RECEIVED BY TIC MAR 30 1973

33012

SAN-858-5

MATERIALS SURVEY FOR A RADIOISOTOPE HEAT SOURCE FOR AN ARTIFICIAL HEART DEVICE

Prepared for
U.S. ATOMIC ENERGY COMMISSION

THIS DOCUMENT CONFIRMED AS
UNCLASSIFIED
DIVISION OF CLASSIFICATION
BY I.L. Cuccchiara/Wer
DATE 6/11/73

TRW
SYSTEMS GROUP

ONE SPACE PARK • REDONDO BEACH • CALIFORNIA

S0675

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

SAN-858-5

MATERIALS SURVEY
FOR A
RADIOISOTOPE HEAT SOURCE FOR AN
ARTIFICIAL HEART DEVICE

Contract AT(04-3)-858

June 19, 1971

Prepared by:

S. E. Bramer

S. E. Bramer
Heat Source Development

Approved by:

H. Lurie

H. Lurie, Project Manager
Artificial Heart Heat Source

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

TRW
SYSTEMS GROUP

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

ABSTRACT

A comprehensive material survey was undertaken to select materials and material combinations for the artificial heart radioisotope heat source. Material property and fabrication data were obtained from a review of the pertinent literature and discussions with material suppliers and fabricators. Fuel compatibility data were obtained from Mound Laboratory, Los Alamos Scientific Laboratory and Donald W. Douglas Laboratory efforts. Other material interaction data were obtained from past heat source technology programs. The refractory metal and noble metal alloys best meet the requirements for sufficient strength to ensure fuel containment, material compatibility and oxidation and corrosion resistance. The physical and mechanical properties of these materials are given and a summary of the single-layer, two-layer, and three-layer material systems for heat sources is presented.

CONTENTS

	<u>Page</u>
1. INTRODUCTION AND SUMMARY	1
2. MATERIALS	1
2.1 Material Candidates for Heat Source Components	2
2.2 Data Affecting Material Selection	3
2.2.1 Material Availability and Cost	5
2.2.2 Property Data	5
2.2.3 Fuel Compatibility	7
2.2.4 Compatibility of Combined Material System Candidates	14
2.2.5 Heat Source/Thermal Energy Storage Material (TESM) Compatibility	27
3. FABRICATION	28
3.1 Candidate Forming Methods	30
3.2 Candidate Welding Processes and Joint Designs	31
3.2.1 Weld Process and Procedures	31
3.2.2 Comparison of Welding Processes	32
3.2.3 Joint Design	34
3.3 Cleaning	36
3.4 Heat Treating	37
4. QUALITY ASSURANCE	37
4.1 As-Received Material	38
4.2 Fabrication	38
4.3 Heat Source Assembly	39
5. SUMMARY OF DATA	39
6. RECOMMENDED CAPSULE COMBINATIONS	39
REFERENCES	47
APPENDIX - MATERIAL PROPERTIES	49

1. INTRODUCTION AND SUMMARY

The data required to select materials and material combinations for the artificial heart heat source parametric study were obtained from past experience in radioisotope heat source programs, review of the pertinent literature and discussions with material suppliers and fabricators. The parametric analyses will lead to the design of a heat source for an artificial heart device to operate in the temperature range of 1350 to 2000°F. The design, fabrication and testing of radioisotope heat sources under several RTG, heater, and heat source technology programs have resulted in a sizable data bank of material combinations to meet compatibility and safety requirements. Fuel compatibility testing at Mound Laboratory (MRC), Los Alamos Scientific Laboratory (LASL) and Donald W. Douglas Laboratory (DWDL) permitted selection of fuel containment material candidates. Material interaction data from various heat source technology programs provided an insight as to the compatibility between materials and the use of diffusion barriers. The results of limited thermal energy storage material (TESM) compatibility testing with clad candidates, at Hittman and TRW, are reported. A review of the literature, and contacts with material suppliers, provided the information on the raw material availability, cost and mechanical and physical property data, while yielding piece part fabrication and quality assurance information. An analysis of raw material availability, fuel compatibility, interactions between materials, and stress rupture data was finally used to obtain the optimum material system combinations to be used in the parametric study.

2. MATERIALS

The heat source for an artificial heart device must satisfy a large number of design constraints while utilizing near-term state-of-the-art materials and fabrication processes. Foremost among the design constraints is the requirement to provide complete isotope containment, not only during long-term exposure to vacuum or inert gas environments at operating temperatures of either 1350 or 2000°F, but also during potential accidents which could occur during, or at the end of, the proposed minimum 10-year life.

The encapsulation system of the isotopic fuel must serve three primary functions:

- 1) Provide material compatibility between the fuel form and the encapsulating member
- 2) Provide sufficient strength to ensure fuel containment
- 3) Provide oxidation and corrosion resistance.

Traditionally, for high temperature heat source applications, a separate member was used to fulfill each of these three requirements. The first two requirements have been satisfied using refractory metals which have excellent high temperature metallurgical stability in both vacuum and inert gas environments. However, this class of materials is very susceptible to oxidation at elevated temperatures and must be protected during handling and accident environments. The third requirement was, therefore, satisfied by use of noble metal clads which are oxidation and corrosion resistant but have relatively low strengths at the operating and accident temperature levels.

Discussed below are the candidate materials for the heat source concepts, data affecting material selection, fabrication, quality assurance, and recommendations for capsule material combinations to be used in the parametric study.

2.1 MATERIAL CANDIDATES FOR HEAT SOURCE CONCEPTS

The operational and accident environments to which the artificial heart heat source may be exposed dictate that high-temperature superalloys or the refractory metals should be the candidate materials of construction. The nickel- and cobalt-based superalloys, such as Inco 718, René 41 and Haynes 25, although less expensive, and having good fabricability, oxidation resistance, and lower density, cannot be considered for the heart heat source because their relatively low melting points would not satisfy the cremation requirement (TRW Report 18963-71-4 "Delineation of Selection Criteria for a Radioisotope Heat Source for an Artificial Heart Device"). In addition, prolonged operation at high temperatures would result in degradation of their metallurgical and mechanical characteristics, thus seriously impairing their ability to survive impact, fragmentation, or crushing environments. The refractory metal alloys of columbium, molybdenum, tantalum, and tungsten will satisfy

the operational and accident environmental requirements if protected by an oxidation resistant outer clad. These alloys have high melting points (approximately 4200°F for columbium, 4800°F for molybdenum, 5500°F for tantalum, and 6100°F for tungsten), excellent stability at high temperatures in inert or vacuum environments, and adequate ductility at typical impact temperatures. However, their disadvantage lies in their poor oxidation resistance at elevated temperatures and their high raw material and fabrication costs. The clad material candidates are limited to platinum-based alloys, palladium, iridium and new promising alloys with high melting points (greater than 3000°F) and long term oxidation resistance at elevated temperatures, such as the Oak Ridge Alloy (Pt-30Rh-10W) and Hafnialloy.

Capsule concepts for the artificial heart device consist of three-layer, two-layer, and single-layer construction. In the single-layer capsule, the material must obviously serve as liner, strength member, and clad. In the two-layer system, the inner layer can act as a liner/strength member and the outer layer can act as a strength member/clad. Finally, in the three-layer system, the functions of the three layers are separated.

Table 1 lists the candidate capsule materials for each of the concepts. Current radioisotope heat sources utilize tantalum-based and molybdenum-based alloys in conjunction with a noble metal clad. As discussed above, the superalloys have been eliminated from consideration. In addition, columbium alloys are not being considered because they offer no advantages over the tantalum and molybdenum alloys and no experience or data are available on their use in this application. The only tungsten alloy having been considered for very high temperature (> 2000°F) radioisotope heat source applications is the W-25Re alloy because of its considerably higher strength and good fuel compatibility. Advanced alloys such as the noble metal tungsten alloy (Oak Ridge) and the hafnium-noble metal alloy (TRW) are included as candidates because of their promise to enhance fuel containment in accident environments.

2.2 DATA AFFECTING MATERIAL SELECTION

The possible number of material combinations based on the above table is of course quite large. However, a considerable number of these systems can be eliminated on the basis of material availability and unfavorable data such as tensile strength, fuel compatibility, and inter-material compatibility.

Table 1. Candidate Capsule Materials

Single-Layer Capsule Systems	Two-Layer Capsule Systems		Three-Layer Capsule Systems		
	Liner	Clad	Liner	Strength Member	Clad
Hafn alloy	Ta-10W	Pt-20Rh	Ta-10W	T-111	Pt-20Rh
Oak Ridge Alloy	TZM	Oak Ridge Alloy	TZM	TZM	Pd
Pt-20Rh	Mo	Pd	Mo	W-25Re	Oak Ridge Alloy
Pd	Mo-50Re	Hafn alloy	Mo-50Re		
	W-25Re		W-25Re		
	Pt-20Rh		Pt-20Rh		
	Ir		Ir		

2.2.1 Material Availability and Cost

The principal elements (tantalum, molybdenum, hafnium and tungsten) of the candidate refractory metals are readily available. However, several of the material candidates for the liner and clad contain elements which are not very abundant. Specific material availability and cost must, therefore, be evaluated since the radioisotope heat source for the artificial heart device is projected to be a high production item (approximately 20,000 devices/year are currently projected). Table 2 lists the availability and projected yearly requirement of the heart heat source for rhenium and the noble metals (References 1 and 2). Only iridium would present a problem because the amount required per year is greater than the new raw material output per year.

The cost of the candidate alloys in sheet and tube form is presented in Table 3. Unalloyed molybdenum, TZM, and Ta-10W alloys in sheet and tube form are the least expensive of the candidate materials, whereas the T-111 tantalum alloy is approximately three times as expensive. The platinum alloys are the most expensive with Mo-50Re and W-25Re alloys ranking second and third, respectively. Palladium is considerably less expensive than Pt-20Rh alloy. No firm costs are currently available for the advanced Oak Ridge and Hafn alloy alloys. Estimates indicate that the Oak Ridge Alloy will be slightly higher in cost than Pt-20Rh and Hafn alloy will be slightly higher in cost than T-111.

2.2.2 Property Data

Material property data for the candidate heat source alloys (Ta-10W, T-111, Mo, TZM, Mo-50Re, W-25Re, Pt-20Rh, Pd, Ir, Hafn alloy and Oak Ridge Alloy) are presented in the appendix. A comprehensive review of the literature was made and discussions were held with material suppliers and Oak Ridge representatives to obtain the latest data. The material suppliers consisted of Wah Chang, Climax Molybdenum Co., and International Nickel Co. and Engelhard, who supplied data on tantalum alloys, molybdenum alloys, and noble metals, respectively. The properties reported include density, melting point, thermal conductivity, specific heat, thermal expansion, emissivity, tensile, and stress rupture. The tensile strength, by itself, is not used in the design of the heat source. However, when the tensile strength is considered in conjunction with the elongation (the area under the stress-strain

Table 2. Availability of Critical Raw Materials

Material	Amount in Earth's Crust (lbs)	New Raw Material* Output (lbs/year)	Amount Required** (lbs/year)
Rhenium	2,000,000	20,000 - 30,000	800
Platinum	<2,000,000	75,000 → 100,000	1700+
Rhodium	++	5000	450+
Palladium	++	35,000 → 53,000	1400
Iridium	++	1500	2600

*Current Western World output

**Based on requirements for 20,000 heat sources/year

+For Pt-20Rh alloy

++By-product of nickel-bearing and new-found platinum-bearing ores

Table 3. Candidate Raw Material Costs

Material	Cost	
	Sheet	Tube (1-inch Dia. x 0.020 wall)
Ta-10W	\$ 47/lb	\$13/linear inch
T-111	\$142/lb	\$40/linear inch
Unalloyed Mo	\$ 32/lb	\$6.50/linear inch
TZM	\$ 40/lb	\$15/linear inch
Mo-50Re	\$1420/lb	\$100/linear inch
W-25Re	\$1000/lb*	\$75/linear inch*
Palladium	\$ 42/oz	~ \$35/linear inch
Pt-20Rh	\$137/oz	\$100/linear inch
Oak Ridge Alloy	\$150/oz*	\$150/linear inch*
Hafn alloy	\$200/lb*	\$75/linear inch*

*Estimate

curve) a measure of toughness results to provide a measure of resistance to impact.

