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MATERIAL INTERACTIONS OF THE OPERATIONAL
SNAP-9A GENERATOR

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June 1964

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ABSTRACT (U)

The design of the SNAP-9A generator includes a number of different materials in intimate contact. This report discusses the possible interactions among the materials and their effect on the postulated re-entry breakup sequence of the generator. Experimental evidence obtained under static vacuum conditions for all of the interactions possible is presented.

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MATERIAL INTERACTIONS OF THE OPERATIONAL
SNAP-9A GENERATOR

SECTION I -- INTRODUCTION

Associated with the production and use of radioactive isotopic materials is a multiplicity of safety problems. Certain of these problems, in particular those involving contamination and disposal, are amplified when such material is fabricated into a system to be placed in orbit. These problems, especially that of contamination, become acute in the event of a mission abort, which may be defined as any major deviation from the planned launch and flight program.

The SNAP-9A power generator is an integral part of a vehicle designed to be placed in orbit. This generator uses a radioactive isotopic material as a fuel; hence, the possibility of a mission abort requires scrutiny, for uncontrolled release of the fuel could cause undesirable contamination and create radiation hazards.

The possibility of an abort on the launch pad or during the early part of the ascent, such as would lead to impact of the vehicle on the surface of the earth with subsequent uncontrolled release of the fuel, was borne in mind in the design of the generator and its fuel capsules, and in the choice of launch sites. Consequently, the probability that fuel release and contamination of a large area will result from such an early abort is relatively small.

An abort late in the ascent, such as might result from any malfunction of the second-stage propulsion unit, could place the SNAP-9A generator vehicle in a low-altitude, short-lived orbit. Such an orbit would have a lifetime of several days, after which the vehicle would re-enter the earth's atmosphere. Since radioactive decay of the fuel material during such a short flight would be negligible, it is essential that the generator break up upon re-entry and release the fuel capsules at an altitude sufficiently high that pieces from the fuel container burnup, as well as the fuel material itself, can be dispersed as small noncoalescing particles above 100,000 feet.

Upon re-entry of the SNAP-9A vehicle, the generator is supposed to undergo a passive breakup involving the following postulated sequence of events:

1. The outer magnesium alloy container vaporizes or burns up as a result of re-entry heating.
2. The "Min K" (99+% SiO₂) sloughs off.
3. The graphite heat-accumulator blocks separate to release the fuel capsules.
4. The fuel-capsule containers burn up, permitting the isotopic fuel to be exposed to burnup and dispersal. (Or, more simply, permitting the isotopic fuel to be dispersed above the minimum altitude.)

A number of questions have been posed about the possibility that material interactions could adversely affect this passive separation sequence. These questions arise from consideration of two distinctly different re-entry modes. The first is the desired, or optimum, re-entry, in which the vehicle undergoes a slow pitching motion. In this mode, the SNAP-9A generator would describe an arc, and the sides of its container would receive the major portion of the heat flux. The second is the stable, "generator-first" re-entry.¹ In this second mode, the major portion of the heat flux would be on the forward end of the SNAP-9A generator. This second re-entry mode has

¹In both modes, the SNAP-9A vehicle re-enters with the generator forward or first.

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small but finite probability of occurring, as a result of changes recently made to the design of the generator vehicle. (These changes include alteration of the ground plane and the addition of tail fins which provide the vehicle with some degree of aerodynamic stability.)

Of interest in the optimum mode of re-entry are the interactions, if any, of the magnesium alloy container and the graphite heat accumulator blocks with the "Min K" (SiO_2) insulation, and the possibility of vacuum self-welding between (1) the graphite blocks, (2) the stainless steel, and (3) the titanium and the Haynes Alloy 25. The problem in the case of a stable, generator-first re-entry involves the melting of one or several of the materials of construction, and the possible reactions of these molten materials with the materials with which they are in contact.

The orientation of the various materials used in the fabrication of the power generator, and the physical relationships among them, are shown in Figures 1 and 2. The groups of materials experimentally studied were chosen to duplicate the relationships shown. All possible interactions were investigated; however, the possible interactions will not necessarily all occur. Which ones will occur, and the extent to which they will occur, are questions heavily dependent on the temperatures of the external and internal parts of the generator. The temperatures, in turn, will be determined by the re-entry attitude. Disassembly will depend upon the comparison, with respect to the time temperature relationship during re-entry, between the aerodynamic forces tending toward breakup and the forces resulting from melting and interactions.

