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ZIRCONIUM -- URANIUM COMBUSTION STUDY

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Development Directorate AIR FORCE SPECIAL WEAPONS CENTER Air Force Systems Command Kirtland Air Force Base New Mexico

Project No. 1831, Task No. 183101



(Prepared under Contract AF 29(601)-4954 by Fred E. Littman, Stanford Research Institute, Menlo Park, California)

HEADQUARTERS AIR FORCE SPECIAL WEAPONS CENTER Air Force Systems Command Kirtland Air Force Base New Mexico

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FOREWORD

Many people have contributed to this study, and this report is but the sum total of these efforts. In particular, Mr. Arthur Reed, who operated the arc image furnace, and Mr. Dante Petro, who spent many hours developing satisfactory metallographic techniques, are largely responsible for the success of this study. The importance of the Technical Meetings of the contractors of Project 1831, who, under the auspices of the AFSWC Project Officers, guided the over-all direction of these studies, can hardly be overestimated and is gratefully acknowledged.

This report is further identified by the contractor's project number, PMU-3920

ABSTRACT

This investigation was designed to provide an experimental verification of the melting and oxidation behavior of fuel rod material of the type used in SNAP-2 and SNAP-10A auxiliary power reactors. Samples of zirconiumuranium alloy, both hydrided and unhydrided, were subjected to static conditions approximating re-entry trajectories. Heat flux and pressure conditions encountered during re-entry were selected to bracket ballistic parameter ranges of $W/C_DA = 20$ to $W/C_DA = 300$ and altitude ranges of 400,000 to 150,000 feet. An arc image furnace capable of delivering up to 900 BTU/ft²sec was used as a source of radiant flux.

The results suggest that under these conditions no melting of the fuel rods is likely to occur. The material lost hydrogen and became almost completely oxidized. The resulting material, though mechanically weaker and more brittle than the starting material, was also extremely refractory owing to the high melting point of the zirconium oxide.

PUBLICATION REVIEW

This report has been reviewed and is approved.

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I INTRODUCTION

When nuclear-powered energy sources are carried in aerospace vehicles, it will be necessary to dispose of them or recover them at the end of their projected flights. Incineration during their re-entry into earth's atmosphere has been considered as a practical means of safe disposal, provided that this process results in particles small enough to remain suspended for a long time or to become sufficiently dispersed.

The present Air Force task (183101) under Project No. 1831, was devised specifically to obtain information regarding the burn-up of nuclear auxiliary power systems of the SNAP 10A and 2 type, which are compact nuclear reactors in which zirconium-uranium hydride fuel elements are used. These fuel elements (and the fission product inventory they contain at the end of the projected life) constitute the main hazard of radioactive contamination, if they should reach the earth intact.

This study was an experimental investigation of the fate of a zirconium-uranium hydride fuel elements subjected to simulated re-entry conditions of stagnation heating and pressures. Its purpose was to determine, insofar as possible with available laboratory apparatus and reasonable effort, the melting and oxidation behavior of this material, to obtain information on the particle size of the resulting products of combustion, and to establish a generalized model of the behavior of fuel rod material under re-entry conditions.

II SUMMARY

Samples of hydrided and unhydrided fuel rod material (90% Zr-10% U) were exposed to heat fluxes and pressures simulating a variety of re-entry trajectories characteristic of a satellite returning in a decaying orbit. The temperature which the sample reached under these conditions was measured and the reacted samples were examined microscopically. The results obtained suggest that hydrided zirconium-uranium fuel elements will not melt or disintegrate under these conditions. Although temperatures up to the melting point of zirconium ($\sim 3850^{\circ}$ R) were reached, incipient melting occurred under only one set of conditions. There was no evidence of formation of a liquid layer. This was probably due to the simultaneous formation of an oxide (and nitride) layer on the surface; such a layer would be highly refractory (the melting point of zirconium dioxide is $\sim 5340^{\circ}$ R).

The rapid loss of hydrogen on heating resulted in a weakening of the mechanical strength of the material, both directly through the formation of numerous cracks and fissures, and indirectly by internal oxidation, leading to the friable oxide. Oxidation was enhanced only in runs that simulated trajectories of considerable duration, when the samples remained hot and in contact with air after the evolution of hydrogen had ceased. In the case of short-term trajectories, resulting from low-altitude ejection, the evolving hydrogen effectively protected the sample from oxidation, though cracking did, of course, occur. The cracking appeared to be due to the 10% reduction in volume accompanying dehydrogenation.

The evolving hydrogen combined with oxygen to form steam, at least at higher ambient pressure. This reaction apparently took place on the surface of the sample.

III EXPERIMENTAL

At the Technical Working Group Meeting held at General Dynamics/ Astronautics in San Diego on April 3-6, 1962, the re-entry conditions were agreed on. In order to bracket reentry conditions likely to be met in practice, it was decided to investigate conditions encountered by an object re-entering in a polar orbit starting at the north pole, having a velocity of 25,680 ft/sec, a zero entering angle, and a ballistic parameter W/C_DA of 20 (rod normal to trajectory) and 300 (rod axial) lb/sq ft. It was further assumed that for each ballistic parameter the ejection of the fuel rods would occur at each of these altitudes: 400, 300, 250, 200, 150, or 100 K ft; for convenience the trajectories were designated the "a" and "b" series according to the following scheme.