Gaps in the data exist. The principle problem is due primarily to the fact that some data have not yet been generated. Another part of the problem is due, however, to the fact that tests were conducted on materials having chemical compositions and/or heat treatments not applicable to the material form projected for this application. Shown in the data, for example, is the wide scatterband for TZM due to carbon content and stress relief temperature. These data indicate that if this alloy is selected, the material specification will have to be very specific in defining these variables. Notably lacking are reliable, long-term creep data to enable more accurate extrapolations to ten-year life for selectively vented capsules.

R. G. Donnelley of Oak Ridge provided a status of the advanced Oak Ridge family of alloys (Pt-Rh-W). Apparently, Oak Ridge is concentrating their efforts on the Pt-30Rh-10W alloy, and property data should be available in the near future. This alloy is expected to have increased strength and comparable ductility and fabricability to the Pt-26Rh-8W for which tensile data are reported. As of this date only small button ingots (40 to several hundred grams) have been produced. In the near future, large ingots will be prepared to produce sufficient sheet to initiate sheet metal fabrication and additional property studies.

Hafnialloy has been produced as 15-pound ingots which have been forged and rolled into sheet and fluted (spin forged) into tubes. The available tensile properties were obtained on Hf-3Pt alloy produced as the sheet product. The tensile values can be regarded as minimum because of the manner in which the tests were conducted (low strain rate, in air). The data generated for Hafnialloy show that its toughness at 2000°F is comparable to that of T-111. In the near future, larger ingots (up to 100 to 200 lbs) will be produced under expected AEC/SNS funding. Material will be forged and rolled into sheet, and property data such as thermal expansion and additional tensile data will be obtained. In addition, capsule forming studies will be initiated.

2.2.3 Fuel Compatibility

No fuel/liner compatibility data are available for the very high purity (0.05% or 500 ppm total impurity content) electrorefined pressed and sintered

PuO_2 (Reference 3). Data have, however, been obtained for production grade fuel (1-2% total impurity content) and purified fuel (0.23% or 2300 ppm total impurity content) with the candidate refractory metals. The fuel forms used were $^{238}\text{PuO}_2$ microspheres and pressed and sintered $^{238}\text{PuO}_2$ (PPO). Test durations were reported for considerably shorter times than 10 years, and in some cases the test temperatures were considerably higher than the 1350-2000°F operating range of the heart heat source. These data do include an evaluation of stoichiometric versus substoichiometric PuO_2 fuel (PuO_{2-x} , where $x \leq 0.2$). In addition, the data were obtained on test specimens whose fuel volume-to-container area ratio or fuel volume-to-container volume ratio may have been different from the actual hardware system, to result in different degrees of interaction observed; i.e., if the ratio were greater, the interaction would be expected to be greater. Because of these factors and others, such as temperature and time effects on material solubility in plutonia, vapor transport, diffusion, and oxide precipitation (Reference 4), complications arise in the development of satisfactory mathematical correlations that are usable for predicting other time/temperature conditions. Thus a fuel/liner compatibility program, such as that proposed in Reference 5, aimed at simulating the artificial heart heat source conditions as to type of fuel used, containment materials, exposure conditions, and geometry/volume considerations, would be advisable. However, the available compatibility data presented below, provide an insight as to fuel/liner response and justification as to candidate fuel containment materials recommended for the parametric study. The liner materials discussed are Ta-10W, unalloyed molybdenum, TZM, Mo-50Re, W-25Re, Pt-20Rh, Oak Ridge Alloy (Pt-30Rh-10W), and Hafn alloy. T-111 and Pd are not being considered as liner materials.

2.2.3.1 $\text{PuO}_2/\text{Ta-10W}$

No eutectics or other catastrophic chemical interactions occur between PuO_2 microspheres and Ta-10W at or below 2730°F. Interactions that do occur are apparently a combination of PuO_2 impurity reactions and fuel-released oxygen reactions with the Ta-10W. Interactions due to impurities would certainly be greatly reduced in the case of electrorefined fuel.

Using an empirical equation based on Mound Laboratory experiments with production grade fuel, intergranular penetration of Ta-10W by oxygen after

10 years at 1830°F would be ~ 0.007 inch (Reference 6). Intergranular attack in the 2100° to 2190°F range, however, is much more severe; examples of 0.040-inch deep intergranular attacks after one year were reported. Although somewhat surprising, Mound data seem to show better compatibility at 2730°F than at the lower temperatures. No intergranular corrosion was observed after 200 hours at that temperature, and only impurity reactions were detected at the interface.

The purified fuel (Reference 4), which has a lower impurity content than production fuel but a significantly higher impurity content than electrorefined fuel, showed improvements in chemical compatibility. This was also shown by a Mound experiment using SRP (Savannah River Product) PuO_2 with less than 0.5 wt. percent total impurities. SRP PuO_2 in contact with Ta-10W was heated for 2000 hours (~ 83 days) at 3090°F and only approximately 0.004 inch of intergranular attack was reported. Therefore, the use of very high-purity electrorefined fuel would hopefully assure minimum reaction with Ta-10W. In addition, the use of substoichiometric fuel would reduce the interaction even further because less free oxygen would be available to react with a Ta-10W liner. This has been verified by the results obtained at DWDL for the higher temperature exposures of molybdenum and tungsten alloys with purified substoichiometric fuel (Reference 4).

In view of the available compatibility data and experience from prior programs, Ta-10W is a good heat source liner candidate for the artificial heart device at temperatures up to about 1500°F. Additional high temperature data for the electrorefined stoichiometric and substoichiometric fuel are needed to determine the applicability of Ta-10W at higher temperatures.

2.2.3.2 PuO_2 /Unalloyed Molybdenum

The same comments made concerning Ta-10W can be made here regarding the lack of eutectics and catastrophic interactions between PuO_2 and Mo below 2730°F. Molybdenum does, however, react with PuO_2 in three modes according to Mound Laboratory data (Reference 6): 1) reactions with impurities, 2) mass transfer, and 3) grain boundary penetration by PuO_2 at 3090°F and above. The third reaction in Mound's words "is somewhat puzzling".

The exposure of Mo to pure PuO_2 (DWDL - Reference 4) at 3270°F for 2000 hours resulted in very limited bulk oxide precipitation, intergranular

oxide formation and surface roughening at the molybdenum/plutonia interface. The resistance of unalloyed Mo to attack above 3270°F may be related directly to the rapid loss of oxygen through the molybdenum container walls; post-test examination of the plutonia showed oxygen/plutonium ratios in the 1.70 to 1.72 range. This rapid loss of oxygen prevented any formation and transport of molybdenum oxide, but did not prevent dissolution of molybdenum in the plutonia (Reference 4). This lack of mass transfer noted at DWDL is contrary to the measurements made at MRC at slightly lower temperatures.

LASL, Reference 5, reported that a PuO_2 - 18 wt. % ThO_2 solid solution fuel was compatible (no interaction) with molybdenum for 8300 hours at 1700°F. Metallographic and electron microprobe analyses of Mo held in contact with a special high purity batch of PuO_2 (no Cr and Fe) at MRC (Reference 6) for 1000 hours at 2730°F also showed no reaction with the fuel or impurities. LASL (Reference 3) reported, however, that an electrorefined PuO_2 pellet may contain 60 ppm of Cr and 150 ppm of Fe. Production grade PuO_2 containing several times that amount of Cr and Fe embrittled surface layers formed on the Mo when exposed to 2190°F for one year (Reference 6). Again, these data show the need for experiments using fuel and conditions typical of the heart heat source.

The data reported above showed some minor interaction between Mo and PuO_2 at considerably higher temperatures than the maximum expected heart heat source temperature of 2000°F, but no interaction at 1700°F with PuO_2 - ThO_2 solid solution fuel. Unalloyed molybdenum is considered capable of containing the very high purity electrorefined PuO_2 fuel over the projected 10-year life of the heart heat source at temperatures up to 2000°F.

2.2.3.3 PuO_2 /TZM

The data presented above for unalloyed molybdenum would probably hold true for TZM in the temperature range under consideration because TZM contains less than 1% of alloying elements (0.5% Ti and 0.08% Zr). Mound Laboratory data (Reference 6) indicate no direct interaction between PuO_2 and TZM after one year at 2190°F, after 2000 hours at 2730°F and after 1000 hours at 3090°F. A small amount of mass transfer was noted, but far less than with pure molybdenum. This result is difficult to understand because of the small amount of alloying content in the TZM. It was obvious from

these experiments however, that PuO_2 cation impurities can exert a powerful influence on the reaction interface, since the TZM surface was noticeably hardened after exposure to 2190°F for one year. LASL (Reference 5) has also shown that PuO_2 is compatible with TZM for 9000 hours at 1700°F and 5000 hours at 3270°F.

TZM is a good liner candidate for a heat source using electrorefined PuO_2 at temperatures to 2000°F. If a small amount of molybdenum mass transfer occurs, it would probably be tolerable in a heat source with a nonselective vent since the entrances to the helium paths are macro-sized and three-dimensional and, therefore, not easily plugged by deposition of molybdenum. TZM should be evaluated under the worst case conditions required for the heart heat source with the high purity electrorefined fuel.

2.2.3.4 $\text{PuO}_2/\text{Mo-50Re}$

As in the case of TZM, Mound Laboratory data (Reference 6) indicate no direct reaction between PuO_2 and Mo-50Re. Only reactions with impurities resulting in embrittled surface layers were observed after 60-, 90-, and 180-day tests at 2190°F. Tests for 2000 hours at 3090°F with Savannah River Product, which has less impurities than the production grade, showed approximately 0.003 inch random penetration of PuO_2 into Mo-50Re. Mass transport of Mo was not reported.

LASL (Reference 5) reports that PuO_2 -10 wt. % ThO_2 solid solution fuel is chemically compatible (no reaction) with Mo-50Re for 4700 hours at 1700°F. It is postulated that the high purity electrorefined fuel would be no less compatible.

Mo-50Re was exposed at 3270°F for 2000 hours with production grade and purified PuO_2 at DWDL (Reference 4). Exposures to 15,000 hours are continuing at the present time. No molybdenum vapor transport was observed because oxygen loss through the container walls was too rapid to permit metal transport for an extended period. Oxide precipitation occurred in the Mo-50Re when in contact with purified PuO_2 . Plutonium was located on the surface and as a precipitate near the metal surface. No plutonium was detected in solution in the metal (detection limit 0.8% Pu); iron, chromium, nickel or cobalt were not found either. Attack by the production grade plutonia was greater than that of the purified plutonia; oxides had penetrated

deeper along grain boundaries in areas both in direct contact with the fuel and away from the fuel.

The above data suggest that Mo-50Re may be considered as a prime candidate for fuel containment at the maximum heat source operating temperature of 2000°F. Since no test data are available using high purity electrorefined fuel, a test program to verify this conclusion should be conducted.

2.2.3.5 PuO₂/W-25Re

Compatibility tests with W-25Re have been conducted for temperatures above 1830°F. MRC reports that no reaction was detected with PuO₂ for time periods up to 501 days at 1830°F (Reference 6). Some minor reaction at the surface was seen at 2200°F for 60 days. DWDL conducted numerous tests with impure and pure stoichiometric and substoichiometric fuel at 3250°F and above (Reference 4). Limited PuO₂ precipitation at the grain boundaries was noted. Pure fuel was less reactive than impure fuel, and substoichiometric fuel was less reactive than stoichiometric fuel.

2.2.3.6 PuO₂/Pt-20Rh

Based on Mound Laboratory tests, Pt-20Rh can be considered as a liner material candidate only if stoichiometric PuO₂ is used in a non-reducing atmosphere. Mound Laboratory tests at 1470°F and 1830°F for 180 days showed that stoichiometric PuO₂ is chemically compatible with Pt-20Rh. One-year tests at 2190°F with Pt-40Rh and pure Pt also demonstrated good chemical compatibility. A 90-day test at 1830°F between pure Pt and PuO_{1.8}, however, showed considerable interaction.

Compatibility experiments at LASL with production grade PuO₂ (Reference 5) showed no reaction with Pt-20Rh or Pt-40Rh for 5000 hours at 1700°F. Scheduled for test at DWDL is Pt-30Rh at 2730°F with production grade and purified PuO_{1.8} for 1000 and 5000 hours.

A Pt-20Rh liner could be used up to 2000°F in a system with a selective vent or a nonselective pressure relief device venting to air or inert gas (He or Ar). In vacuum, however, the PuO₂ will decompose to PuO_{2-x} which apparently enhances reaction with the Pt-20Rh. If venting to vacuum becomes a requirement, therefore, the introduction of an oxide, which will release oxygen at the operating temperature to retard the decomposition of PuO₂, is a possible solution.