In considering the force which may be binding the generator together, it is necessary to take into account (1) the possibility that certain areas may have been wetted or affected by the possible material reactions, and (2) the question of whether the reacting materials remain molten. The numerical values of the forces due to the surface tension of the unreacted molten materials, and of the forces due to the reacted materials, both solid and liquid, are not known. However, the order of magnitude of the surface tension forces may be inferred from Table I.

Figure 1. External view of the SNAP-9A system attached to the Transit 5BN

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Figure 2. The SNAP-9A generator (cross section)

TABLE I
Surface Tension of Liquid Metals

Metal	Temperature (°C)	Surface Tension of Liquid Metal (dynes/cm)
Aluminum	700	900
Antimony	635	383
Bismuth	300	376
Cadmium	370	608
Copper	1140	1120
Gallium	30	735
Gold	1120	1128
Iron	1530	1700
Steel		950-1220
Lead	350	442
Magnesium	700	542
Mercury	20	476.1
Potassium	64	119
Selenium	220	105.5
Silver	995	923
Sodium	100	206.4
Thallium	313	(446)
Tin	700	538
Zinc	700	750

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SECTION II -- THEORETICAL ASPECTS

A qualitative working hypothesis for contact adhesion (self-welding) may be generalized as follows: under ultrahigh vacuum conditions resulting in ultraclean contact surfaces, solids will bond (self-weld) to each other when in contact if (1) a compound is formed between the components of the contacting materials, and/or (2) one of the materials in the contact pair is soluble, in a solid state, with the other. Other factors may augment or decrease the expected binding; however, it is believed that compound formation and solubility are the largest of the driving forces involved.

For the case in which molten materials are in contact with solid materials, quantitative predictions are difficult, in spite of the fact that the theory of surface wetting and surface tension is well developed, and in spite of the fact that a limited amount of data is available. Because molten materials, both metals and inorganic salts, are highly reactive, it is virtually impossible to apply theory and data. It is more practical, in predicting the wetting and spreading of a molten material on the surface of a solid, to consider such data on possible chemical reactions and solubilities as can be obtained from available binary phase diagrams (Table II). However, predictions of the reactions and solubilities of elements of multicomponent systems on the basis of data on the reactions and solubility of these elements in binary systems can be misleading; they should be used with care until experimental verification is possible.

TABLE II

Examples of Wetting by Liquid Metals in
a Hydrogen Atmosphere
(In all instances contact angle $\theta = 0$)

<u>Solid Substrate</u>	<u>Liquid</u>	<u>Temperature (°C)</u>	<u>Nature of Interaction</u>
γ -Fe	Al	700	Compound
γ -Fe	Sn	920-1000	Compound
Cu	Ag	850	Solid solution
Cu	Sn	400	Compound
Cu	Te	400	Compound
Cu	Zn	300	Solid solution
Ni	Ag	1000	Solid solution
Ni	Bi	310-470	Compound
Ni	Cd	400	Compound
Ni	Pb	358-700	Solid solution
Ni	Sb	700	Compound
Ni	Sn	358	Compound
Ag	Cd	400	Solid solution
Ag	Sb	550	Solid solution
Ag	Sn	300	Solid solution
Ag	Zn	500	Solid solution
Au	Ag	1000	Solid solution
Au	Cd	350	Compound
Au	Pb	400	Compound
Au	Sn	275	Compound
Au	Zn	450	Compound

Consider for the moment, with these principles in mind, the contact pairs that would be of interest, respectively, in each of the two re-entry modes discussed earlier. In the optimum re-entry attitude, with heat flux on the sides of the container, the following contact pairs are of interest:

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1. Magnesium alloy container/SiO₂ ("Min K")
2. SiO₂/graphite
3. Graphite/graphite
4. Graphite/Haynes Alloy 25
5. Stainless steel/titanium
6. Titanium/Haynes Alloy 25.

The generator container is an alloy of magnesium, thorium, and manganese. All three of these elements can form silicates, and the elements magnesium and thorium can form silicides. Temperatures near the melting point of silicon dioxide (1470° to 1710°C, depending on crystal form) are necessary before these materials will react. However, such temperatures are not compatible with the presence of the magnesium and alloying elements. Magnesium melts at 651°C, and sublimates at 621°C when heated in vacuum. The relationship of vapor pressure and altitude to the temperature at which magnesium sublimates is shown in Figure 3. It is expected that this relationship will generally hold true, except as it may be modified by the presence of the alloying agents. Thus, no reaction is expected to occur between the magnesium alloy and the "Min K."

Figure 3. Phase diagram of magnesium²

The two reported compounds between silicon and carbon are silicon carbide and disilicon carbide. The reaction $\text{SiO}_2 + 3\text{C} = \text{SiC} + 2\text{CO}$ occurs at temperatures between 1550° and 2200°C in an arc furnace. It is expected that the "Min K" and the graphite will cease to be in contact long before this temperature is reached.