Diseasi an IIA	Traje	ectory
<u>(K ft)</u>	W/C _D A=20	₩/C _D A=300
400	a	ъ
300	1a	1b
250	2a	2b
200	3a	3b
150	4a	4b
100	5a	5b

In the heat transfer calculations, a cold wall temperature of 500^{0} R was assumed. This assumption tends to make the calculated flux higher than that to be expected in practice. These trajectories and the corresponding instantaneous and accumulated flux were to be calculated by GD-Astronautics and distributed to the interested contractors.

The trajectories and heat transfer calculations became available in June and were used to determine a series of flux and pressure conditions simulating those undergone by a fuel rod ejected at several altitudes during re-entry. Samples of zirconium-uranium alloy, both hydrided and unhydrided, were then subjected to treatments simulating these conditions. The flux and pressure conditions of the two families of trajectories are shown in Fig. 1 and 2. The flux data were obtained directly from Figs. 4-6 of General Dynamics Quarterly Report No. 3. The stagnation pressures corresponding to these conditions were calculated from ambient pressures and corresponding to these conditions were calculated from ambient pressures and the corresponding Mach number according to¹

¹ M. G. Meyer, Real Gas Equilibrium Properties, Lockheed No. LMSO-TXA-111 (1961)

$$P_{st} = 1.348 M_{\infty}^2 P_{\infty}$$

where P_{st} designates the stagnation pressure, M_{∞} the ambient Mach number, P_{∞} the ambient pressure. The data, based on the ARDC Model Atmosphere (1959) are shown in Tables I and II. The "5a," "4b," and "5b" trajectories were not included in the experimental approach, because in those trajectories the total integrated heat content drops to a very low figure, as was pointed out by Philbin² in the listing shown in Table III. The amount of heat required to melt a 1.25-inch fuel element is of the order of 2,400 Btu.³

For experimental simulation of the trajectories, both the flux and the pressure values were averaged over periods of time ranging from 300 to 20 seconds, depending on the slope of the curves, as shown in the accompanying diagrams (e.g., Fig. 14). Thus the continuous curve of flux and stagnation pressure was approximated by a step function. The actual flux used was increased by 50% in most runs to compensate for two factors: the experimental conditions, which permitted the heating of one face of the sample only while at the same time it was radiating from five sides; and, offsetting this to some extent, the reduction of stagnation point flux to average flux by a factor of three due to the tumbling of the rod. Because of the small sample size, the relatively long heating periods, and the experimental arrangement, it can be assumed that the sample is at substantially uniform temperature and that all heat loss is by radiation. Under these circumstances the required arc flux is obtained by simply equating the cold wall input of the arc (thus compensating to some extent for the cold wall assumption noted above in the heat transfer calculations) to the stagnation heating of a rod rotating at more than about 1 cps.^4

$$\epsilon \dot{q}_i A_i = \frac{1}{3} q_s A_t$$

where ϵ is the emissivity, q heat flux, A area, and the subscripts i, s, and t refer to arc image input, stagnation, and total. With $\epsilon = 0.8^5$ and our particular wedge-shaped sample (A_i/A_t = 0.275), we find q_i = 1.5 q_s, requiring a 50% increase in incident flux.

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² Philbin, E. J. Re-entry and Disposal Phenomena for NAPS - Third Quarterly Report. (AE62-0683) General Dynamics/Astronautics

³ Zlotnick, M., et al. Investigation of Re-entry Destruction of NAPS (AFSWC-TR-61-69), AVCO Corp., Wilmington, Mass. (RAD-TR-61-28)

⁴ Philbin, E. J. Re-entry and Disposal Phenomena for NAPS - Fifth Quarterly Report. (AE 63-0011) General Dynamics/Astronautics.

⁵ Furman, S. C. and P. A. McManus. Metal Water Reactions. IX. (GEAP-3338) Vallecitos Atomic Lab., General Electric Co.

Table I

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FLUX AND PRESSURE CONDITIONS FOR SELECTED TRAJECTORIES NORMAL TRAJECTORIES($W/C_D = 20$)

Altitude (kilofeet) † Nominal Actual		Time From Re-Entry (sec)	Time From Ejection (sec)	Mach No.	Press Ambi (inc Hg	ure,* ent hes)	Press Stagr (ind Hg	sure,* nation ches c)	Flux, Stagna- tion (Btu/ ft ² -sec)
			"a" T	rajectory	7				
400 350 300 250 200 150 100 50	400 347 297 248 197 140 92 51	0 800 1150 1325 1400 1450 1500 1575	0 800 1150 1325 1400 1450 1500 1575	17.0 26.0 29.9 26.2 16.3 6.5 1.2 0.3	5.3 2.6 3.6 6.9 7.5 6.3 4.7 3.3	-7 -6 -5 -4 -3 -2 -1 0	2.0 2.6 4.4 6.4 2.7 3.6	-4 -3 -2 -1 0 -	5 20 75 270 240 20 0 -
			"1a"	Trajector	y				
300 250 200 150 100 50	298 250 200 150 98 50	1300 1575 1650 1695 1745 1830	0 275 350 395 445 530	30.2 26.3 16.8 7.9 1.6 0.4	3.6 6.2 6.7 4.3 3.6 3.3	-5 -4 -3 -2 -1 0	4,5 5.7 2,5 3,6 -	-2 -1 0 -	100 310 350 80 0 -
			"2a"	Trajector	у				
250 200 150 100 50	252 199 150 102 50	1825 1950 1995 2040 2130	0 125 170 215 305	28.6 16.2 7.8 1.9 0.4	8.5 6.9 4.3 3.0 3.3	-4 -3 -2 -1 0	6.1 2.5 3.6 - -	-1 0 + -	380 325 95 0 -
			"3a"	Trajector	у				
$ \frac{200}{150} \\ 100 \\ 50 $	204 151 101 50	2000 2075 2125 2210	0 75 125 210	23.5 7.6 1.8 0.4	5.6 4.2 3.1 3.3	-3 -2 -1 0	4.2 3.3 - -	0 0 - -	900 80 0 -
			''4a''	Trajector	`y				
$ \begin{array}{r} \underline{150} \\ \underline{100} \\ \underline{50} \end{array} $	158 101 51	2075 2150 2235	0 75 160	19.6 1.6 0.4	3.2 3.1 3.3	-2 -1 0	1.7 - -	1 - -	7 6 0 0 -