2.2.3.7 PuO₂/Oak Ridge Alloy (Pt-30Rh-10W)

No compatibility data are available for this alloy at this time. Compatibility test programs with this material are being initiated at LASL and DWDL. No details are available as to the LASL program, but DWDL plans to evaluate this alloy in combination with production grade and purified PuO_{1.8} at 2730°F for 1000 and 5000 hours. Although compatibility data with other materials are not yet available, this advanced alloy is being considered because of its promise of enhancing fuel containment during accidents due to its very high strength at 2000°F and reported oxidation resistance. Compatibility testing with electrorefined fuel at 2000°F is recommended.

2.2.3.8 PuO₂/Hafn alloy

No PuO₂/Hafn alloy testing has been conducted; however, surface-stabilized Hafn alloy is expected to be chemically compatible with PuO₂ up to at least 2500°F. Surface-stabilized Hafn alloy is prepared by heating the Hafn alloy in air for 16-18 hours in the 2000° to 2200°F range. This stabilization process forms an extremely adherent 0.002 to 0.003-inch thick layer of HfO₂-Pt composite on the surface of the alloy. The HfO₂-Pt layer should be very stable in contact with PuO₂ or PuO_{2-x}. There are no data to suggest eutectic formation between PuO₂ and HfO₂; one would, however, expect solid state diffusion to occur between the two oxides to form solid solutions at reasonable rates above 3000°F. The oxide solid solutions should be quite compatible with the platinum-rich alloy which forms at the virgin Hafn alloy metal/HfO₂ interface.

TRW has used a PuO₂ fuel form simulant, cerium oxide (CeO₂), in compatibility tests with Hafn alloy. In one test sequence, both Hf-3Pt and Hf-10Pt were exposed to CeO₂ for 24 hours in vacuum at 2000°F. Compatibility with CeO₂ in both cases was excellent. The surface stabilization treatment given to both samples proved to be effective as no evidence of reaction was found in the two cases. The interior structure of both alloy specimens was the same as if the samples had been exposed to a vacuum environment in the absence of CeO₂.

Although compatibility data with PuO₂ are not available, this advanced alloy is being considered because of its promise of enhancing fuel containment

during accidents by providing good oxidation resistance and high toughness. Based on the promise projected for this advance alloy system, compatibility testing with electrorefined fuel at 2000°F is recommended.

2.2.4 Compatibility of Combined Material System Candidates

Contact between dissimilar materials in multilayered radioisotope heat sources raises a question of compatibility between adjacent components. The data reported below are based on compatibility testing performed under closely-controlled conditions (References 7 to 14) and predictions based on analyses of phase diagrams. A parabolic relationship was used to extrapolate data in time, and a Larson-Miller type relationship was used to correlate the thickness of the interaction zone with temperature and time. Ten-year compatibility estimates were then made on basis of these correlations. Excessive interaction between materials may necessitate the use of diffusion barriers. Data on alumina, platinized alumina, zirconia, tantalum carbide, and zirconium carbide are presented. Also, the compatibility data for the following material combinations are presented below: Ta-10W/T-111, Ta-10W/TZM, TZM/T-111, W-25Re/TZM, Pt-20Rh/Ta-10W, Pt-20Rh/T-111, Pt-20Rh/TZM, Pd/Ta-10W, Pd/T-111, and Pd/TZM. No data are presented for combinations where Mo-50Re, Oak Ridge Alloy and Hafn alloy are candidates because no testing has been performed to date. Additional material combinations of interest for which no data are available include: Mo/T-111, W-25Re/T-111, Mo/W-25Re, W-25Re/Pt-20Rh, and W-25Re/Pd. TRW is currently planning some Mo-50Re and Hafn alloy compatibility tests and tests on HfO_2 and MgO as diffusion barriers under IR&D funds.

2.2.4.1 Compatibility Data Correlation

In the past, compatibility test data have often been correlated using a parabolic relationship between diffusion depth and time for each temperature. Since material interdiffusion is a time-temperature dependent phenomenon, as is creep, an investigation was recently conducted at TRW to correlate diffusion depth with temperature as well as time by using a Larson-Miller type parameter.

$$d = f(P)$$

where

$$P = T (C + \log_{10} t)$$

such that

d = diffusion depth (mils)

T = Temperature ($^{\circ}\text{R}$)

C = Constant

t = time (hrs)

The values for the constant, C , may be determined by performing a least squares curve fit of the data available. It was found that seventy-three percent of all data points analyzed (43 points) (References 9 and 15) fell within a $\pm 15\%$ scatterband of the least squares curve. Although very long term data are not available to verify the accuracy of predictions based on these curves, it is felt that the predictions may be used as a guide to compatibility between candidate materials for the projected heat source life of 10 years.

2.2.4.2 Material Compatibility Data

The available data are summarized in Table 4, which also includes data for candidate diffusion barriers between the candidate metallic materials. The extent of the interactions was determined by microstructural, microhardness, and electron microprobe analyses, as well as analyses of phase diagrams.

Ta-10W/T-111. The thickness of the interaction zone (in this case diffusion of hafnium from the T-111 into the Ta-10W) at the interface after 271 days was only 0.0015 inch at 1550 $^{\circ}\text{F}$, and 0.002 inch at 1900 $^{\circ}\text{F}$. A Larson-Miller type relationship for this couple is shown in Figure 1 and predicts 0.0024 and 0.008 inch interaction at 1350 $^{\circ}\text{F}$ and 2000 $^{\circ}\text{F}$, respectively, in 10 years. Since both of these materials are tantalum-based alloys, no problems should be encountered after 10 years and diffusion barriers will not be required.

Ta-10W/TZM. The interaction zone for a Ta-10W/TZM couple (Reference 7) was 0.0012 inch at the interface after exposure to 2000 $^{\circ}\text{F}$ for 30 days. Using the parabolic rate relationship, it is predicted that the interaction zone after ten years' exposure at 2000 $^{\circ}\text{F}$ might be as great as 0.013 inch. Since Ta, W and Mo form solid solutions at all concentrations, no problems would be expected unless excessive void formation occurs. Excessive voids may

Table 4. Candidate Materials Interaction Data

Interface	Time at Temp (Days)	Interaction Zone Thickness (Inches)								
		1200°F	1550°F	1600°F	1900°F	2000°F	2200°F	2250°F	2500°F	2600°F
Pt-20Rh/T-111 (8)*	3	0.0001	0.0004	NT**	0.0012	NT	NT	NT	NT	NT
Pt-20Rh/T-111 (12)	3.1	NT	NT	NT	NT	NT	NT	NT	0.0049	NT
Pt-20Rh/T-111 (12)	4.2	NT	NT	NT	NT	0.0016	NT	NT	NT	NT
Pt-20Rh/T-111 (12)	16.8	NT	NT	NT	NT	0.0025	NT	0.0028	NT	NT
Pt-20Rh/T-111 (8)	30	0.0002	0.0007	NT	0.0030	NT	NT	NT	NT	NT
Pt-20Rh/T-111 (12)	42	NT	NT	NT	NT	NT	NT	0.0072	NT	NT
Pt-20Rh/T-111 (9)	271	NT	0.0021	NT	0.0070	NT	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Platinized Alumina/T-111(8)	3	No Inter-action	No Inter-action	NT	No Interaction	NT	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Platinized Alumina/T-111(12)	4.2	NT	NT	NT	NT	<0.002	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Platinized Alumina/T-111(8)	30	No Inter-action	No Inter-action	NT	0.0012	NT	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Platinized Alumina/T-111(9)	271	NT	No Inter-action	NT	0.008+0.005***	NT	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina/T-111(12)	3.1	NT	NT	NT	NT	NT	NT	NT	0.005	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina/T-111(12)	4.2	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina/T-111(12)	42	NT	NT	NT	NT	NT	NT	0.005	NT	NT
Pt-10Rh/Plasma-Arc-Sprayed Alumina/T-111(10)	30	NT	NT	NT	NT	NT	NT	NT	NT	0.008+0.005***
T-111/Ta-10W (8)	3	No Inter-action	No Inter-action	NT	0.0004	NT	NT	NT	NT	NT
T-111/Ta-10W (8)	30	No Inter-action	0.0007	NT	0.0011	NT	NT	NT	NT	NT
T-111/Ta-10W (9)	271	NT	0.0015	NT	0.0022	NT	NT	NT	NT	NT

*Numbers in parentheses are references

**NT - No test data available

***Barrier of 0.005 inch consumed while 0.008-inch is total interaction zone in the noble metal and TZM (approximately 0.004 inch in each)

Table 4 (Continued). Candidate Materials Interaction Data

Interface	Time at Temp (Days)	Interaction Zone Thickness (Inches)								
		1200°F	1550°F	1600°F	1900°F	2000°F	2200°F	2250°F	2500°F	2600°F
T-111/Mo-50Re (12)	4.2	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
Pt-20Rh/Ta-10W (10)	1.8	NT	NT	0.0004	NT	0.0008	0.0016	NT	NT	NT
Pt-20Rh/Ta-10W (10)	18	NT	NT	0.0006	NT	0.0011	0.0046	NT	NT	NT
Pt-20Rh/Ta-10W (10)	180	NT	NT	0.0021	NT	0.0055	0.0111	NT	NT	NT
Pt-10Rh/Ta-10W (12)	42	NT	NT	NT	NT	0.0041	NT	0.0046	NT	NT
Pt-20Rh/TZM (12)	0.4	NT	NT	NT	NT	NT	NT	0.0014	NT	NT
Pt-20Rh/TZM (12)	3.1	NT	NT	NT	NT	NT	NT	NT	0.0031	NT
Pt-20Rh/TZM (12)	4.2	NT	NT	NT	NT	0.0014	NT	NT	NT	NT
Pt-20Rh/TZM (12)	16.8	NT	NT	NT	NT	0.0026	NT	0.0028	NT	NT
Pt-20Rh/TZM (7)	30	NT	NT	NT	NT	0.005	NT	NT	NT	NT
Pt-20Rh/TZM (12)	42	NT	NT	NT	NT	NT	NT	0.0063	NT	NT
Pt-20Rh/TZM (7)	123	NT	NT	NT	NT	0.006	NT	NT	NT	NT
Pt-20Rh/Mo-50Re (13)	7	NT	NT	NT	NT	0.0043	NT	NT	NT	NT
Pt-20Rh/Mo-50Re (13)	70	NT	NT	NT	NT	0.0084	NT	NT	NT	NT
Ta-10W/TZM (12)	4.2	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
Ta-10W/TZM (12)	16.8	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
Ta-10W/TZM (7)	30	NT	NT	NT	NT	0.0012	NT	NT	NT	NT
T-111/TZM (8)	3	No Inter-action	No Inter-action	NT	No Interaction	NT	NT	NT	NT	NT
T-111/TZM (12)	4.2	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
T-111/TZM (12)	16.8	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
T-111/TZM (8)	30	No Inter-action	No Inter-action	NT	No Interaction	NT	NT	NT	NT	NT
T-111/TZM (9)	271	NT	NT	NT	No Interaction	NT	NT	NT	NT	NT

Table 4 (Continued). Candidate Materials Interaction Data

Interface	Time at Temp (Days)	Interaction Zone Thickness (Inches)								
		1200°F	1550°F	1600°F	1900°F	2000°F	2200°F	2250°F	2500°F	2600°F
Mo-50Re/TZM (12)	4.2	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Mo-50Re/TZM (12)	16.8	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
W-25Re/TZM (7)	30	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina/TZM(12)	0.4	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina/TZM(14)	30	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina/TZM(7)	80	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Pt-20Rh/Chemical Vapor Deposition Alumina/TZM(14)	30	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Pt-20Rh/Chemical Vapor Deposition Alumina/TZM(13)	80	NT	NT	NT	NT	No Interaction	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Zirconia/TZM(14)	30	NT	NT	NT	NT	0.0032	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Zirconia/TZM(13)	80	NT	NT	NT	NT	0.0043	NT	NT	NT	NT
Pt-20Rh/Chemical Vapor Deposition Zirconia/TZM(14)	30	NT	NT	NT	NT	0.002	NT	NT	NT	NT
Pt-20Rh/Chemical Vapor Deposition Zirconia/TZM(13)	80	NT	NT	NT	NT	0.0037	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Tantalum Carbide/TZM(14)	30	NT	NT	NT	NT	0.0043	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Tantalum Carbide/TZM(13)	80	NT	NT	NT	NT	0.0085	NT	NT	NT	NT
Pt-20Rh/Chemical Vapor Deposition Ta Carbide/TZM(14)	30	NT	NT	NT	NT	0.004	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Zirconium Carbide/TZM(14)	30	NT	NT	NT	NT	0.002	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Zirconium Carbide/TZM(13)	80	NT	NT	NT	NT	0.0098	NT	NT	NT	NT
Pt-10Rh/Plasma-Arc-Sprayed Alumina/TZM(10)	3	NT	NT	NT	NT	NT	NT	NT	NT	No Interaction
Pt-10Rh/Plasma-Arc-Sprayed Alumina/TZM(10)	30	NT	NT	NT	NT	NT	NT	NT	NT	No Interaction