Theoretically, the self-welding of graphite to graphite is not expected to occur, since there is no driving force: i.e., since the free energy of the system would not decrease. However, in one informally reported case, self-welding occurred under conditions of very high loading pressures and high temperatures. The conditions of the experiment in question are being repeated, but no quantitative numbers

²A. G. Guy, Elements of Physical Metallurgy, Addison-Wesley Pub. Co., Inc., Reading, Mass., 1951, p. 67.

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have been reported as yet, and it is believed that the binding forces will be found to be small. In any case, graphite-to-graphite self-welding is not expected to interfere with the disassembly sequence of the generator.

According to the manufacturer, Haynes Alloy 25 will not react to form carbides up to temperatures of 1038°C. In the re-entry mode under consideration, it is not likely that the Haynes Alloy 25 will reach these temperatures: the generator should break up and release the capsules before the temperature of the system is high enough for such a reaction to occur.

In the optimum re-entry mode, the contact pairs considered above will not react to interfere with generator disassembly. The fuel capsules should be released to free flight within the calculated time scales. The fuel capsules/titanium washer/stainless-steel foil system will vacuum weld and act as a single unit for a short period after the generator breakup. In the initial portion of free flight, this capsule/washer/foil system will resemble a pie plate with candles mounted normal to the surface. The lifetime of this system may be very short, and it will certainly be no longer than is necessary to burn up the stainless-steel foil. The vacuum welding that occurs will not interfere with the projected breakup sequence. It will, however, increase the time necessary for burnup of the fuel capsules over that predicted on the basis of a freely falling capsule.

At the other extreme, in the stable, generator-first re-entry mode, the problems are highly complex. First, many more contact pairs and groups must be considered. Second, the temperatures reached at the forward end of the generator are expected to be high enough to melt the various fabrication materials. And finally, a number of alternative reactions, and combinations of reactions, are possible.

Some of the possible contact pairs and groups, as shown in Figure 2, are:

1. Magnesium alloy/SiO₂ ("Min K")
2. "Min K"/stainless steel (Type 304)
3. Stainless steel/titanium
4. Stainless steel/graphite
5. Titanium/graphite
6. Titanium/Haynes Alloy 25
7. Haynes Alloy 25/graphite
8. Haynes Alloy 25/tantalum
9. Haynes Alloy 25/stainless steel/graphite.

The following is a discussion, based on theoretical considerations, of the various alternative sequential occurrences which are possible in a stable, generator-first re-entry.

Since in this mode there is no pitching motion, the forward end of the generator will be subjected to the major portion of the heat flux. The magnesium lid of the generator should be removed approximately 800 seconds before the sides of the generator container are sufficiently vaporized to release the graphite heat accumulator blocks and thereby permit the fuel capsule to fall in free flight. The forward, interior portion of the generator assembly will be subjected to the major portion of the heat before the generator breaks up. The magnesium alloy lid will sublime before the temperatures necessary for reacting with "Min K" are reached. The loss of the lid will expose the "Min K" to the heat flux and to the aerodynamic forces.

One possibility is that aerodynamic forces will cause the "Min K" to slough off; however, if the "Min K" remains, there are two other possibilities. The "Min K" could absorb the heat, protecting the substructure until the sides of the generator container vaporize and the breakup proceeds as postulated, or, if the heat flux is sufficient, the "Min K" could melt. Molten "Min K" (SiO₂) is a chemically reactive material with a low surface tension. It will react with the stainless steel, and also, under the impetus of aerodynamic forces, it will flow toward the periphery of the generator. There it could flow between the graphite segments and/or between the graphite and the fuel capsules, cementing these materials together.

If the "Min K" is sloughed off, the stainless-steel foil will be exposed to the heat flux and to the aerodynamic forces. Again in this case, there are two alternatives: (1) the stainless-steel foil may be blown off by the aerodynamic forces or (2) it may remain in position. Since it is highly probable that the stainless-steel/titanium/Haynes Alloy 25 system will be vacuum welded together, the second

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alternative is more probable. If the stainless-steel foil remains, and if the sides of the magnesium container are still intact, and if the heat flux is sufficient, the foil on the forward face of the reactor may melt. If this stainless-steel foil melts, it will react with the graphite, forming carbides, and will simultaneously dissolve the titanium washer. If a temperature gradient exists along the longitudinal axis of the generator, then the molten mass of unreacted stainless steel and carbides could solidify and cement the graphite segments together. At the same time, the material from the stainless-steel/titanium reaction could react with the Haynes Alloy 25 of the fuel capsule and cement it to the graphite segments.