*Number following initial entry indicates power of ten multiplicant †Nearest datum point furnished by computer

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Table II

Altitude (kilofeet) Nominal Actual [†]		Time From Re-Entry (sec)	Time From Ejection (sec)	Mach No.	Pressure,* Ambient (inches Hg)		Pressure,* Stagnation (inches Hg)		Flux, Stagna- tion (Btu/ ft ² -sec)		
			"b" т	rajectory							
400 350 300 250 150 100 50	400 350 298 252 158 109 57	0 300 1300 2000 2075 2125 2125	0 300 1300 2000 2075 2125 2175	17.0 25.8 30.2 23.5 19.6 14.6 3 2	5.3 2.2 3.8 5.6 2.2 2.2 2.2	-7 -6 -5 -3 -2 -1	2.1 2.0 4.7 4.2 1.1 6.3 3 5	-4 -3 -2 0 1	5 10 50 470 730 480 10		
"1b" Trajectory											
300 250 200 150 100 50	297 250 201 149 98 49	1150 1350 1480 1560 1610 1660	0 200 330 410 460 510	29.9 28.4 23.4 19.2 12.1 2.2	3.6 6.2 6.4 4.5 3.6 3.6	-5 -4 -3 -2 -1 0	4.3 6.7 4.7 2.2 7.1 2.3	-5 -1 0 1 1 1	50 200 490 780 400 10		
			"2b"	Trajector	у						
250 200 150 100 50	249 199 149 100 49	1325 1400 1450 1490 1540	0 75 125 165 215	26.3 22.0 19.1 13.3 2.2	6.9 6.9 4.5 3.3 3.6	-4 -3 -2 -1 0	6.5 4.5 2.2 7.8 2.3	-1 0 1 1	160 430 800 450 10		
	"3b" Trajectory										
200 150 100 50	197 152 102 51	1400 1435 1465 1505	0 35 65 105	16.3 14.9 12.6 2.7	7.5 40 3.0 3.3	-3 -2 -1 0	2.7 1.2 6.4 3.2	0 1 1 1	180 320 440 5		

FLUX AND PRESSURE CONDITIONS FOR SELECTED TRAJECTORIES AXIAL TRAJECTORIES ($W/C_nA = 300$)

*Number following initial entry indicates power of ten multiplicant

†Nearest datum point furnished by computer

Total Heating Total Heating Trajectory Trajectory (Btu/ft^2) (Btu/ft²) 60 x 10³ 202×10^3 я b 61 x 10³ 153×10^3 1a 1b 42×10^{3} 89 x 10³ 2a 2b 22 x 10³ 3ъ 38 29 x 10³ 9 x 10³ 4a 4b 3×10^{3} 2 x 10³ 5b 5a 0.1 x 10³

TOTAL STAGNATION POINT HEATING OF EXPOSED FUEL ELEMENTS

Table III

A. Equipment and Procedure

1. Arc Image Furnace

The major piece of equipment used is an arc image furnace equipped with 14-inch elliptical mirrors. This apparatus, a lineal descendant of the mirror system used by Archimedes in 212 B.C. to burn the ships of the invading Roman navy at Syracuse, consists of a carbon arc system as a source of energy, and a pair of elliptical mirrors whose function is to collect and transfer this energy to another plane, the "furnace" area. Flux densities of the order of 900 Btu/ft²-sec can easily be obtained by commercial equipment and maintained for 20-30 minutes, the lifetime of a carbon electrode. These flux densities correspond to a black body temperature of over $5000^{\circ}R$. Control of the flux density can best be achieved by decreasing the area of the subtended angle of the secondary mirror by means of continuous or step diaphragms.

The sample rested on a zirconia column, which was contained in a borosilicate glass exposure chamber. For experiments at less than atmospheric pressure, a thin-walled, spherical container was used, which did not absorb a measurable amount of radiant energy. For high pressure experiments, a thick-walled glass container was used, which reduced the incident energy by about 20%. Fig. 3 shows a schematic diagram of the equipment, Fig. 4 an over-all view, Fig. 5 a detail of the adjustable diaphragm and sample container. Fig. 6 shows the high pressure container, Fig. 7 a close-up of a typical sample.

The furnace was calibrated with a radiometer developed by the Naval Defense Radiological Laboratories. This meter consists essentially of a small blackened silver disk, to which a thermocouple is connected. The disk is embedded in a water-cooled copper block, which constitutes an infinite heat sink. The temperature reached by the black target is directly proportional to the heat input; the instrument is calibrated

in terms of millivolt output per Btu/ft^2 -sec flux having a constant of 41.6 Btu/ft^2 -sec-mv. Fig. 8 shows the results obtained by using this meter for two different sets of mirrors. The new mirrors were used for all experiments requiring high flux levels.