Table 4 (Continued). Candidate Materials Interaction Data

Interface	Time at Temp (Days)	Interaction Zone Thickness (Inches)								
		1200°F	1550°F	1600°F	1900°F	2000°F	2200°F	2250°F	2500°F	2600°F
TZM/Plasma-Arc-Sprayed Platinized Alumina (9)	3	No Inter-action	NT	NT	NT	NT	NT	NT	NT	NT
TZM/Plasma-Arc-Sprayed Platinized Alumina (12)	3.1	NT	NT	NT	NT	NT	NT	NT	No Interaction	NT
TZM/Plasma-Arc-Sprayed Platinized Alumina (9)	30	NT	No Inter-action	NT	NT	NT	NT	NT	NT	NT
TZM/Plasma-Arc-Sprayed Platinized Alumina (9)	271	NT	NT	NT	No Interaction	NT	NT	NT	NT	NT
Pt-20Rh/Plasma-Arc-Sprayed Alumina (13)	170	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
Mo-50Re/Plasma-Arc-Sprayed Alumina (13)	7	NT	NT	NT	NT	No Inter-action	NT	NT	NT	NT
Mo-50Re/Plasma-Arc-Sprayed Alumina (13)	70	NT	NT	NT	NT	0.0002	NT	NT	NT	NT

result in reduced resistance to impact accident environments.

TZM/T-111. A 271-day test at 1900°F and a 17-day test at 2000°F in vacuum showed no interaction at the interface. This combination has promise as a chemically compatible material combination for the heart heat source.

W-25Re/TZM. Less than a 0.0001-inch interaction zone resulted when the W-25Re/TZM couple was exposed to 2000°F for 30 days (Reference 7).

Pt-20Rh/Ta-10W and Pt-10Rh/Ta-10W. The interaction zones for Pt-20Rh/Ta-10W and Pt-10Rh/Ta-10W have been measured at several temperatures. TRW (Reference 10) has determined the interaction zone for the Pt-20Rh/Ta-10W couple after exposure to 1600, 2000 and 2200°F for times up to 4320 hours. The use of the Larson-Miller type parameter (Figure 2) predicts 0.0019 and 0.019 inch interaction zones for exposures to 1350 and 2000°F, respectively, at 10 years. AI (Reference 12) has determined the interaction zone for the Pt-10Rh/Ta-10W couple after exposure to 2000 and 2250°F for 1000 hours. A plot of these data on Figure 2 shows that the values deviate approximately 25% from the "least squares fit" curve. These data indicate that the Pt-10Rh alloy has essentially the same reactivity as Pt-20Rh with Ta-10W.

Pt-20Rh/T-111. The thickness of the interaction zone between Pt-20Rh and T-111 (consisting primarily of intermetallic compounds) after 271 days was 0.002 inch at 1550°F and 0.007 inch at 1900°F (Reference 9). After 42 days at 2250°F, the thickness of the interaction zone was 0.007 inch (Reference 12). Predicted interaction zone thicknesses after ten years at 1550°F and 1900°F by the parabolic rate equation are 0.008 inch and 0.026 inch, respectively. Using the Larson-Miller type relationship (Figure 3), an interaction zone of 0.0017 and 0.032 inch at 1350 and 2000°F, respectively, is predicted after ten years. Thus, the use of a diffusion barrier is required for operation of this couple at 2000°F. Candidate barriers are discussed later in this section.

Eutectic formation at temperatures above 3000°F has been detected.

Pt-20Rh/TZM. The thickness of the interaction zone between Pt-20Rh and TZM after 30 and 123 days at 2000°F was 0.005 inch and 0.006 inch, respectively (Reference 9). After 42 days at 2250°F, the thickness of the interaction

Figure 3. Pt-20Rh/T-111 Interaction

zone was 0.0063 inch (Reference 12). The predicted interaction zone thickness after 10 years at 2000°F by the parabolic rate equation is 0.053 inch. A diffusion barrier is required for operation of this couple at 2000°F.

Pd/T-111, Pd/Ta-10W and Pd/TZM. Chemical compatibility data for Pd/T-111, Pd/Ta-10W, and Pd/TZM have not been found. A search of the Ta, Hf, and W binary phase diagrams with Pd revealed no low melting eutectics.

The Ti, Zr, and Mo binary phase diagrams with Pd indicate a Pd-Ti eutectic at 2624°F and a Pd-Zr eutectic at 1886°F. The existence of these low melting eutectics leads one to speculate that Pd would be less compatible with TZM than with T-111 at 2000°F.

Ta Alloys/Oak Ridge Alloy and Mo Alloys/Oak Ridge Alloy. Although no compatibility data are available for these candidate couples, it appears that the remarks made for Pt-20Rh as a couple with the tantalum and molybdenum alloys would be applicable since the Oak Ridge Alloy is essentially a Pt-Rh alloy with a small tungsten addition. Since the Oak Ridge Alloy may have an advantage over Pt-20Rh, such as greater impact resistance, it is being considered for the heart heat source as an advanced material system. It is recommended that compatibility tests of these couples be initiated to determine the validity of the above premise.

Ta Alloys/Hafnialloy. No compatibility and limited phase diagram data are available for the Ta-Hf binary system. Elliott (Reference 11) indicates that a eutectic temperature of about 3810°F exists for Hf-20Ta. Hf-W alloys have higher melting points (Reference 11). Thus, these couples can be considered for the artificial heart heat source based on analysis of the phase diagrams. It is recommended that compatibility testing be initiated between Ta-10W/Hafnialloy and T-111/Hafnialloy to determine if any problems exist between these couples.

Mo Alloys/Hafnialloy. No compatibility and limited phase diagram data are available for the Mo-Hf binary system. Elliott (Reference 11) indicates that a eutectic temperature of 3510°F exists for Hf-30Mo. The small amount of Ti and Zr in the TZM alloy should not present any problems below 3600°F (Reference 11). A eutectic of 3015°F exists with Hf-19Re alloy to indicate that problems may exist with the Hafnialloy/Mo-50Re couple. It is recommended that compatibility testing be initiated between TZM/Hafnialloy and Mo-50Re/Hafnialloy to determine if any problems exist between these couples.

Diffusion Barriers. Diffusion barriers may be required at the liner/strength member and strength member/clad interfaces to prevent deleterious chemical interactions. If materials compatibility data show or predict the formation of extensive brittle intermetallics at the interface, or extensive solid solution between the metal heat source components, a diffusion barrier is required. The presence of these brittle intermetallics and new solid solutions at material interfaces precludes one from using the original material engineering data to predict the behavior of the system in this new materials configuration. A system without the required diffusion barriers becomes more unpredictable with time at a rate strongly dependent on exposure temperature. Use of diffusion barriers, however, should be limited since they themselves can create chemical compatibility problems within a system; each time a diffusion barrier is used, two new material interfaces are created.

Alumina. The available plasma-arc-sprayed alumina diffusion barrier data can be summarized as follows:

- Plasma-arc-sprayed alumina is an excellent diffusion barrier for a Pt-20Rh/TZM interface in a static vacuum and an inert gas environment up to at least 2600°F (Reference 7).
- Plasma-arc-sprayed alumina has retarded the interaction between Pt-20Rh and T-111 in a static vacuum for relatively short times at 2000°F and 2250°F (Reference 12).

Platinized Alumina. Platinized alumina (TRW-100M) consists of a uniform dispersion of platinum crystallites in alumina and was originally developed as a high emittance coating. Its performance as a diffusion barrier is very similar to plasma-arc-sprayed alumina. The available plasma-arc-sprayed platinized alumina diffusion barrier data can be summarized as follows:

- Plasma-arc-sprayed platinized alumina is an excellent diffusion barrier for a Pt-20Rh/T-111 interface in a static vacuum or inert gas atmosphere up to 1550°F.
- Although plasma-arc-sprayed platinized alumina retards the interaction between Pt-20Rh and T-111, it is a poor diffusion barrier for long-term exposure in a static vacuum or inert gas atmosphere at 1900°F.
- Plasma-arc-sprayed platinized alumina is compatible with TZM in a static vacuum or inert gas atmosphere up to 1550°F. It would therefore be expected to be a good diffusion barrier for a TZM/Pt-20Rh interface under the same conditions.

Zirconia. Plasma-arc-sprayed zirconia diffusion barriers have prevented interactions at a Pt-20Rh/TZM interface in dynamic vacuum at 2000°F. However, since some interaction was seen at the Pt-20Rh/Zirconia interfaces, plasma-arc-sprayed zirconia is not considered to be a good diffusion barrier candidate for these interfaces in an artificial heart device heat source.

TaC and ZrC. TRW (References 13 and 14) investigated the use of TaC and ZrC, deposited by plasma-arc-spray and chemical vapor deposition, as diffusion barriers for the Pt-20Rh/TZM interface. Interaction zones of 8.5 and 9.8 mils for TaC and ZrC, respectively, were measured for an exposure at 2000°F for 80 days. This degree of interaction for such a short time is too excessive for the artificial heart heat source.

Surface-Oxidized Hafn alloy. Plasma-arc-sprayed diffusion barriers would probably not be required in the design concepts using Hafn alloy. An excellent 0.002- to 0.003-inch thick diffusion barrier can be formed on Hafn alloy in-situ by simply oxidizing it for 16 to 18 hours in the 2000 to 2200°F temperature range. The atomically bonded diffusion barrier consists of an outer layer of hafnia/Pt separated from the base Hafn alloy metal by a platinum-rich Pt-Hf alloy. This stable oxide diffusion barrier may be capable of retarding the interaction between Hafn alloy and tantalum alloys and Hafn alloy and molybdenum alloys discussed above. Thus it is recommended that surface-oxidized Hafn alloy be included in any materials interaction studies.

Materials Interaction Conclusions

In general, conclusions on chemical compatibility can be made as follows:

- The liner/strength member/clad combinations considered in Table 1 may interact chemically to some extent, although no low melting point eutectics or other catastrophic reactions are known or predicted.
- Combinations of materials synthesized from the same base materials rank best on an arbitrary chemical compatibility/stability scale. Examples include Ta-10W liner/T-111 strength member, and Mo-50Re liner/TZM strength member.
- Combinations of materials synthesized from different base materials rank next on the chemical compatibility/stability scale. Examples include Ta-10W liner/TZM strength member/Pt-20Rh clad, Ta-10W liner/

Hafnialloy strength member, and TZM liner/T-111 strength member/Pt-20Rh clad. Interdiffusion and intermetallic compound formation may occur in all these cases at elevated temperatures.

- Plasma-arc-sprayed alumina appears to be an effective diffusion barrier between TZM and Pt-20Rh.
- Hafnialloy can be conditioned to form its own integral diffusion barrier. If this preconditioning step were taken, it is projected that a Hafnialloy strength member would be chemically compatible with each of the candidate liner materials.

2.2.5 Heat Source/Thermal Energy Storage Material (TESM) Compatibility

In order to provide adequate thermal energy during peak demand periods, thermal energy storage materials (TESM) may be utilized in the thermal converter system. These materials are normally stable lithium, sodium, or magnesium halide salt eutectics. TESH for a Stirling cycle engine could be LiF/NaF operating at 1250°F-1300°F or LiF/MgF₂ operating at 1400°F. TESH for a Rankine cycle engine could be LiF/LiCl operating at 1000°F.