Should the aerodynamic forces be sufficient to remove the stainless-steel foil, the titanium washer would be exposed. If the washer is exposed, it may be removed or it may remain, the latter being more probable because of vacuum welding between the washer and the fuel capsule. If the titanium melts, it will react with the graphite and with the Haynes Alloy 25 of the fuel capsule. These reactions could result in (1) welding together of the graphite blocks, (2) welding together of the fuel capsule and the graphite block, or (3) both (1) and (2) simultaneously. If the titanium washer remains and does not melt, then it may protect the Haynes Alloy 25 from excessive heating, so that the temperature of the fuel capsule remains below the carburization temperature of the Haynes Alloy 25. If this were to occur, and if all of the preceding materials had been removed, and if none of the reactions had taken place, and if the sides of the magnesium can had been removed, the graphite accumulator would separate and the fuel capsules could fall freely and possibly burn up.

If all of the previously discussed materials (magnesium alloy, "Min K," stainless-steel foil, and titanium washer) are removed by action of the aerodynamic forces, then the major portion of the heat flux will be directed onto the end caps of the fuel capsule. The Haynes Alloy 25 end cap should melt approximately 280 seconds before enough of the generator container side is removed to permit the graphite accumulator segments to separate. If the Haynes Alloy 25 melts, it will react rapidly with the graphite and with the tantalum liner of the fuel capsule. The ensuing reactions could lead to the cementing of the graphite blocks, and, thereby, could prevent the tantalum liner containing the isotopic fuel from slipping out of the capsule.

The possibility of the material interactions outlined above led to an experimental program to obtain evidence of the postulated reactions. The predictions made are based on studies of binary systems. In multicomponent systems, it is possible that some of the predicted reactions would not occur.

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SECTION III -- EXPERIMENTAL APPARATUS AND PROCEDURE

The majority of the experimental work described below was performed using an induction heating coil to heat a cylindrical graphite receptacle containing the materials to be studied.

In some tests, the materials were in direct contact with the graphite; in others, the materials were placed in crucibles of either alumina or beryllia and then placed in the graphite susceptor. Some of the experiments were performed with the metal in contact with a split graphite rod mounted in the graphite. For other experiments, the top of the graphite susceptor was machined into a dish shape and the susceptor placed in a quartz tube which was then evacuated to pressure of 1×10^{-6} torr. The induction coils were placed outside the quartz tube and centered with respect to the graphite receptacle. Figure 4 is a photograph of the heater in operation. Temperatures were monitored using the optical pyrometer shown on the tripod. The induction power supply can be seen in the background.

Figure 4. Photograph of the apparatus in operation

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At the outset of the heating experiment, as the temperature of the susceptor began to increase, the graphite began to outgas and the pressure in the system changed from 1×10^{-6} torr to approximately 1×10^{-4} torr. To allow the pumping system to catch up with the degassing of the graphite and maintain the pressure between 1×10^{-4} and 5×10^{-5} torr, initial heating proceeded slowly. In all cases, however, the pressure in the system approximated an altitude range of 300,000 to 400,000 feet. When the pressure in the system stabilized, the heat rate was rapidly increased until the desired temperature was attained. This temperature was held for the predetermined length of time, after which the power was shut off. The specimen was then allowed to cool to room temperature while still under vacuum.

Some early experimental work was also performed using a controlled atmosphere resistance furnace. The tests in this furnace were performed using nitrogen or helium at 1-atmosphere pressure or at a vacuum of approximately 10 microns. In the vacuum tests, the furnace was evacuated and the specimen brought to temperature. In the studies in atmospheres of nitrogen or helium, the furnace was pumped to a vacuum, after which the desired gas was admitted. The pressure in the furnace was raised to 1-atmosphere with either nitrogen or helium, and a constant flow rate of 5 cubic feet per hour was maintained. The temperature of the furnace was then raised to the melting temperature of the Haynes Alloy 25.

After the heating cycle, the specimens were first examined macroscopically, and then, after being sectioned, polished, and etched, microscopically. Other tests, including studies with an electron microprobe, were run to resolve uncertainties as to microstructure and composition. Table III is a résumé of the materials studied and the interactions noted. It includes pertinent data as to temperature, type of interaction, and length of time during which the specimen was maintained at a temperature.