Pressures were measured with an assortment of gages, depending on the desired range. A McLeod gage was used for pressures up to 0.1 inch, a manometer up to atmospheric pressure, and a Bourdon gage for higher pressures. Pressure, as well as flux, was changed stepwise as indicated on the diagrams representing the several trajectories.

Optical measurement of temperatures proved to be quite difficult because reflection of the arc on the surface of the sample resulted in high readings. The problem was eventually solved by using a Micro Pyrometer, which was focused on a hole 0.025-inch in diameter and 0.150-inch deep, drilled into the side of the sample. The hole can be seen clearly in Fig. 7. Drilling holes into the hydrided samples was exceedingly difficult, but was eventually accomplished with a supersonic drill. The temperature readings thus obtained closely approximate black body temperatures, since it can be shown that an apparent emissivity of 0.99 results with a hole having a depth of 2.6 diameters and a surface emissivity of $0.75.^{6}$

2. Experimental Procedures

Except for a few early runs in which cubic samples were used, the samples were wedge-shaped, approximately 3/16 inch on edge. Two types of material were used: zirconium-10% uranium alloy, and hydrided material of the same starting composition, containing about 1.9 atoms of hydrogen per atom of zirconium. The uranium was not enriched, so no special precautions were required in handling it. Typical photomicrographs of the starting material are shown in Fig. 9 and 10. The alloy has a very fine crystal structure, without characteristic markings. The hydrided material shows very large, banded crystals, characteristic of hydrides containing more than 1.6 atoms of hydrogen per atom of zirconium.

The alloy was easily cut and drilled by conventional cutting tools. The hydride, being hard and brittle, was cut with a wet carborundum wheel, and drilled by a supersonic drill, as mentioned previously. The filings of hydrided material were pyrophoric; the alloy did not appear to be.

Once the furnace was calibrated, the sample, resting on a zirconia column, was positioned in the exact focus by means of a jig. The pressure was then adjusted to correspond to the first step of the trajectory which was to be simulated. The proper flux was assured by a corresponding setting of the diaphragm. The arc was then lit and exposure was begun by using a shutter. Changes from one level to the

⁶ Sparrow, E. M. et al., Radiation Characteristics of Cylindrical Enclosures, J. Heat Transfer 1962, pp 73-81.

next were made as rapidly as possible by adjusting a bleeder valve and the diaphragm, without dowsing the arc.

Temperature readings were taken at frequent intervals and an average reading was recorded for each step. The sample was closely observed through the telescope of the Micro Pyrometer during the run.

When it became apparent that moisture formed and condensed on the walls of the containing vessel under certain conditions, motion pictures of this action were taken at a 64-frames/sec frequency. Kodachrome film was used for this record.

3. Metallography

Surface preparation of zirconium alloy samples is quite tricky, because it tends to "smear." A number of grinding and polishing techniques were tried. The following seemed to give the best results.

The samples were mounted in cold-set plastic, about 1/16-inch was ground off to remove strained material, and the resulting face was polished on 600-grit silicon carbide paper. The polish marks were removed with a chemical etch consisting of 47% nitric acid (65%), 5%hydrofluoric acid (48%), and 48% hydrogen peroxide (3%). The etching solution was applied with a cotton swab, and rinsed off after 10-15 seconds with distilled water. If necessary, the etching was repeated until a satisfactory surface was obtained. The sample was then rinsed in methanol and dried with warm air. It had to be examined within 1-2 hours, since an oxide coating seemed to form overnight. Typical metallographs of the structure of the alloy and hydride as received are shown in Figs. 9 and 10.

In general, it appeared that the less the samples were "worked" the better the results. The surfaces obtained by the method described were reproducible.

X-ray diffraction analyses were run on a number of early samples, but did not seem to provide much information, primarily because the equipment used "sees" all of the sample at once and therefore produces an integrated result.

B. Experimental Results

1. Preliminary Runs

The uranium-zirconium alloy and hydride samples did not become available until April. Some preliminary work was therefore performed with reactor grade zirconium, of which a supply was on hand.

In the first series of experiments, a zirconium target 0.030-inch thick and 3/8-inch in diameter was used. The sample was placed in the focus of the arc-image furnace at an incident flux level of the order

of 250-300 Btu/ft²-sec for about 30 seconds. The first sample was heated in air at atmospheric pressure. It became covered with a white oxide, but did not melt. Metallographic examination showed that the sample was completely converted to zirconium oxide (Fig. 11a).

Another sample was enclosed in a Vycor tube and maintained at 100 mm pressure of air. It was heated similarly. It did not melt, but became covered with a dark oxide layer. Metallographic examination showed an oxide case to the depth of about 10% of the sample and conversion of the remaining metal to β -zirconium. Extensive internal cavitation was present (Fig. 11b).

The third sample was heated in an evacuated, sealed Vycor tube. This sample partially melted; the remaining zirconium retained the structure of α -zirconium (Fig. 11c).

From the foregoing it appears that partial or complete oxidation of zirconium inhibits the melting of zirconium samples (the oxide melts $1650^{\circ}F$ higher than the metal). Oxidation also stabilizes the β -phase of zirconium. The oxidation is slow enough, moreover, that it does not become self-sustaining (at atmospheric pressure), even when the sample is heated by an incident flux of the order of 250 Btu/ft²-sec.

