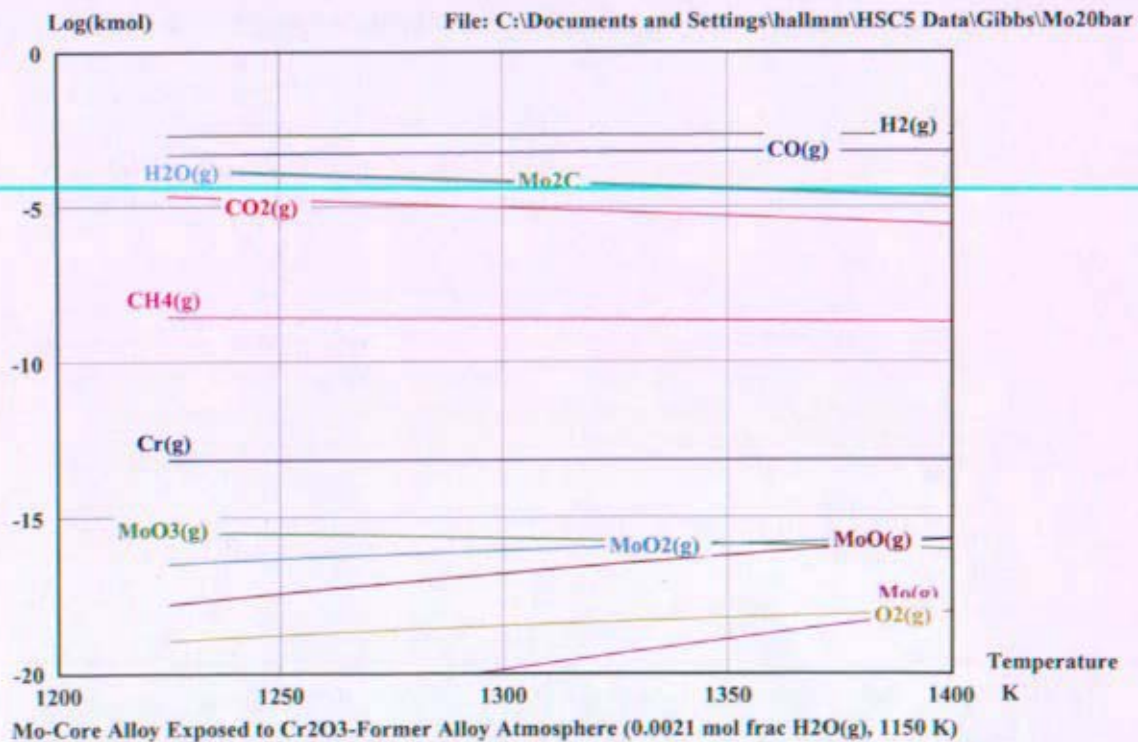


Figure 15. Reaction products formed by exposure of 1400 K Mo to an environment formed by reaction of Cr_3C_2 with the maximum allowed water burden to prevent formation of free carbon at 1150 K.

3. Whether or not an oxide or carbide forms in a given environment depends on the relative concentrations and reactivities of oxidizing and carburizing constituents in the environment. The principal reactive gases in an HTGR are water vapor (causes oxidation and decarburization), carbon monoxide (causes oxidation and carburization), methane (causes carburization) and hydrogen (causes oxide reduction). Also present are low concentrations of carbon dioxide (a product of carbon monoxide oxidation), oxygen and nitrogen (an essentially non-reactive gas due to the relatively slow kinetics of reaction).
4. The SNPP environment that will develop is not known. The concentrations of residual gases, which cannot be entirely eliminated, will depend on the initial purity of the charging gas, the effectiveness of the system de-gas procedure, the sorption and desorption rates, reactivities, carbon content and relative surface areas of the structural materials that are exposed to the closed environment and unknown concentrations of organic and inorganic impurities from various plant components. An effort is currently underway to gather the SNPP system information, solubilities and kinetic data needed to predict the oxidation and carburization potentials of the SNPP environment.
5. In order to proceed without *â priori* knowledge of the SNPP environment, we assume that adsorbed water is the primary reactive impurity in the system at reactor start up and is the source of water vapor and hydrogen. We further assume that carbon present in SiC and the superalloys, which can react with water vapor and hydrogen, is the only source of carbonaceous reactive impurities in the He/Xe working gas and that carbon is present in excess of equilibrium amounts. These assumptions are found to be reasonable when applied to an analysis of the Graphite Core-HTGR (GC-HTGR) for which the typical concentrations of impurity gases are known. The analysis proceeds by calculating the concentrations of reaction products as a function one independent variable, the assumed water burden present on reactor start up.
6. Use of these analysis assumptions and methods to evaluate candidate SNPP materials shows that alloys of Nb are inherently incompatible with the heat resistant superalloys. Thermochemical analyses show that Nb is both oxidized and carburized when exposed to a 1400 K environment that was established by reaction of 1150 K water vapor with chromia former superalloys having chromium carbides. Alloys of Nb and Ta require environments having very low oxidation and carburization potentials to avoid absorption of embrittling concentrations of oxygen and carbon. Use of Nb and Ta alloys in an oxidizing and carburizing environment will require development of oxidation and carburization resistant coatings.
7. The superalloys will be decarburized in an environment dominated by Nb alloys due to the inability of the superalloys to form protective chromia surface oxides in this environment. The same result is expected for Ta alloys as Ta has reactivity for oxygen and carbon similar to that of Nb. The candidate Brayton Unit iron-nickel-, cobalt- and nickel-base superalloys require environments having oxidation potentials high enough to form the Cr and Al surface oxides that protect these alloys from embrittlement due to internal oxidation and carburization and loss of creep strength due to excessive decarburization and dealloying by evaporation of low vapor pressure metals like chromium. Due to the much higher oxygen and carbon reactivities of Nb and Ta, protective Cr oxides will be reduced and carbon transferred from the superalloys to Nb and Ta alloys.
8. Analysis of the refractory metal Mo, alloys of which are being considered for core applications, shows that Mo is compatible with Brayton Unit superalloys. Molybdenum alloys are not subject to environmental embrittlement by oxygen due to very low solubility for oxygen. Moreover, at oxidation potentials where protective Cr₂O₃ oxides form on superalloys, Mo alloys form neither a surface oxide nor internal carbides.
9. Analysis of SiC, which is a candidate SNPP core material, shows that SiC is thermochemically compatible with the superalloys. SiC forms a protective surface oxide at oxidation potentials required to form protective Cr₂O₃ oxide. However, the analyses reported here show that a

rather large water burden, approaching that of a GC-HTGR, is required to stabilize Cr_2O_3 oxide in the presence of SiC. Success of the SiC option will depend on an ability to establish and maintain the oxidation potential of the SNPP at or above this level. A potential disadvantage of SiC is the possibility that free carbon "dust" may form resulting in thinning of SiC components and circulating carbon throughout the system. An issue for use of SiC that is not addressed here is the greater potential for release of fission products and the potential negative impact that these may have when transported around the SNPP system.

10. An advantage of both Mo- and SiC - core HTGRs is that both would have an environment functionally similar to that of a graphite core HTGR (GC-HTGR), which allows use of the extensive GC - HTGR database of superalloy design properties currently available. Additional testing is required to extend these superalloy databases to the lower carburization potential of a Mo - core HTGR.
11. Whatever materials are selected, development of passive methods for controlling the oxidation and carburization potentials of the SNPP environment may be necessary to assure satisfactory performance of reactor core, Brayton Unit and other power conversion plant materials.

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