TABLE III
Summary of Interaction Studies

Materials	Temperature	Time	Results
Haynes-graphite	Above and below the melting point of Haynes 25 (2450°F)	30 seconds to 4 minutes	Interaction and wetting of graphite by Haynes above melting point
Haynes-tantalum-graphite	Melting point of Haynes 25	1 minute to 3 minutes	Progressive solution of tantalum by molten Haynes
Haynes-titanium-graphite	Below the melting point of Haynes to above the melting point of titanium (3035°F)	2 minutes	Interaction of molten Haynes with titanium and graphite
Haynes-titanium	Below the melting point of Haynes to above the melting point of titanium	2 minutes	Rapid wetting of titanium by molten Haynes
Titanium-graphite	Above and below the melting point of the titanium	30 seconds to 2 minutes	Very strong affinity of liquid titanium for graphite
Magnesium-"Min K"	Below and above the melting point of magnesium (1200°F)	30 seconds to 2 minutes	No interaction
304 stainless-steel + graphite	Melting point of stainless steel (2550°F)	30 seconds	Carbide formation
304 stainless-steel + titanium and graphite	Melting point of stainless steel	30 seconds	Carbide formation and solution of titanium in stainless steel

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SECTION IV -- EXPERIMENTAL DETAILS AND ANALYSIS OF REACTIONS AND RESULTS

The details of the graphite crucible, with the relative positions of the split carbon rod and metal sample shown, are illustrated graphically in Figure 5. The quartz tube which serves as a vacuum chamber is not shown, but is located between the induction coils and the graphite crucible. In most of the experiments, the graphite crucibles served as the susceptor, container, and reactant. This experimental arrangement was used for all experiments in the graphite/Haynes Alloy 25 system studies.

Figure 5. Test specimen (drawing and cross section)

Graphite/Haynes Alloy 25

Haynes Alloy 25 is a solution of chromium, nickel, tungsten, iron, and impurities, in cobalt. The alloy is mostly one phase with very little precipitate present (see Figure 18, p. 19). Except for nickel and cobalt, all the constituents of Haynes Alloy 25 form carbides.

Molten Haynes Alloy 25 reacts very rapidly with graphite, precipitating carbides of its constituents and essentially destroying the alloy. Figure 6 is a photograph of one of the split carbon rods which has reacted with Haynes Alloy 25. (This sample was inductively heated under vacuum.) The reacting alloy wets and spreads on the graphite. In this photograph, note (1) the fact that the lower portion of the metal foot is covered with graphite torn from the surface of the crucible (insert), and (2) the contact angle that the various metal lumps make with the graphite surface. The photograph in Figure 7 is typical of the materials that were melted in the resistance furnace under either helium or nitrogen atmosphere. This particular sample was melted in nitrogen atmosphere. The sample was molten for approximately 15 minutes and the reactions with the graphite was quite extensive. Figure 8 shows the lower portion of the graphite rod; the reacted metal has receded from the area of attack. This is believed to be a consequence of the presence of nitrogen and/or of the slow cooling rate. Figure 9 is a photomicrograph of the sectioned and polished Haynes Alloy 25 lump that reacted with the graphite. Actually, the material is no longer Haynes Alloy 25, but rather a conglomerated mass of precipitated carbides and carbon dissolved in cobalt.

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(Inset shows portion of graphite crucible where large metal "foot" reacted with graphite)

Figure 6. Split graphite rod test piece after reactions with Haynes Alloy 25

Figure 7. Graphite rod reacted with Haynes Alloy 25 (resistance furnace, nitrogen atmosphere)

(Lower portion of rod shown in Figure 7 rotated 90 degrees left to right)

Figure 8.

Details of attack shown in Figure 7

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(Photomicrograph at 28X.
Etchant: 60 ml HCL, 15 ml HNO₃)

Figure 9.

Metallographic section of Haynes Alloy 25 reacted with graphite

Graphite/Haynes Alloy 25/Titanium

Three groups of experiments were performed using combinations of titanium, graphite, and Haynes Alloy 25. Each group consisted of a series of experiments in which the material combinations were inductively heated under vacuum to just below or just above the melting point of the solid of lowest melting temperature.

Graphite/Titanium -- The first series of experiments concerned the interaction of titanium metal with graphite. In the first experiment of this series, high purity titanium (0.3 to 0.4 gm) was placed in the dished graphite susceptor crucible and heated to just below the melting temperature of the titanium. The crystals were macroscopically examined after the heating and cooling cycle was completed. The upper surface of the titanium crystals had darkened considerably, while the surface in contact with the graphite remained bright (Figure 10). Two of the crystals appeared to have welded together; however, these fell apart when an attempt was made to remove them from the crucible. The graphite crucible showed no evidence of reaction at the point of contact with the crystals. In the second experiment, the system was heated to the melting temperature of the titanium. As soon as its melting temperature was reached, the titanium penetrated the graphite susceptor. Figure 11 is a top view photograph of the crucible after the titanium melted. Figure 12 is a cross-section photograph of the same crucible. Note how completely the titanium has penetrated the graphite.