Hittman Associates (Reference 16) conducted a literature survey and a small test program to determine TESH containment for two TESH materials, i.e., LiF/NaF and LiF/LiCl. The materials evaluated for compatibility in air, which are of interest to this program, were the platinum and palladium noble metals. Table 5 gives the results of this investigation. For the times and temperatures of exposure investigated, Hittman came to the conclusion that the Pt and Pd alloys are compatible with both TESH materials.

Table 5. Molten Salt/Fuel Container Compatibility
in Air (Hittman Associates, Inc.)

Material	Stirling TESH		Rankine TESH		Results
	Temp (°F)	Time (hrs)	Temp (°F)	Time (hrs)	
Pt, Pt-10Rh	1250	257	1000	257	No attack
Pt-20Rh, Pd	1250	575	1000	575	No attack
	1250	1008	1000	1008	No attack
Pt, Pt-10Rh	2000	24	-	-	No attack
	2000	48	-	-	No attack
	2000	72	-	-	No attack
Pt, Pt-10Rh	2400	2	-	-	No attack

In an IR&D program at TRW, the compatibility of Pt-20Rh and two TRW Hafnialloy compositions was investigated in air with three candidate TESM materials. Table 6 summarizes these results. In this program some discoloration was noted on the Pt-20Rh samples exposed to 1280 and 1300°F with LiF/LiCl and LiF/NaF, respectively. At lower temperatures and higher temperatures this phenomenon was not observed. The Hf-10Pt alloy performed well at the stated test conditions, whereas the Hf-3Pt alloy showed mixed results.

These limited test results indicate that compatibility of TESM with most clad materials presents no significant problems. However, it is recommended that long term, controlled tests be initiated to determine the compatibility of TESM with the artificial heart heat source materials of construction when the selection is finalized after the parametric study. No data are apparently available for the compatibility of the candidate refractory metals with TESM should this be required during an accident environment.

3. FABRICATION

Capsules utilizing refractory metals have been successfully produced for a number of radioisotope heat source programs. Among these programs are the Transit RTG, Pioneer RHU Heater, the Pioneer RTG (which utilized a Transit capsule but of shorter length), the DART, and the Large Radioisotope Heat Source Capsule (LRHSC).

Piece parts were produced by a number of different methods. Tantalum and platinum alloy piece parts were produced from sheet material by deep drawing, hydroforming, and tube drawing, whereas molybdenum alloys were prepared by machining of bar stock. Molybdenum alloy piece parts can, however, be produced from sheet by roll-forming, radial draw-forming, spinning, hydroforming, or drop hammer and Ceco stamp forming and tube drawing as demonstrated by the fabrication of ramjet engines, rocket motors, and lifting body airframe piece parts. Joining of the refractory metal candidates by TIG and EB welding has been demonstrated.

A prime consideration in the selection of the fabrication and weld processes must be the production of hardware at a minimum cost while

Table 6. Molten Salt/Fuel Container Compatibility in Air at TRW

TRW Speciman Designation	Material	Temperature (°F)	Time (hours)	TESM	Remarks Regarding Compatibility at Test End
100	Pt-20Rh	1200	2	LiCl-LiF	Good
200		1300	2	LiF-NaF	Good
300		1200	41.3	LiCl-LiF	Good - Some Discoloration
400		1300	41.3	LiF-NaF	Good - Some Discoloration
500		2200	1.6	NaF-MgF ₂	Good
600	Pt-20Rh	2200	0.9	LiCl-LiF	Good
700		2200	0.9	LiF-NaF	Good
a	Hf-10Pt	1200	41.3	LiCl-LiF	Good
g		1200	2	LiCl-LiF	Good
h		1300	2	LiF-NaF	Good
i		1300	41.3	LiF-NaF	Good
j		2200	1.6	NaF-MgF ₂	Good
k	Hf-10Pt	2000	2	LiF-NaF	Good
l		2000	2	LiCl-LiF	Good
1-a	Hf-3Pt	1200	41.3	LiCl-LiF	Good
2-a		1300	41.3	LiF-NaF	Some Reaction
3-a		1200	2	LiCl-LiF	Good
4-a		1300	2	LiF-NaF	Good
5-a		2200	1.6	NaF-MgF ₂	Reaction
6-a	Hf-3Pt	1350	4	NaF-MgF ₂	Reaction
7-a		1570	24		
	Hf-3Pt	2000	2	NaF-MgF ₂	Reaction

followed by

satisfying the design and safety requirements. Discussed below are methods for producing the piece parts, weld joint design, applicable welding processes, and cleaning and heat treating requirements.

3.1 CANDIDATE FORMING METHODS

The method employed to produce the heat source components depends on the selected configuration. Similar techniques will produce hemispherically ended cylindrical piece parts and hemispherical piece parts from sheet material. Deep drawing, in either a mechanical press or hydropress, has been shown in past programs to be capable of producing high quality refractory metal heat source parts of this geometry. Tubular sections have been produced by drawing, spin forging or machining bar stock by gun drilling.

To improve fabricability, the Ta-10W, T-111, Mo-50Re and platinum-rhodium alloys would be obtained in the recrystallized condition, whereas the commercially pure molybdenum and TZM would be obtained in the as-worked, stress-relieved condition. The relatively poor ductility of TZM at low temperatures means that this alloy requires care in machining and in handling fabricated parts during inspection and assembly. Although the tantalum and platinum-rhodium alloys are more fabricable than the molybdenum alloys, it has been demonstrated by TRW, and others, that TZM parts can be satisfactorily formed and welded.

Depending on the material, different processes are applied to the forming of refractory metal sheet. The tantalum alloys (Ta-10W and T-111), commercially pure molybdenum and Mo-50Re may be formed with the hydropress at room temperature to produce the required shapes. In the Transit program, the tantalum alloys and Pt-20Rh piece parts were formed by deep drawing in a mechanical press. More recently, however, the hydropress process was investigated for deep-drawing Transit piece parts. This effort resulted in satisfactory piece parts which met all requirements at reduced cost because less preform stages were required and rework was reduced to meet specifications. TZM sheet, on the other hand, will have to be heated to temperatures of 200-800°F during hydropress forming operations. Such molybdenum alloys have, however, been successfully hydroformed to shapes similar to the projected shapes indicating that no undue problems are expected if one of these materials becomes a candidate (References 17 to 19).

No large quantity sheet metal forming studies have been initiated for the Oak Ridge Alloy and Hafnialloy. If forming problems exist for these alloys, because of low room-temperature ductility, the same procedures as used to successfully form TZM parts can be utilized.

At this time, it appears that a single forming step followed by a coining (re-strike) operation on a hydropress utilizing a single punch would be feasible for the tantalum, molybdenum and noble metal alloys. A heat treatment would be required between the principle forming and re-strike operations. Hopefully, as experience is gained, only a trim and weld joint edge preparation would be required. If a coining/spinning operation is required, a simple coining die would be used to form the radius of curvature whereas, a single mandrel would be used to spin-form the remainder of the part. The potential of producing a part having an adequate surface finish by the spinning method is not as great as that by the hydro-form method. A heat treat operation and possible sizing will also be required to place the material in the optimum metallurgical condition for welding. The tooling is relatively inexpensive for both forming methods. To produce the piece parts for the test program, sheet thickness will be over-size and a finish machine operation will be performed.

If the geometry selected is flat-ended, drawn tubing will be more economical than machining. In addition, for unalloyed molybdenum and TZM, drawn tubes would have the most optimum microstructure for welding. Drawn tubing, no matter which alloy is selected, would be the most economical approach to obtain heat source components.

3.2 CANDIDATE WELDING PROCESSES AND JOINT DESIGNS

Sound, reliable weldments can be consistently produced to join refractory metal components. To accomplish such welds, however, the joint design and fit-up during assembly, weld processes and procedures, and materials must be considered. These factors also affect total cost and ease of production of fueled assemblies at the fueling agency.

3.2.1 Weld Process and Procedures

To obtain optimum welds, only the tungsten inert gas (TIG) and electron beam (EB) weld processes, with no introduction of externally-fed filler metal, are being considered for this program. These processes are capable

of producing the clean, uncontaminated welds of minimum weight that have been successfully obtained in past heat source programs. Table 7 summarizes the parameters which must be considered in selecting either the TIG or EB process to produce the weld joints for the heat source assembly. It is known that the EB process is always more costly than the TIG process because of the need to provide a vacuum as well as amortizing high equipment costs. However, as can be seen in the table, the EB process is the only one that can consistently produce good weldments in molybdenum alloys.

Included in the table are other considerations for making sound weldments, such as the need for atmosphere control, start-up and shut-off slope controls, and pre-heat of the weld joint. A comparison of the candidate welding processes and weld evaluation procedures for fueled heat sources are discussed below.

3.2.2 Comparison of Welding Processes

When considering the assembly of fueled heat sources, the selection of the weld process requires additional considerations. These considerations include the flexibility of the TIG versus EB processes, the need for expensive duplicate EB facilities if burn-through should occur and the weld chamber becomes contaminated with radioactivity, complexities imposed by transporting the heat source in and out of the EB weld chamber, and the possible effect of poor heat transfer in the heat source gaps.

No clear-cut conclusions can be made regarding the flexibility of the TIG versus EB processes. For example, the TIG process allows easy positioning and alignment of the weld torch to the joint line, whereas the alignment of the focusing system of the EB welder is more difficult, especially if a full penetration angular-butt joint is required. On the other hand, EB welding not only minimizes the possibility of contamination of the material being welded, but gives less distortion and a much smaller heat-affected zone than the TIG fusion weld process. These advantages are very important where close tolerances must be held. Another consideration, although not directly related to flexibility, is that the EB process requires a high vacuum. With the fuel continuously outgassing, an extra-high capacity vacuum pumping system may be required.

Contamination of the weld chamber by vaporization of the "live" fuel is possible if a burn-through should occur in the weld. Since the TIG welding

Table 7. Summary of Weld Processing and Quality as a Function of Material

Candidate Materials	Candidate Weld Process	Weld Quality	Status	Remarks
Ta-10W	TIG	Excellent	Developed	Requires high purity inert atmosphere to prevent embrittlement
	EB	Excellent	Developed	Requires a high vacuum to operate EB gun
T-111	TIG	Excellent	Developed	Same remarks as for Ta-10W TIG weld
	EB	Excellent	Developed	Requires a high vacuum to operate EB gun
Unalloyed molybdenum	TIG	Fair	Semi-developed	Cratering during arc-strike and shut-off results in cracking. Poor joint fit-up can result in high induced stresses resulting in cracking and blow-through. Repair welds are impossible. Special weld fixtures with pre-heat are required. Controlled atmosphere is required to prevent embrittlement by oxidation.
	EB	Good	Developed	Joint fit-up critical. Defocusing of beam permits pre-heat of joint to several hundred degrees prior to welding. Sloping of power during arc-strike and shutoff minimizes cratering.
TZM	TIG	Poor	Semi-developed	More critical and same remarks apply as for unalloyed molybdenum
	EB	Good	Developed	Same as for unalloyed molybdenum
Mo-50Re	TIG	Excellent	Developed	This is the easiest molybdenum alloy to weld and requires the least care during the process.
	EB	Excellent	Developed	Same remarks as for Mo-50Re TIG weld.
Hafnialloy	TIG	Good	Promising	Small amount of effort indicates no problems in making sound weldments.
	EB	Good	Promising	
Pt-20Rh	TIG	Excellent	Developed	Possible to obtain single grains across a sheet thickness of 20 mils.
	EB	Excellent	Developed	

process requires a glove box facility, relatively little expense is involved in replacing the chamber and the TIG weld torch assembly should melt-through occur. However, the replacement of an EB weld chamber is significantly more expensive. To minimize the possibility of such contamination, weld rings placed between the weld joint and the fuel, as used in the Transit heat source while TIG welding the fuel liner, should be considered in the design.

The complexity of transporting the fueled heat source in and out of the EB chamber for a multi-layer design is another consideration in weld process selection. Unless the EB chamber is attached to the glove-box, precautions must be instituted to prevent a hazard to personnel. For the Transit and Pioneer RTG heat sources and for the Pioneer RHU heaters, TIG welding was used to assemble the various components. The only EB weld employed was the joining of the PRD to the liner cap prior to inserting the solid fuel form in the liner body.

The last consideration in selecting the weld process is the possible need to provide an efficient heat transfer medium, such as helium, in the gaps of a multi-layered heat source. Such gas would be present in the glove box during TIG welding but would, of course, be absent during the EB welding operation.

Although the advantages and disadvantages of the candidate weld processes have been presented, the selection of the weld process will have to await selection of the heat source materials.