In another series with zirconium the targets were cubes measuring about 3/16-inch on each side. The cubes were supported on an alumina rod in air and heated at different fluxes to equilibrium temperatures. As heating progressed, the samples oxidized heavily, becoming covered with a white, nonadhering oxide layer which rapidly thickened, until the whole sample was transformed into oxide.

The first tests of zirconium-uranium alloy were also performed with cubes (3/16-inch). On heating in air at atmospheric pressure, the samples showed a behavior similar to that of the zirconium samples. The unhydrided material oxidized quietly, without melting, at fluxes from 100 to 600 Btu/ft²-sec. The hydrided samples behaved similarly at low flux densities, oxidizing quietly and developing numerous fissures. At high flux densities, sudden eruptions occurred, resulting in the spilling of molten metal from the inside of the cube. The metal quickly solidified and became converted into an oxide. Marked cavitation inside the cubes was noted.

The appearance of the reacted samples at a flux of about 600 Btu/ft^2 -sec, both from the outside and after sectioning, is shown in Figs. 12 and 13.

2. Simulation of "a" trajectories

The first set of trajectories to be simulated were the "a" trajectories, corresponding to the flight path of an object having a ballistic parameter W/C_DA of 20 lb/ft². They are characteristic of the conditions encountered by a fuel rod ejected from the reactor at various altitudes in a normal orientation, that is, with their long axis perpendicular to the direction of flight.

A graph of the "a" trajectory (ejection altitude 400,000 ft) and the appearance of typical samples subjected to the conditions detailed in Table IV are shown in Figs. 14 and 15. The samples show a much larger grain structure than the starting material and a characteristic columnar structure on the oxidized surface. The hydrided sample shows no traces of the original structure and is honeycombed with voids. There is no evidence of oxidation.

In Table IV and all of the tables to follow, \bar{q}_g denotes the average stagnation flux calculated for the time interval in question (in the

Table IV

Time	Pressure	Flux (Btu/	<pre>/ft²-sec)</pre>	Temperature (⁰ R)						
(sec)	(inches Hg)	ā	ā	Alloy a-6	Alloy a-7	Hydride a-8	Hydride a-9			
300		45	68	2255	2330	2255	2200			
100	0.04	90	145	2530	2690	2330	2240			
100	0.24	200	300	3100	3190	2580	2510			
100	1.40	340	510	3390	3500	3030	3010			
100	2.40	140	210	2740	2800	2310	2320			

SIMULATION OF "a" TRAJECTORY

present case shown in Fig. 14) and \bar{q}_i the actual (with some exceptions to be noted below) input flux, increased to compensate for the difference between aerodynamic heating and radiative heating of only one surface, as discussed above.

The "la" trajectory (ejection at 300,000 ft) was run similarly. The trajectory and the appearance of the reacted samples are shown in Figs. 16 and 17. The samples are quite similar to the previous ones in appearance: again there are large crystals with a martensitic fine structure, columnar crystals around the edges of the sample, and the characteristic voids in the hydride sample. The large round cavity in the alloy sample is a drilled hole. There seems to be a thin oxide coating around the periphery of both samples. Data on simulation of the "la" trajectory are shown in Table V.

The "2a" trajectory (ejection at 250,000 ft), Fig. 18, was also run at higher flux. Again there was the same characteristic behavior of the samples: there was no melting, the temperature attained by the hydrided sample was bout 200° R lower than for the alloy, and there was a fairly rapid dehydrogenation evidenced at times by a movement of the sample on

Table V

Time (sec)	Pressure	Flu (Btu/ft	x ² -sec)	Temperature (⁰ R)								
	(inches Hg)	q _s	[†] i	Alloy 1a-5	Alloy 1a-6	Alloy 1a-7	Hydride 1a-8	Hydride 1a-9	Hydride 1 a- 10			
100	0.1	110	165	2615	2850	2940	2420	2260	2260			
100	0.2	150	225	2760	2870	3050	2580	2350	2370			
100	0.6	265	395	3050	3230	3260	3190	2740	2795			
50	2.0	365	545	3335	3445	3515	3500	2940	3050			
50	4.0	215	120	2850	3155	3190	3140	2600	2635			
50	2.6	45	25	2220	2200	2200	2260	2220	2240			
					1							

its pedestal. The photomicrographs (Fig. 19) showed very little change in the appearance of the alloy, only dehydrogenation of the hydrided sample. Data for the simulated trajectory are in Table VI.

Beginning with the "3a" trajectory, the shape of the heat pulse changes. Instead of a maximum-type curve, we now have a decaying pulse,

Table VI

SIMULATION	OF	"2a"	TRAJECTORY

Time (sec)	Pressure	Flu (Btu/ft	x ² -sec)	Temperature (^O R)						
	(inches Hg)	iq _s	ī	Alloy 2a-5	Alloy 2a-6	Alloy 2a-7	Hydride 2a-8			
50	0.8	365	545	-	3100	2970	2830			
50	1.0	360	540	-	2995	3010	2850			
50	1.9	265	395	2740	2650	2670	2615			
50	3.7	100	150	2260	2260	2240	2260			
50	1.6	10	15	2040	2095	2095	2132			

starting off at high values and dropping off rapidly. The total integrated heat content, represented by the area under the curve, decreases very rapidly as ejection occurs at successively lower altitudes. The trajectory and photomicrographs of the samples are shown in Fig. 20, 21, and 22. Figure 21 shows the appearance of a run made at \dot{q}_g . The hydride sample (No. 3a-3) is of particular interest, as it shows a transition of hydrided to unhydrided material. The "regular" sample of hydride, run at the higher flux level q_i reached a temperature almost 360° R higher. The alloy sample resembled (3a-2) run at lower flux levels, showing onlv a thin, adhering oxide layer and no profound changes in the metal. The hydrided sample showed a pattern of fine fissures, rather uniformly distributed through the sample (Fig. 22). Data are shown in Table VII.