Figure 10.

Titanium crystals heated in contact with graphite (unreacted)

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Figure 11. Top view of graphite crucible in which titanium has been melted

Figure 12. Cross section of graphite crucible shown in Figure 11

In the third experiment, as already shown in Figure 5 (p. 14), a split graphite rod was mounted in a hole in the graphite receptor. The rod was surrounded with titanium crystals, and the whole system heated to the melting temperature of titanium in 5 minutes and held there for 30 seconds under a vacuum of 10^{-4} torr. The titanium penetrated the graphite as soon as it melted. As expected, the split carbon rods were welded or cemented together as well as to the graphite crucible.

Haynes Alloy 25/Titanium -- The second group of experiments investigated the interaction of Haynes Alloy 25 with titanium. In these experiments, the graphite susceptor was machined to hold a small beryllium oxide crucible. The materials under study were placed in the beryllia crucible and inductively heated in vacuum (1) to below the melting point of the Haynes Alloy 25, (2) to the melting point of the Haynes Alloy 25, and (3) to the melting point of the titanium.

In the first experiment the exposed surfaces of the materials darkened, but no other interaction was observed. The materials did not adhere to each other (Figure 13); however, no real significance can be attached to the absence of reaction. No special efforts were expended to assure ultraclean surfaces prior to heating or to maintain ultrahigh vacuum. The vacuum varied from 2×10^{-6} torr at the beginning of the heating, to as low as 9×10^{-4} torr at 950°C , and finally to 5×10^{-5} torr at 1043°C --at which point the heater was turned off.

(Heated to 1043°C in beryllium crucible. The light portion on the Haynes Alloy 25 is the area of contact with the titanium crystal.)

Figure 13.

Titanium crystal and Haynes Alloy 25 heated to below melting

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In the second experiment, the maximum temperature indicated by the optical pyrometer was 1285°C.³ This is slightly below the lowest quoted melting temperature for the Haynes Alloy 25 (melting range, 1329° to 1410°C). However, visual observation showed that reaction had occurred and the power to the induction heater was turned off. Figure 14 is a macrophotograph of the beryllia crucible and the reaction product. Compare the microstructure of the reaction mass (Figures 15 and 16) with that of unreacted titanium (Figure 17) and unreacted Haynes Alloy 25 (Figure 18). Not all of the Haynes Alloy 25 reacted; however, all of the titanium dissolved and was no longer recognizable.

(Heated to the melting temperature of the Haynes Alloy 25)

Figure 14.

Beryllium oxide crucible with reacted mass of titanium/Haynes Alloy 25

(100X; Etchant: 3 ml HF, 6 ml HNO₃)

Figure 15. Microstructure of reacted mass of titanium/Haynes Alloy 25

³Temperatures quoted have not been corrected for emissivity of the material or for the quartz container.

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(Etchant: 3 ml HF and 6 ml HNO_3)

Figure 16. Unreacted Haynes Alloy 25 in the reacted mass of titanium/Haynes Alloy 25

(110X; Etchant: 3 ml HF, 6 ml HNO_3)

Figure 17. Microstructure of the surface of a sample titanium crystal before exposure or reaction

(100X; Etchant: 60 ml HCL, 15 ml HNO_3)

Figure 18. Microstructure of the surface of a Haynes Alloy 25 sample before exposure or reaction

Studies at the melt temperature of the titanium provided no additional information. The materials reacted when the melt range of the Haynes Alloy 25 was reached. When the system reached an indicated 1550°C , boiling began. At 1640°C , the beryllia crucible began to bounce in the graphite susceptor and at 1745°C the molten mass rose in the beryllium oxide crucible. The power to the system was turned off at this

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point. Figure 19 is a macrograph of the beryllium oxide crucible after it was deliberately split following the cooling period. Note that the molten material reacted with the beryllia crucible, wet the surface, and penetrated the walls.