3.2.3 Joint Design

A butt-type joint provides an assembly of minimum weight. Four different joint configurations can be considered: the straight-butt, step-butt, and angular butt which can be used to weld hemispherical sections, and the melt-down, which is an extension of the straight-butt and can be used to weld a flat disc to a tube section. Figure 4 illustrates each of these joints. Each has been successfully utilized in joining refractory metal alloys, and each has advantages and disadvantages depending on cost to fabricate, fixture requirements, and assembly design.

Figure 4. Candidate Weld Joint Designs

Straight-Butt. The straight-butt joint is fabricated at a low cost. The edge preparation is critical because it must be normal to the part surface and the centerline; however, the need for extreme accuracy in concentricity and roundness is minimized. The chief draw-back for the straight-butt joint is the need for precise fixtures to provide the accurate alignment critical for sound weldments.

Step-Butt. A step-butt joint has been successfully utilized in the Transit and Pioneer RTG heat sources and in the Large Radioisotope Heat Source program. This success can be attributed to the great care taken to provide extremely precise diameters, concentricities and roundness at the joint. These requirements resulted in high fabrication and inspection costs. An advantage of the step-butt joint is that no fixtures are required for precision assembly.

Angular-Butt. The angular-butt joint combines the good aspects of the straight-butt and the step-joint. The angular-butt configuration is producible at reasonable cost, but care in machining must be followed to produce the mating edges to close angular tolerances and squareness to the centerline. Problems occur in adequately inspecting these edges to assure accurate fit-up. As with the step-butt joint, complicated fixtures for assembly to maintain alignment would not be required.

Melt-Down. The fabrication of a joint for the melt-down weld is the least critical and least costly of all the techniques. There is no need to produce two tubular components whose edges fit precisely for assembly. Even the tolerances for squareness of the edge can be relaxed, because extra material is provided in the closure disc to fill voids during the molten phase of the weld process. Only a simple hold-down fixture is required to

place and center the closure disc over the tubular cross-section. Two welds are required to effect closure of each heat source layer. This type weld has been successfully utilized in the Pioneer heater units.

The type of weld joint to be used will depend on the design of the heat source, the results of the parametric study, and consultation with the fueling agency. It is probable that a step-butt joint would be preferred for a hemispherical-ended heat source design and the melt-down joint would be employed for a tubular flat-ended heat source design.

3.3 CLEANING

Cleaning of the tantalum- and molybdenum-base alloy refractory metals is extremely critical because surface contamination can result in embrittlement of the material and the welds during the welding operation and during operation at elevated temperatures. Surface contamination results from the use of lubricants during forming and machining operations and from hot-forming as is required with some molybdenum alloys. Mechanical removal of contaminants is readily accomplished by a vapor blast (liquid hone) process. Grit blasting is not recommended because of the possibility of embedding the powder particles and further contaminating the material. Chemical cleaning by immersion is the most economical and most reliable means for removing surface contamination. Fortunately, the candidate alloys discussed above lend themselves to being cleaned by chemical processes.

Tantalum Alloys. During the Transit and Pioneer RTG programs, an Atomics International chemical cleaning procedure specification (Reference 20) was successfully utilized. This procedure involved the successive immersion of the parts in various acid and basic solutions with rinses in distilled water.

Molybdenum Alloys. Molybdenum alloys are chemically cleaned by a number of different solutions (Reference 21) described as follows:

Molten Caustic Bath - Removes heavy oxide coatings.

Hot 10% Caustic Soda in Water - Removes thin oxide films

Hot Complex Acid - Prepares surface for welding.

"Ryan" Solution - Prepares surface for welding.

"Bell Laboratory" Solution - Produces ultraclean surfaces for electronic applications and could be used for these parts.

Platinum Alloys (Including Oak Ridge Alloy). During the Transit and Pioneer RTG programs, an Atomics International chemical cleaning procedure specification (Reference 22) was successfully utilized. Parts are cleaned in acid solutions and rinsed in water.

Hafn alloy. The cleaning procedures for Hafn alloy are not as critical as those for the tantalum alloys because it appears, from a qualitative standpoint, that it has a greater tolerance for oxygen as denoted by its oxidation resistance and excellent ductility when tensile tested in air at 2000°F. The procedure consists of acid washes with rinses in distilled water.

3.4 HEAT TREATING

Heat treatment of the tantalum- and molybdenum-base alloy refractory metals is extremely critical because the lack of furnace atmosphere control can severely embrittle the material. Tantalum alloys will be heat treated under high vacuum conditions, whereas molybdenum-base alloys can be heat treated in either vacuum (10^{-4} torr or better), dry hydrogen (dew point less than -60°F) or pure inert atmospheres. Atomic International tantalum alloy heat treat specification (Reference 23) was successfully utilized to heat treat Transit and Pioneer RTG capsule piece parts. Cracked ammonia and nitrogen atmospheres are to be avoided in the heat treatment of molybdenum alloys because of possible nitriding and embrittlement of the surface.

Pt-20Rh can be heat treated in still air. Because of the tungsten content in the Oak Ridge Alloy, heat treatment in vacuum is recommended. Atomics International platinum alloy heat treat specification (Reference 24) was successfully utilized to heat treat Transit and Pioneer RTG capsule piece parts. Hafn alloy was annealed at 1600°F in both vacuum (10^{-4} torr or better) and inert gas atmospheres. Heat treatment in an air furnace is only used to prepare the in-situ formed oxide layer to impart interdiffusion resistance.

4. QUALITY ASSURANCE

The quality requirements of the Artificial Heart Heat Source program make it mandatory to have a knowledge of material and fabrication history, material properties, and material integrity in the as-received and fabricated conditions. Procurement specifications will be generated and records

will be maintained of these procurement specifications, vendors, heat or batch numbers, receiving dates and receiving inspection. All material will be serialized to maintain traceability from the starting raw material to the finished assembled heat source.

4.1 AS-RECEIVED MATERIAL

Material vendors will be required to certify and document fabrication history, heat treatments and/or annealing schedules, because performance of refractory metals is dependent on the raw material fabrication processes. Data submitted by material producers on nondestructive test results, impurity chemical content, tensile properties (yield strength, tensile strength, modulus of elasticity and elongation), and microstructure constitute proof of conformance to material specifications.

Nondestructive testing may consist of visual examination, and dye penetrant and ultrasonic pulse echo tests. The visual examination and dye penetrant test determine surface conditions as to cracks, pits, laps and seams. The ultrasonic pulse echo determines internal quality for inclusions, porosity and especially the presence of delaminations in molybdenum alloys.

Impurities beyond specified limits can result in material embrittlement and affect secondary fabrication such as forming and welding. Impurities normally considered critical are oxygen, nitrogen, hydrogen and carbon for the molybdenum and tantalum alloys whereas iron and gold are critical for the platinum alloys. Strength of refractory metals is also affected by impurity content. Thus, data on tensile yield, tensile ultimate, and elongation are required for the raw materials.

Fabricability of refractory metals is dependent on the material's microstructure. The tantalum alloys are most fabricable when the grains are equiaxed as a result of a recrystallization anneal. Molybdenum alloys are most fabricable in the stress relieved condition having a fibrous microstructure. Therefore, the material specifications will contain these requirements.

4.2 FABRICATION

Quality of fabricated piece parts will be ascertained by controlling processes and conducting some nondestructive and destructive tests. Control of forming and machining procedures assure dimensional and surface quality

control, whereas control of cleaning and heat treating processes, as stated above, assure ductility.

4.3 HEAT SOURCE ASSEMBLY

The quality of the assembled heat source must also be controlled. During fabrication development and fabrication of test heat sources for the design development test program, dye penetrant inspection in conjunction with visual examination will assure weld surface quality. Internal weld quality will be determined by X-ray radiography and microstructural examination of example welds. For live-fueled units, visual examination only will be utilized to assure surface integrity. The internal quality of all weldments will be assured by microstructural analyses of samples welded prior to and after completing the welding of each live assembly. To further assure the quality of the assembled heat source, helium leak checking of each layer, after final closure, can be performed. Mass spectrometer helium leak detectors are used routinely and have sensitivities for detecting leaks as small as 5×10^{-11} std-cc/sec.

5. SUMMARY OF DATA

The key data required for selection of materials and material system combinations are summarized in Tables 8 and 9. Factors for material selection include density, raw material availability, raw material cost, forming and welding experience and stress to rupture, if a selective vent is required in the system. Compatibility with the fuel and between the various capsule materials becomes a dominant factor, especially for the highest projected operating temperature of 2000°F. Ability to withstand accident environments (especially impact) and contain the radioisotope fuel is also a factor for selecting the capsule material(s).

6. RECOMMENDED CAPSULE COMBINATIONS

An analysis of the raw material availability, strength (impact), fuel compatibility, and material interaction data permitted initial selection of candidate material system combinations for the parametric study. The inclusion of other criteria such as cost and scheduling will be part of the

Table 8. Summary of Factors for Selection of Materials for Heart Heat Source

Material	Density (lbs/in ³)	Availability	Cost		Fabrication Experience	Weldability	Stress to Rupture in 10 years (ksi)	
			Sheet \$/lb	Tube (1-in Dia by 0.020 wall) \$/linear in.			1350°F	2000°F
Ta-10W	0.608	Readily available as sheet and bar.	47	13	Liners successfully fabricated & assem- bled for Transit & Pioneer RTG heat sources & Pioneer RHU heaters. Tran- sit & Pioneer RTG heat sources were formed sheet metal & Pioneer RHU was drawn tubing.	Liners successfully TIG welded for Tran- sit & Pioneer RTG heat sources & Pion- eer RHU heaters. The RTG's were circum- ferential butt welds & RHU's were a cir- cumferential melt down weld.	60	19
T-111	0.604	Readily available as sheet and bar.	142	40	Strength members successfully fab- ricated and assem- bled for Transit & Pioneer RTG heat sources. Transit & Pioneer RTG's were formed sheet metal & Pioneer RHU was machined tubing. However, drawn tubing has been produced.	Strength members successfully welded with same remarks as for Ta-10W above.	65	14
Unalloyed Mo	0.369	Readily available as sheet and bar.	32	6.50	Sheet readily form- able at RT-800°F depending on the degree of forming. Drawn tubing has been produced.	Weldable by EB pro- cess better than TIG but preheat of joint area gives best results for both processes.	25	8
Mo-50Re	0.469	Production of 20,000 units/yr requires 800-2700 lbs/yr depending on starting sheet forming blank size.	1420	100	Sheet formable at RT but may require intermediate an- neals. There is some limited ex- perience in the fabrication of cap- sule piece parts.	Readily weldable by the TIG & EB pro- cesses.	53.8	7.9
TZM	0.360	Readily available as sheet and bar.	40	15	Sheet readily formable at 200-800°F depending on the degree of form- ing. Drawn tubing and machined parts have been produced.	Same remarks as for unalloyed Mo.	98	31

Table 8 (Continued). Summary of Factors for Selection of Materials for Heart Heat Source

Material	Density (lbs/in ³)	Availability	Cost		Fabrication Experience	Weldability	Stress to Rupture in 10 years (ksi)	
			Sheet \$/lb	Tube (1-in Dia by 0.020 wall) \$/linear in.			1350°F	2000°F
Pt-20Rh	0.677	Readily available as sheet & bar. Production of 20,000 units/yr requires 1700-4700 lbs/yr Pt & 400-1200 lbs Rh/yr depending on starting sheet forming blank size.	2200	100	Clad successfully fabricated & assembled for Transit & Pioneer RTG's & Pioneer RHU heaters. Same remarks as for Ta-10W above.	Clad successfully TIG welded for Transit & Pioneer RTG's & Pioneer RHU heaters. Same remarks as for Ta-10W above.	6.2	1.2
Palladium	0.434	Current output 34,000 lbs/yr & 53,000 lbs/yr projected Western World output in 5 yrs. 20,000 units/yr requires 1400-4700 lbs/yr depending on starting sheet forming blank size.	675	35	Limited, if any, experience available for heat source applications. Sheet readily formable.	Readily weldable.	0.4	0.1
Oak Ridge Alloys Compositional Range: Pt-30%-- Rh-10%W	Pt-30Rh-10W 0.592	The remarks stated above for Pt-20Rh alloy would essentially be applicable with a slightly greater amount of Rh being required for 20,000 units/yr	2400	150	No sheet metal forming experience. Finished products may not be available in time for current program due to lack of fabrication development.	No problems based on initial efforts reported.	No data Available	No data Available
Hafn alloy Compositional Range: a. Hf-0.5 to 3Pt b. Hf-0.5 to 3Pd c. Hf-0.1 to 3Pt- 0.1-3Pd	Hf-3Pt: 0.485	Estimated Western World supply of Hf is 500,000,000 lbs.	200	75	Ingot material successfully spin forged at 1400-1700°F. Small sheet samples rolled at RT. No sheet metal forming experience. Finished products may not be available in time for current program due to lack of fabrication development.	No problems based on initial efforts reported.	No data Available	No data Available