Table VII

Time		Flux (Btu/ft ² -sec)		Temperature (⁰ R)						
(sec)	(inches Hg)	ष _s	¶i	Alloy 3a-2*	Alloy 3a- 5	Alloy 3a-6	Hydride 3a-3*	Hydride 3a-7	Hydride 3a-8	
20	4.5	700	900	3500	3480	3895	3140	3535	3515	
20	4.0	340	510	3320	2960	3010	3010	2060	2995	
20	4.4	160	240	2545	2260	2420	2400	2385	2400	
20	3.9	40	60	2420	2110	2110	2310	2145	2185	

SIMULATION OF "3a" TRAJECTORY

*Run at q_s

The "4a" trajectory has an even shorter history of exposure--only 60 seconds (Fig. 23). Starting out with a strong heat pulse at relatively high gas pressures--almost an atmosphere--results in a distinct oxide layer on the outside of the alloy sample, followed by a layer of columnar crystals (Fig. 24). The interior of the sample shows little change. They hydrided sample lost hydrogen very rapidly under these conditions and shows a regular pattern of large fissues. It is noteworthy that there is no oxide formed on this sample, suggesting that the evolving hydrogen consumes all of the oxygen available at the surface of the sample. The data for this run are shown in Table VIII.

Table VIII

Time	Pressure	Flux (Btu/ft ² -sec)		Flux Pressure (Btu/ft ² -sec) Temperature					.)
(sec)	(inches Hg)	q _s	व्	Alloy 4a-5	Alloy 4a-6	Hydride 4a-7	Hydride 4a-8		
20	20.8	480	720	3715	3680	3370	3500		
20	4.4	140	210	2330	2260	2220	2185		
20	2.4	48	72	2110	2165	2145	2110		

SIMULATION OF "4a" TRAJECTORY

During several of the hydride runs simulating "3a" and "4a" trajectories, transient accumulation of moisture was noticed on the walls of the confining vessel. The phenomenon is described in more detail in section B-4.

3. Simulation of "b" Trajectories

The heating and pressure histories of fuel elements entering the atmosphere in an axial alignment to the trajectory differ considerably from those of elements entering in a crossflow position. The much higher value of the ballistic parameter W/C_DA resulted not only in a higher heat flux but also in much higher stagnation pressures experienced by these elements. This in turn was conducive to more rapid oxidation of the samples; and since the resulting zirconium dioxide is a very refractory material, its presence counteracted the effects of the higher heat flux and prevented melting of the specimens.

Since above-atmospheric pressures were encountered in simulation of the "b" trajectory (Fig. 25), the high pressure reaction chamber (described earlier) was used. The alloy sample (Fig. 26) showed curiously little change other than a growth of grain size. A heavy oxide coating, which was apparently cohesive enough to prevent further damage, is in evidence. The hydrided material, on the other hand, was heavily oxidized, both externally and internally. This was undoubtedly due to the many fissures which developed on dehydriding, which made rapid oxidation possible. Data for this run are shown in Table IX.

The discrepancy in appearance of hydrided samples representing the "4a" and "b" trajectories, both of which were mechanically weakened by the extensive fissuring, appears to be due primarily to the duration of the run. The longer time available on the "b" trajectory permitted heavy oxidation of the dehydrided sample.

Table IX

Time	Pressure	<pre>Flux (Btu/ft²-sec)</pre>		Temperature ([°] R)			
(sec)	(inches Hg)	· q _s	q _i	Alloy b-5	Alloy b-6	Hydride b-7	Hydride b-8
400	0.2	60	90	2470	2510	2530	2470
200	0.4	130	195	2650	2650	2635	2615
200	2.4	320	480	3085	3155	3335	3245
100	18.4	640	900	3640	3695	3820	3840
100	66.0	320	480	3085	3155	3065	3140
100	66.0	320	480	3085	3155	3065	3140

SIMULATION OF "b" TRAJECTORY

The "1b" trajectory (Fig. 27) was simulated with both calculated \dot{q}_{s} flux and 50% excess. An increase of temperature of about 200°R resulted from this change. The temperature reached at maximum flux was 3800° R-sufficient to cause melting of the front surface of the alloy sample, as can be seen in Fig. 28. The sample was also heavily oxidized. The hydrided sample, though badly broken up and oxidized, did not show any evidence of melting, having reached a maximum temperature of only 3680° R. The conditions are detailed in Table X.

The "2b" trajectory, shown in Fig. 29, is characterized both by a very high peak flux and pressure, and by a relatively short duration of the heat pulse (about 200 seconds). The photomicrograph of the alloy sample shows considerable oxidation, and that of the hydrided sample shows evidence of violent degassing, such as large cavities, but only a moderate amount of oxidation. The samples did not melt. Table XI gives details of conditions.