Figure 19. Beryllia crucible containing Haynes Alloy 25 and titanium heated to 1745°C (optical pyrometer)

Graphite/Haynes Alloy 25/Titanium -- The final group of this series consisted of two experiments in which all three of the components were heated together. In the first experiment, in which the highest temperature measured was below the melting temperature of the Haynes Alloy 25, there were no interactions. In the next experiment, the highest temperature reached melted the Haynes Alloy 25. The titanium dissolved in the Haynes Alloy 25, but did not react with or penetrate the graphite crucible. The Haynes Alloy 25 reacted with the graphite crucible. Macrophotographs of the reaction mass in situ and of the graphite crucible after removal of the metallic mass are shown in Figures 20 and 21. Figure 22 is a montage of a cross section of the reaction mass. The dark line across the face of the montage shows the line of traverse of the electron microprobe. The lower line is the smoothed response of the probe; set to detect titanium, the peak heights shown are relative. The dark structure at the extreme left is the typical structure or reaction product of a reaction between Haynes Alloy 25 and graphite. This area is virtually devoid of titanium. (However, if the rising base line of the electron microprobe response is considered to show general increase in titanium concentration, then there is a small amount of titanium present in this area.) The highest titanium concentration appears at the interface between the Haynes Alloy 25 graphite section area and the remainder of the sample, with two other localized concentrations. This result was unexpected, since the titanium crystals were resting on the upper surface (right in the photograph) of the Haynes Alloy 25 slug; hence, the highest concentration was expected at the right rather than at the interface of the Haynes Alloy 25 graphite reaction. Note the very close resemblance between the structures shown in the microphotographs in Figures 15 and 22.

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Figure 20. Graphite crucible with reacted mass of titanium/Haynes Alloy 25

Figure 21. Surface of graphite crucible after removal of reacted mass of titanium/Haynes Alloy 25

(Etchant: 60 ml HCL, 15 ml HNO₃)

Figure 22. Microstructure of Haynes Alloy 25/titanium/graphite with qualitative results of microprobe analysis

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Stainless Steel/Graphite/Titanium

Stainless Steel/Graphite -- The reactions of Type 304 stainless steel with graphite and titanium were studied in two experiments. The steel sample was placed in a graphite dish and heated inductively under vacuum. The steel reacted with the graphite, as expected, forming carbides, probably tying up the chromium as $(FeCr)_4C$, destroying the stainless steel as such, and depressing the melting range from that of Type 304 stainless steel to that of an iron/nickel binary mixture. Figure 23 is a macrophotograph of this system after the reaction. The reaction with the graphite occurred before the melting temperature of the stainless steel was reached, as is evidenced by the outline of the initial shape of the steel sample on the surface of the graphite crucible. This outline was not simply an etch on the surface, but was as deep as the original steel sample was thick.

Stainless Steel/Graphite/Titanium -- In the second experiment, several crystals of titanium were placed on top of the stainless-steel sample. The titanium dissolved in the steel sample during the heating cycle; the temperature indicated by the optical pyrometer at the time of reaction was $1280^{\circ}C$. Assuming that the chromium has been bound in the formation of the $(FeCr)_4C$, the titanium can react with and form a number of eutectic mixtures and compounds with iron and nickel. Figure 24 is a photomicrograph of the system after reaction. Following the solution of the titanium, it appears that the reaction product solidified at the temperature at which the reaction occurred.

Figure 23. Graphite crucible reacted with Type 304 stainless steel

Figure 24. Graphite crucible with reacted stainless steel/titanium

Magnesium/"Min K"

A number of attempts were made to study the system "Min K"/magnesium at the melting point of the magnesium. Most of these were failures, in the sense that the magnesium distilled out of the alumina crucible and onto the walls of the vacuum chamber. The "Min K" from these experiments was examined in minute detail and there was no evidence of any reaction. In the final experiment, a single large piece of magnesium was placed in the crucible and covered with an excess of "Min K." The system was heated until the temperatures indicated by the pyrometer was above the published melting temperature of the magnesium ($651^{\circ}C$). Some metal distilled onto the walls of the vacuum chamber; however, a large mass remained in the crucible and

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melted. The "Min K" insulation was carefully examined for wetting and reaction; none was evident. The magnesium mass was then broken out of the alumina crucible and its upper surface, in contact with the "Min K," was examined microscopically and compared to an untreated magnesium sample. The two surfaces were identical except for minor differences in grain size; there was no evidence of interaction between the "Min K" and the magnesium.

Graphite/Haynes Alloy 25/Tantalum

The system Haynes Alloy 25/tantalum/graphite was studied to determine the rate and extent of reaction between Haynes Alloy 25 and tantalum. The experimental system resembled that used in the Haynes Alloy 25/graphite studies; however, the split carbon rod was replaced by a tantalum segment from a flight-tested fuel capsule liner. The system Haynes Alloy 25/tantalum/graphite was heated inductively, under a vacuum of 2×10^{-5} to 5×10^{-4} torr, to the melting temperature of the Haynes Alloy 25. Three experiments were performed, with the samples held at the melt temperatures of the Haynes Alloy 25 for 1, 2, and 3 minutes, respectively. Molten Haynes Alloy 25 readily reacts with, wets the surface of, and dissolves tantalum. The rate of dissolution is approximately 4 to 5 mils per minute, confirming the results of an earlier experiment in which the graphite was not present. The presence of the graphite does not appear to materially affect the rate of solution of tantalum in Haynes Alloy 25.