Table 9. Summary of Compatibility and Safety Data for Selection of Material System Combinations for Heart Heat Source

Candidate Combination	Compatibility				Safety (Impact)
	Fuel/Liner		Between Layers		
	1350°F	2000°F	1350°F	2000°F	
I. <u>Single Layer</u>					
Hafnalloy	Short time data-compatible with CeO ₂ simulant	Short time data-compatible with CeO ₂ simulant	-	-	In solid bar form, it performed similarly to T-111 at 2000°F, 300 ft/sec and 45° impact angle. Good oxidation resistance.
Oak Ridge Alloy	Probably OK	?	-	-	No data available on impact but good oxidation resistance.
II <u>Two Layer</u>					
Ta-10W/Hafnalloy	OK	No	Probably OK	No data available, but in-situ formed oxide barrier coating on Hafnalloy would probably perform OK.	Ta-10W had some breaching in Transit RTG-program. Hafnalloy-same remarks as above. If Hafnalloy breaches, Ta-10W would powder when exposed to air.
Ta-10W/Oak Ridge Alloy	OK	No	Probably OK	Barrier between layers would be required based on results of T-111/Pt-20Rh couple.	Same remarks as above for Ta-10W and Hafnalloy performance.
Ta-10W/Pt-20Rh	OK	No	OK	Same remarks as above for Ta-10W/Oak Ridge Alloy.	Ta-10W & Pt-20Rh had some breaching in Transit program. If clad breaches, Ta-10W will powder if exposed to air

Table 9 (Continued). Summary of Compatibility and Safety Data for Selection of Material System Combinations for Heart Heat Source

Candidate Combination	Compatibility				Safety (Impact)
	Fuel/Liner		Between Layers		
	1350°F	2000°F	1350°F	2000°F	
II. <u>Two Layer</u> (Continued)					
Mo/Hafn alloy	OK	OK	Probably OK	No data available, but in-situ formed oxide barrier coating on Hafn alloy would probably perform OK.	No impact data available for unalloyed Mo. Performance of Hafn alloy same remarks as above. If Hafn alloy breaches, Mo oxidation will be by volatilization when temperature >1200°F
Mo/Oak Ridge Alloy	OK	OK	OK	Al ₂ O ₃ barrier between layers would be required.	Same remarks as above for unalloyed Mo and Oak Ridge Alloy.
Mo-50Re/Hafn alloy	OK	OK	OK	Same remarks as above for unalloyed Mo/Hafn alloy.	No impact data available for Mo-50Re. Same remarks as above for Hafn alloy.
Mo-50Re/Oak Ridge Alloy	OK	OK	OK	Al ₂ O ₃ barrier between layers would be required.	Same remarks as above for Mo-50Re and Oak Ridge Alloy.
Pt-20Rh/Hafn alloy	OK	OK	OK	Probably OK. If required, in-situ formed oxide barrier coating on Hafn alloy would probably be compatible.	Pt-20Rh as clad intermittently breached on impact in Transit RTG program. Same remarks as above for Hafn alloy. No catastrophic oxidation problem with Pt-20Rh liner as with tantalum or molybdenum alloys if Hafn alloy (outside) layer breaches.
Pt-20Rh/Oak Ridge Alloy	OK	OK	OK	OK	Same remarks as above for Pt-20Rh & Oak Ridge Alloy. No catastrophic oxidation of Pt-20Rh liner as stated above.
TZM/Pt-20Rh	OK	OK	OK	Al ₂ O ₃ barrier between layers would be required.	TZM welded construction impact performed satisfactorily in DART program at 1300 to 2000°F & 200 ft/sec velocity. Some failures have been reported in threaded joint parts under Pioneer Program. Pt-20Rh impact performance same as above. If clad breaches TZM oxidation will be by volatilization when temperature >1200°F.
TZM/Hafn alloy	OK	OK	OK	Same remarks as above for Mo/Hafn alloy couple.	Same remarks as above for Mo/Hafn alloy combination.
TZM/Oak Ridge Alloy	OK	OK	OK	Al ₂ O ₃ barrier between layers would be required.	Same remarks as above for TZM and Oak Ridge Alloy. Catastrophic oxidation of TZM may occur as stated above if Oak Ridge Alloy breaches.

Table 9 (Continued). Summary of Compatibility and Safety Data for Selection of Material System Combinations for Heart Heat Source

Candidate Combination	Compatibility				Safety (Impact)
	Fuel/Liner		Between Layers		
	1350°F	2000°F	1350°F	2000°F	
III. <u>Three Layer</u>					
Ta-10W/T-111/Pt-20Rh	OK	No	OK	Barrier between T-111 & Pt-20Rh would be required. However, a candidate barrier is unknown at this time.	Met Transit RTG & Pioneer RHU requirements even though liner & clad occasionally breached in Transit program. When clad breaches, Ta alloys powder in air.
Mo/T-111/Pt-20Rh	OK	OK	OK	Mo & T-111 probably compatible. Same remarks as above for T-111/Pt-20Rh couple.	Mo may perform equivalent to Ta-10W but no experience with welded construction. If clad & strength member breach, Mo will volatilize above 1200°F and T-111 will powder in air.
Mo-50Re/T-111/Pt-20Rh	OK	OK	OK	Same remarks as above for T-111/Pt-20Rh couple.	Same remarks as above for Mo/T-111/Pt-20Rh combination.
Pt-20Rh/T-111/Pt-20Rh	OK	OK	OK	Same remarks as above for T-111/Pt-20Rh couple.	If breaches occur in all three members, Pt-20Rh liner would still be capable of containing fuel even though it might also breach.
Ta-10W/TZM/Pt-20Rh	OK	No	OK	Al ₂ O ₃ barrier coating would be required between TZM and Pt-20Rh at 2000°F.	Same remarks as above for TZM/Pt-20Rh combination. Ta-10W would powder due to oxidation if TZM and clad breaches or TZM volatilizes if clad breaches.
Mo-50Re/TZM/Pt-20Rh	OK	OK	OK	Same remarks as above for TZM/Pt-20Rh couple.	Same remarks as above for TZM/Pt-20Rh combination. Same remarks as above for volatilization of Mo alloys if Pt-20Rh breaches.
Pt-20Rh/TZM/Pt-20Rh	OK	OK	OK	Same remarks as above for TZM/Pt-20Rh couple.	Same remarks as above for TZM/Pt-20Rh combination. However, if clad breaches & TZM volatilizes, the Pt-20Rh liner would still remain intact to contain fuel.
Mo/T-111/Oak Ridge Alloy	OK	OK	OK	Same remarks as above for Mo/T-111/Pt-20Rh.	Same remarks as above for Mo/T-111/Pt-20Rh. However, Oak Ridge Alloy may have better chance of remaining intact because of its much higher tensile strength than the Pt-20Rh.

parametric study itself. A summary of the single-, two- and three-layer material systems for heat sources containing nonselective and selective vents and operating at 1350 or 2000°F is given in Table 10. The following ground rules were used in deriving the candidate material systems:

- Iridium can be eliminated as a clad candidate because of unavailability of the raw material.
- Palladium can be eliminated as a clad candidate because no compatibility data with other materials are available, because its melting point is only 300°F above the anticipated cremation temperature, and because its stress to rupture is extremely low.
- Pt-20Rh and palladium can be eliminated from consideration for single layered capsules because of their low relative strength and their susceptibility to grain growth at elevated temperatures.
- W-25Re can be eliminated as a liner or strength member material because no compatibility data are available between it and other capsule materials such as T-111 and Pt-20Rh, because of its extremely high density (0.714 lb/in³), and because of difficulties in machining and fabricating this alloy.
- Ta-10W can be considered a liner material at 1350°F only because of potential incompatibility with the fuel at 2000°F.
- T-111 can be considered as a strength member at 2000°F if an adequate diffusion barrier is found to eliminate interaction with Pt-Rh alloys. (A test program to determine the adequacy of hafnia and magnesia as diffusion barriers is currently being planned under IR&D funding.)
- Mo and Mo-50Re are preferred as a liner over TZM because of their lower ductile/brittle transition temperatures and easier fabricability. These materials are proposed as liners only. In a two-layer system having a liner/strength member combination, TZM will be used because of its higher strength.
- A three-layer system of TZM or Mo/TZM/clad will not be considered since it has no advantages over a TZM/clad system.
- Selective vent technology allows this vent to be welded into the noble metal alloys only (including the Oak Ridge Alloy). Thus one of the capsule members must be a Pt-Rh alloy.
- The Oak Ridge Alloy has little advantage over Pt-20Rh in a three-layer capsule containing a nonselective vent. For a selective vent, however, this material must be considered because of its higher strength.

Table 10. Candidate Material System Combinations for Parametric Study

Non-Selective Vent - 1350°F

<u>Single-Layer</u>	<u>Two-Layer</u>	<u>Three-Layer</u>
Hafn alloy	Ta-10W/Pt-20Rh	Ta-10W/T-111/Pt-20Rh
Oak Ridge Alloy	Ta-10W/Hafn alloy	Mo/T-111/Pt-20Rh
	Ta-10W/Oak Ridge Alloy	Mo-50Re/T-111/Pt-20Rh
	TZM/Pt-20Rh	Pt-20Rh/T-111/Pt-20Rh
	Mo/Hafn alloy	Ta-10W/TZM/Pt-20Rh
	Mo/Oak Ridge Alloy	Mo-50Re/TZM/Pt-20Rh
	Mo-50Re/Hafn alloy	Pt-20Rh/TZM/Pt-20Rh
	Mo-50Re/Oak Ridge Alloy	
	Pt-20Rh/Hafn alloy	
	Pt-20Rh/Oak Ridge Alloy	

Selective Vent - 1350°F

<u>Single-Layer</u>	<u>Two-Layer</u>	<u>Three-Layer</u>
Oak Ridge Alloy	Ta-10W/Pt-20Rh	Ta-10W/T-111/Pt-20Rh
	Ta-10W/Oak Ridge Alloy	Ta-10W/T-111/Oak Ridge Alloy
	TZM/Pt-20Rh	Mo/T-111/Pt-20Rh
	Mo/Oak Ridge Alloy	Mo/T-111/Oak Ridge Alloy
	Mo-50Re/Oak Ridge Alloy	Mo-50Re/T-111/Pt-20Rh
	Pt-20Rh/Hafn alloy	Mo-50Re/T-111/Oak Ridge Alloy
	Pt-20Rh/Oak Ridge Alloy	Pt-20Rh/T-111/Pt-20Rh
		Ta-10W/TZM/Pt-20Rh
		Ta-10W/TZM/Oak Ridge Alloy
		Mo-50Re/TZM/Pt-20Rh
		Mo-50Re/TZM/Oak Ridge Alloy
		Pt-20Rh/TZM/Pt-20Rh

Non-Selective Vent - 2000°F

<u>Single-Layer</u>	<u>Two-Layer</u>	<u>Three-Layer</u>
Hafn alloy	TZM/Pt-20Rh	Mo/T-111/Pt-20Rh
Oak Ridge Alloy	Mo/Hafn alloy	Mo-50Re/T-111/Pt-20Rh
	Mo/Oak Ridge Alloy	Pt-20Rh/T-111/Pt-20Rh
	Mo-50Re/Hafn alloy	Mo-50Re/TZM/Pt-20Rh
	Mo-50Re/Oak Ridge Alloy	Pt-20Rh/TZM/Pt-20Rh
	Pt-20Rh/Hafn alloy	
	Pt-20Rh/Oak Ridge Alloy	