The "3b" trajectory (Fig. 31) is characterized by relatively high air pressures and moderately high flux levels, as well as a short overall duration. This combination of factors produced very little change in the alloy sample beyond a thin oxide layer and a layer of columnar crystals. Even the hydrided sample (Fig. 32) shows only moderate disruption, though it is pocked by small voids and fissues. There is remarkably little oxidation. Conditions for this run are shown in Table XII.

Table X

Time	Pressure	Flux (Btu/ft ² -sec)		Temperature (^O R)						
(sec)	(inches Hg)	d ^a s	ā	Alloy* 1b-1	Hydride* 1b-2	Alloy* 1b-3	Hydride* 1b-4	Alloy 1b-5	Hydride 1b-7	
300	0.2	65	100	2370	23 10	2310	2350	2110	2145	
100	1.2	215	325	2470	2385	2490	2435	2760	2185	
100	4.8	415	625	3085	2980	3120	3210	3660	3280	
100	30.0	670	900	3640	3660	3640	3730	3800	3680	
100	30.0	240	360	2510	2400	2565	2760	2740	2650	

SIMULATION OF "1b" TRAJECTORY

*Run at q

4. Formation of Water

As mentioned in section B-3, condensation of steam on the walls of the containing vessel was observed in the simulation of trajectory "3a" and "4a." To explore this further, moving pictures were taken at 64 frames per second under conditions of rapid heating at near atmospheric pressure. The formation of steam, followed by subsequent condensation on the walls, is clearly shown in the film. In Fig. 33 a series of single frames from

Table XI

SIMULATION OF "2b" TRAJECTORY

(Tri ma	Processo	Flux (Btu/	ft ² -sec)	Temperature (^O R)		
(sec)	(inches Hg)	in the second se	व्	Alloy 2b-1	Alloy 2b-2	Hydride 2b-4
50	1.2	245	360	2565	2510	2290
50	8.8	450	675	3280	3320	2940
50	36.8	720	900	3515	3590	3550
50	62.0	400	600	3190	3210	3010

Table XII

Ti me	Flux (Btu/ft ² -sec)		Temperature (⁰ R)			
(sec)	(inches Hg)	ās	ā	Alloy 3b-1	Alloy 3b-2	Hydri de 3b-4
35	10.4	250	375	2470	2580	2330
35	44.0	400	600	3300	3190	3010
35	50.4	240	360	2470	2435	2330

SIMULATION OF "3b" TRAJECTORY

the film are shown. The first frame shows the sample on its pedestal at the start of irradiation (the rectangular object in the background is metal foil); in the second the glass enveloped is heavily covered with condensed steam. The following frames show gradual clearing as the envelope warmed. No flame was observed. It appears evident, however, that at least under the above-stated condition the hydrogen resulting from the decomposition of the hydride is oxidized, probably catalytically on the surface of the sample.

5. Emissivity Measurements

The emissivity of the re-entering fuel rod is of considerable interest because it has a large effect on the equilibrium temperature attained by the rod. When a radiative heat source (such as an image furnace) is used to simulate re-entry conditions, the emissivity, or more precisely its numerical equivalent, the absorptivity of the sample, also enters into the picture. As was pointed out earlier, an emissivity of 0.8 was assumed in arriving at a scaling factor of 150% for the incident flux. If both the "black body" and the "brightness" temperature of an object are known, its emissivity ϵ can be calculated⁵

$$\ln \epsilon = \frac{c_2}{\lambda} \left(\frac{1}{T} - \frac{1}{S} \right),$$

where

TDR-63-15 When an optical pyrometer is used, $\lambda = 6.5 \times 10^{-5}$ cm, since a narrow-band red filter is used in these instruments.

In our experiments the Micro Pyrometer was, as mentioned, normally focused on a small hole in the sample, which acted like a black body. It was hoped that by taking simultaneous surface temperature readings good values of emissivity could be obtained.

The procedure was in practice complicated by a number of factors, such as reflections from the reaction vessel, uneven temperature distribution, and the like. A considerable number of readings were taken under favorable circumstances and the emissivity was calculated. The average value obtained was about 0.65. However, this figure is given only for the record, it is not considered to be reliable, owing to the limitations mentioned above and in view of the General Electric results on zirconium oxide already cited and the recent results reported by Vidya⁷ for hydrided full rod material ($\epsilon \sim 0.78$ in the temperature range of interest).

⁷ Fleming, W. J. Liquid Layer Instability and Droplet Formation Study for Re-entering NAPS--Fourth Quarterly Progress Letter. Vidya Research and Development.

IV CONCLUSIONS & RECOMMENDATIONS

A considerable amount of evidence has been accumulated suggesting that hydrided zirconium-uranium alloy fuel elements will not melt and/or ablate when subjected to flux and pressure conditions simulating a variety of possible re-entry conditons. The conditions encountered seem to be right on the borderline; it is possible that in actual practice slightly higher fluxes would be produced and ablation would result. On the other hand, some of the assumptions made in the calculations of heating rates, such as cold-wall heat transfer, tend to give higher results than might be expected. In any case, it appears that there isn't a sufficient safety factor (asy, of the order of 2) to assure destruction of these elements by incineration.

It must be realized that the particular experimental approach taken deliberately neglected the effect of aerodynamic shear forces present under actual conditions. An evaluation of the possible effect of these forces, even from experiments under dynamic conditions such as a plasma jet stream, is very difficult because of the magnitude of the extrapolation required; from approximately sonic conditions to Mach 25 or 30.