Figures 25 and 26 are photographs of the system graphite/Haynes Alloy 25/tantalum. Figure 25 shows the graphite crucible with the Haynes Alloy 25 and tantalum in position (a portion of the graphite crucible has been cut away). Figure 26 shows the reacted mass removed from the graphite crucible. Note (1) the graphite adhering to the lower surfaces of the mass, (2) the wetting angle (0°) of the Haynes Alloy 25 with the surfaces of the tantalum, and (3) the fact that the Haynes Alloy 25, when molten, crept up the sample and wet and reacted with that portion of the sample that was above the surface of the melt during the heating cycle.

(After heating to the melting point of Haynes Alloy 25 - section removed)

Figure 25. Graphite crucible with Haynes Alloy 25/tantalum reaction product in situ

NOTE: 1. Contact angle of Haynes Alloy 25 on the tantalum
2. Graphite adhering to lower surfaces

Figure 26. Close-up of Haynes Alloy 25/tantalum sample removed from crucible

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Figures 27 and 28 are photographs of a montage of microphotographs of the portion above the surface, showing details of the wetting and creep. Photographs of the composite of microphotographs for each sample are shown in Figures 29 through 31. Detailed analysis of the various phases present has not been made; however, the areas of graphite/Haynes Alloy 25 interaction, and the solution of tantalum in the Haynes Alloy 25, are quite evident. The reaction between the molten Haynes Alloy 25 and the tantalum liner may not be as deleterious as initial impressions would indicate, since the reaction does not affect the possible breakup of the generator during re-entry. However, after release of the capsule, the heat flux will be on the Haynes Alloy 25. When this material melts it will begin to dissolve the tantalum and solubility will reduce the wall thickness of the tantalum liner. This could result in an early exposure of the isotopic fuel to re-entry atmosphere and burnup; however, it is believed that the rate of reaction, the mobility of the fluid, the amount of tantalum in the molten Haynes Alloy 25, and the absorptivity (or emissivity) of the reaction surface should be determined in order that the data necessary for detailed calculation may be made available.

- NOTE:
1. Wetting angle of the Haynes Alloy 25 on the tantalum
 2. Migration of the molten Haynes Alloy 25 into the outer surface of the tantalum liner section

Figure 27. Montage of a cross section of a Haynes Alloy 25/titanium sample held at the melt temperature of Haynes Alloy 25 for 1 minute

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(Etchant: 60 ml HCL, 15 ml HNO₃)

Figure 28.

Montage of a cross section of a Haynes Alloy 25/
tantalum sample held at the melt temperature of
Haynes Alloy 25 for 2 minutes

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A

B

(Tantalum liner, 20 mils thick.
"B" is an enlarged portion of
"A" as marked on "A". Etchant:
60 ml HCL, 15 ml HNO₃)

Figure 29.

Photographs of a montage of the
microstructure of a Haynes Alloy 25/
tantalum sample held at the melt
temperature of Haynes Alloy 25, in
contact with graphite, for 1 minute

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A

B

(Tantalum liner 20 mils thick.
"B" is an enlarged portion of "A"
as marked on "A". Etchant: 60 ml
HCL, 15 ml HNO₃)

Figure 30.

Photographs of a montage of the
microstructure of a Haynes Alloy 25/
tantalum sample held at the melt
temperature of Haynes Alloy 25, in
contact with graphite, for 2 minutes

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A

B

(Etchant: 60 ml HCL,
15 ml HNO₃)

Figure 31.

Photographs of a montage of the microstructure of a Haynes Alloy 25/tantalum sample held at the melt temperature of Haynes Alloy 25, in contact with graphite, for 3 minutes

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SECTION V -- CONCLUSIONS

Qualitative information obtained from experiments performed under static conditions supports the conclusions drawn from theoretical considerations regarding possible material interactions and their effect on the planned passive breakup of the SNAP-9A generator:

1. If the generator re-enters the atmosphere under optimum or near optimum conditions, the planned sequence of breakup would be followed within the time scales predicted.
2. Large deviations from optimum re-entry conditions could lead to material interactions that would either interrupt the planned or predicted time sequence or completely prevent the breakup of the generator.

The extent to which any of the possible interactions affect breakup of the generator will depend on the re-entry attitude and on the pitching velocities, since the re-entry attitude defines the general temperature profile of the re-entering vehicle, and the pitching velocities define the forces available for breakup and the point or points of localization of the heat flux.

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