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Schematic diagram of arc image furnace







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Adjustable diaphragm Figure 5



High pressure container Figure 6



Typical sample Figure 7



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P-3920-20

MATERIAL: ALLOY (90% Zr -10% U) BEFORE HEATING







Photomicrographs of heated and oxidized zirconium Figure 11



ZIRCONIUM-URANIUM ALLOY AFTER HEATING

Figure 12a



ZIRCONIUM-URANIUM HYDRIDE AFTER HEATING

Figure 13a







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ZIRCONIUM-URANIUM ALLOY AFTER HEATING (Section)

Figure 12b

Figure 13b

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Figure 14

H

Samples of material from "a" trajectory Figure 15

P-3920-18

RUN No: a-9 135µ MATERIAL: HYDRIDE TRAJECTORY: a

RUN No.: a-7 135µ **MATERIAL: ALLOY** TRAJECTORY: a



Figure 16



Samples of material from "la" trajectory Figure 17



Figure 18

Samples of material from "2a" trajectory Figure 19

P-3920-17

TRAJECTORY: 20 MATERIAL: HYDRIDE RUN No.: 20-8 135µ



TRAJECTORY: 20 MATERIAL: ALLOY RUN No.: 20-7 135 μ





Figure 20

Sample of material from "3a" trajectory at average stagnation flux Figure 21

P-3920-22



TRAJECTORY: 3a , MATERIAL: ALLOY RUN No.: 3a-2 $\frac{135\mu}{135\mu}$







Figure 23



Samples of material from "4a" trajectory Figure 24

Barry and the second second



Figure 25



Figure 26

Samples of material from "b" trajectory



Figure 27



Samples of material from "1b" trajectory Figure 28



Figure 29





Figure 31



Samples of material from "3b" trajectory Figure 32



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Formation of water Figure 33

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Air Force Special Weapons Center, Kirtland AF Base, New Marico Rpt. Ro. AFSMC-TOR-63-15. ZIROUNIUM URANIUM COMBUSTION STURY. Final Report, April 1963. (2 p. incl illus, tables. Unclassified Report This investigation was designed to provide an experimental verification of the maiting and on- dation behavior of fuel rod material of the type used in SNN-2 and SNN-20M surflaxy power re- used in SNN-2 and SNN-20M subjected to static dation behavior of fuel rod material of the type used in SNN-2 and SNN	 Hest transfer Pressure Radiation hazards Radiation and reduction Rentry Rentry River elements Btructural elements Btaufon and reduction Btaufond Research Inst Btaufond Research Inst Btaufond Research Inst Btaufond Research Inst 	Air Force Special Weapon: Center, Kirtland AF Bese, New Mexico Bese, New Mexico Bese, New Mexico Byt. No. AFSNO-TDR-63-15. ZIRCONIUM URANIUM COMBUSTION STUDY. Final Report, April 1963. (\mathcal{E} p. incl 11lus, tables. Unclassified Report for the investigation was designed to provide an experimental verification of the melting and out dation behavior of fuel rod material of the type action behavior of fuel rod material of the type wised in SNMP-2 and SNMP-10M auxiliary power re- used in SNMP-2 and SNMP-10M auxiliary power re- sectors. Sampes of zirconium-uranium alloy, both 9- hydrided and unbydrided, vere subjected to static conditions approximiting reentry trajectories. Heat flux and pressure conditions encountered during reentry were subjected to bracket ballstic parameter ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and altitude ranges of $W/C_M = 20$ to $W/C_M = 300$ and but altice ranges of $W/C_M = 20$ to $W/C_M = 300$ and but altice ranges of $W/C_M = 20$ to $W/C_M = 300$ and altice ranges of $W/C_M = 20$ to $W/C_M = 300$ and but altice ranges of $W/C_M = 20$ to $W/C_M = 300$ and altice range altice range altice range altice were altice were altice were altice were altice wer	Heat transfer Pressure Reactor fuels condation and reduc- tion Heantry Stant Trajectories simu- lation Uranium alloys ori- dation and reduc- tion Zirconium alloys ori- dation and reduc- tion ABSC Project 1831, Teak 183101 ABSC Project 1831, Teak 183101 ABSC Project 1831, Teak 183101 Contract AF 29(601)- 4954 Read E. Littman

v. SRI Project PML-3920	V. SHI Project PMI-3920
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900 RfU/ft ² -sec was used as a source of radiant flux. The results suggest that under these conditions no malting of the fuel role is likely to occur. The material lost hydrogen and become almost completely ordingad. The resulting material, though mechanically weaker and more brittle than the starting material, was also extremely re- fractory oring to the high milting point of the sirronium oride.	900 HTU/ft ² -sec was used as a source of radiant flux. The results suggest that under these conditions no malting of the fuel role is likely to occur. The material lost hydrogen and became almost completely ording and became almost the starting material, was also extremaly re- fractory owing to the high malting point of the zirconium ordine.
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900 HTU/ft ² -sec was used as a source of radiant flux. The results suggest that under these conditions no maiting of the fail rods is likely to occur. The material lost hydrogen and became almost completely condized more brittla fam. the starting material, was also extremely re- fractory owing to the high melting point of the zirconium ords.	900 HU/tt ² -sec was used as a source of radiant flux. The results suggest that under these conditions of the fuel rois is likely to occur. The material lost hydrogen and became almost completely worldized. The resulting material, though mechanically weaker and more brittle than the starting material, was also extremely re- fractory owing to the high melting point of the stirconium oxide.

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