Selective Vent - 2000°F

<u>Single-Layer</u>	<u>Two-Layer</u>	<u>Three-Layer</u>
Oak Ridge Alloy	TZM/Pt-20Rh	Mo/T-111/Pt-20Rh
	Mo/Oak Ridge Alloy	Mo/T-111/Oak Ridge Alloy
	Mo-50Re/Oak Ridge Alloy	Mo-50Re/T-111/Pt-20Rh
	Pt-20Rh/Hafn alloy	Mo-50Re/T-111/Oak Ridge Alloy
	Pt-20Rh/Oak Ridge Alloy	Pt-20Rh/T-111/Pt-20Rh
		Mo-50Re/TZM/Pt-20Rh
		Mo-50Re/TZM/Oak Ridge Alloy
		Pt-20Rh/TZM/Pt-20Rh

REFERENCES

1. L. B. Hunt and F. M. Lever, "Availability of Platinum Metals - A Survey of Productive Resources in Relation to Industrial Uses." Symposium on Platinum Group Metals of American Chemical Society, New York, September, 1970.
2. J. H. Port, "Rhenium and Rhenium Alloy Technology." Annual Meeting of American Institute of Mining, Metallurgical and Petroleum Engineers, New York, February, 1964.
3. Letter from D. W. Cole to H. Lurie dated 27 May 1971 containing LASL Data Sheets from CMB-1797.
4. R. L. Andelin and J. D. Watrous, "Refractory-Alloy Compatibility with Plutonia-238 at High Temperature," Interim Report, Donald W. Douglas Laboratories Report MDCG2034, February 1971.
5. "Metal Container - $^{238}\text{PuO}_2$ Compatibility," LASL Report CMB11-9739, April 8, 1971.
6. J. E. Selle, J. J. English, P. E. Jeaney and J. R. McDougal, "The Compatibility of $^{238}\text{PuO}_2$ with Various Refractory Metals and Alloys," Interim Report (Draft), Mound Laboratory Report MLM-1706, July 1970.
7. H. M. Hoffman, J. R. Ogren, and R. C. Ham, "DART Capsule Materials Diffusion Study," TRW Report 4811.2.66-005, 21 June 1966.
8. H. M. Hoffman, "Transit Heat Source Materials Compatibility Investigation," TRW Systems IOC 4742.3-70-126, December 1970.
9. H. M. Hoffman, "Transit Heat Source Materials Compatibility Investigation Long Term Test Addendum," TRW Systems IOC 4742.3-70-126A, April 1971.
10. M. Appel, et al., "Development of a High Temperature Vented Radioisotopic Capsule, Final Summary Report," TRW(A)-11069-053, June 1970.
11. R. P. Elliott, Constitution of Binary Alloys, First Supplement. McGraw-Hill Book Co., 1965.
12. R. G. Brengle, "Large Radioisotope Heat Source Capsule Program - Materials Compatibility, Topical Report No. 11," Atomic International AI-AEC-12966, July 3, 1970.
13. "Radioisotope Heated Propellant Reaction Control Systems," Final Report TRW AFRPL-TR-68-150, October 1968.

REFERENCES (Continued)

14. "Radioisotope Heated Propellant Reaction Control Systems," Second Quarterly Report, TRW AFRPL-TR-66-275, October 1966.
15. A. J. Steinberger, "Compatibility Correlation Using the Larson-Miller Parameter," TRW IOC to H. Lurie, 71.4762.2-19, March 18, 1971.
16. "Development of a Radioisotope Heat Source Subsystem for Heart Devices," (Draft-Final Report), Hittman Associates, Inc. HIT-4086-2, November 1970.
17. A. V. Levy and S. E. Bramer, "The Development of Refractory Sheet Metal Structures," National Aeronautical Meeting SAE, October 1959.
18. A. V. Levy and S. E. Bramer, "Fabrication of 2000°F Structural Components," National Aeronautical Meeting SAE, October 1959.
19. R. E. Jackson, "Conventional TZM Sheet Forming Techniques," SAMPE Journal, April/May 1967.
20. Atomics International North American Rockwell Specification ST0610NA0010, "Cleaning Tantalum and Tantalum Base Alloys," 8 October 1969.
21. "Molybdenum Metal," Climax Molybdenum Co., 1960.
22. Atomics International North American Rockwell Specification ST0610NA0011, "Cleaning Platinum and Platinum Base Alloys," 9 October 1969.
23. Atomics International North American Rockwell Specification ST0611NA0008, "Heat Treatment of Tantalum and Tantalum Base Alloys," 8 October 1969.
24. Atomics International North American Rockwell Specification ST0611NA0009, "Heat Treatment of Platinum and Platinum Base Alloys," 8 October 1969.

APPENDIX

MATERIAL PROPERTIES

Table 1. Ta-10W

- A. Density - 0.608 lbs/in^3 (16.83 gm/cc) - (References 1 and 2)
- B. Melting Point - 5495°F - (References 1 and 2)
- C. Thermal Conductivity - Figure 1 (Reference 3)
- D. Specific Heat - Figure 2 (Reference 3)

Specific Heat Coefficients

$$C_p = 0.0279 + 0.00904 \times 10^{-3}T + 0.000617 \times 10^{-6}T^2$$

where

$$C_p = \text{Btu/lb-}^\circ\text{F}$$

$$T = ^\circ\text{F}$$

- E. Thermal Expansion - Figure 3 (References 2 and 3)
- F. Emissivity - (References 4 and 5)

Total emissivity = 0.328 from 2419 to 4399°F

- G. Tensile Properties - Sheet Recrystallized at 2600°F for one hour

Ultimate Tensile Strength vs Temperature - Figure 5 (Ref. 6)

Yield Strength vs Temperature - Figure 7 (Ref. 6)

Modulus of Elasticity vs Temperature - Figure 9 (Ref. 2)

Elongation vs Temperature - Figure 10 (Ref. 6)

- H. Stress to Rupture - Sheet Stress Relieved ($2250^\circ\text{F}/3 \text{ hours}$) and Recrystallized ($2730^\circ\text{F}/1 \text{ hour}$) - Figure 12 (Reference 2)

Table 2. T-111

- A. Density - 0.604 lbs/in³ (16.72 gm/cc) - (Ref. 1)
- B. Melting Point - 5400°F (estimated) -(Ref. 1)
- C. Thermal Conductivity - Figure 1 (Refs. 1 and 7a)
- D. Specific Heat - Figure 2 (Refs. 3 and 7a)

Specific Heat Coefficients - (Ref. 3)

$$C_p = 0.022 + 0.00532 \times 10^{-3}T + 0.000489 \times 10^{-6}T^2$$

where

$$C_p = \text{Btu/lb-}^\circ\text{F}$$

$$T = ^\circ\text{F}$$

- E. Thermal Expansion - Figure 3 (Refs. 2, 3, and 7b)
- F. Emissivity - Figure 4 (Ref. 7e)
- G. Tensile Properties - Sheet Recrystallized at 3000°F for one hour
 - Ultimate Tensile Strength vs Temperature - Figure 5 (Refs. 2, 7b, 7f, and 8)
 - Yield Strength vs Temperature - Figure 7 (Refs. 2, 7b, 7f)
 - Modulus of Elasticity vs Temperature - Figure 9 (Ref. 7g)
 - Elongation vs Temperature - Figure 10 (Refs. 2, 7b, 7f)
- H. Stress to Rupture - Sheet Recrystallized at 3000°F for one hour - Figure 12 (Refs. 7h and 9)

Table 3. Unalloyed Molybdenum

- A. Density - 0.369 lbs/in^3 (10.22 gm/cc) - (Refs. 10 and 11)
- B. Melting Point - 4732°F - (Refs. 10 and 11)
- C. Thermal Conductivity - Figure 1 (Refs. 10 and 11)
- D. Specific Heat - Figure 2 (Refs. 10 and 11)
- E. Thermal Expansion - Figure 3 (Refs. 10 and 11)
- F. Emissivity - Figure 4 (Refs. 10 and 11)
- G. Tensile Properties - Stress Relieved one hour at 1800°F
 - Ultimate Tensile Strength vs Temperature - Figure 5 (Refs. 10, 11 and 12)
 - Yield Strength vs Temperature - Figure 7 (Refs. 10 and 11).
 - Modulus of Elasticity vs Temperature - Figure 9 (Refs. 10, 11, and 13)
 - Elongation vs Temperature - Figure 10 (Refs. 10 and 11)
- H. Stress to Rupture - Stress Relieved one hour at 1800°F - Figure 12 (Refs. 10, 11 and 14)

Table 4. Mo-50Re

- A. Density - 0.496 lbs/in^3 (13.76 gm/cc) - (Refs. 15 and 16)
- B. Melting Point - 4620°F - (Refs. 15 and 16)
- C. Thermal Conductivity - Figure 1 (Ref. 16)
- D. Specific Heat - Figure 2 (Refs. 10, 11 and 16)

Note: Data taken as a weight proportion for the specific heats of unalloyed Mo and Re.

- E. Thermal Expansion - Figure 3 (Ref. 16)
- F. Emissivity - No data available.
- G. Tensile Properties - Recrystallized Sheet

Ultimate Tensile Strength vs Temperature - Figure 5 (Ref. 16)

Yield Strength at R.T. - $123,000 \text{ psi}$ - (Ref. 16)

Modulus of Elasticity at R.T. - $53.3 \times 10^6 \text{ psi}$ - (Ref. 16)

Elongation at R.T. - 16-20% - (Ref. 16)

- H. Stress to Rupture - Recrystallized sheet - Figure 12 (Ref. 17)

Table 5. TZM

A. Density - 0.360 lbs/in³ (9.96 gm/cc) - (Ref. 3)

B. Melting Point - 4732°F - (Refs. 10 and 11)

C. Thermal Conductivity - Figure 1 (Ref. 3)

D. Specific Heat - Figure 2 (Ref. 3)

$$C_p \text{ (Btu/lb-°F)} = 0.065 - 0.00156 \times 10^{-3}T + 0.00293 \times 10^{-6}T^2$$

where

$$C_p = \text{Btu/lb-°F}$$

$$T = \text{°F}$$

E. Thermal Expansion - Figure 3 (Ref. 3)

F. Emissivity - Assume same as unalloyed molybdenum - Figure 4 (Refs. 10 and 11)

G. Tensile Properties - Stress Relieved sheet and bar

Ultimate Tensile Strength vs Temperature - Figures 5 and 6
(Refs. 11 to 14 and 18, 19) (See next page)

Yield Strength vs Temperature - Figures 7 and 8 (Refs. 11 to 14 and 18, 19) (See next page)

Modulus of Elasticity vs Temperature - Figure 9 (Refs. 10, 11, 13)

Elongation vs Temperature - Figures 10 and 11 (Refs. 11 to 14 and 18, 19) (See next page)

H. Stress to Rupture - Stress Relieved sheet and bar - Figure 12
(Refs. 14, 20 and 21)

Table 5 (Continued). Tensile Properties of TZM Sheet

Test Temp (°F)	Material Condition	Carbon Content (%)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Reference
RT	Stress Relieved	.019	124.3(L)*	101.4(L)	13.5(L)	11,14
			126.9(T)*	107.9(T)	4.5(T)	11,14
	Stress Relieved	.054	102.5(L)	-	0	11,14
			139.4(T)	121.1	10.0	11,14
	Stress Relieved 2200°F/1 hr	.016	108.7	105.4	28	13
	Stress Relieved 2200°F/1 hr	.04	124.5	113.5	15	14
			143.5	129.9	12.5	14
392	Stress Relieved	.019	95.6	91.8	38.4	19
400	-	-	122	-	-	12
660	Stress Relieved	.019	85.3	83.1	32.8	19
800	-	-	112	-	-	12
932	Stress Relieved	.019	82.5	82.5	26.4	19
1000	Stress Relieved 2200°F/1 hr	.04	92.1	85.5	6.2	14
			92.4	86.8	7.5	14
1200	Stress Relieved	.019	102.5	-	-	12
			70.3	70.3	22.8	19
1472	Stress Relieved	.019	75.3	67.8	28.7	19
1600	Stress Relieved 2200°F/1 hr	.04	92	-	-	12
			83.1	70.6	5	14
1740	Stress Relieved	.019	87.5	-	11.2	14
			66.2	61.3	27.2	19
1800	Stress Relieved	.019	79.6(L)	-	4.0(L)	11,14
			82.4(T)	-	9.0(T)	11,14
	Stress Relieved	.054	83.4(L)	52.0(L)	10.0(L)	11,14
			89.2(T)	74.1(T)	2.0(T)	11,14
2000	-	-	77.5	-	-	12
2010	Stress Relieved	.019	57.9	54.4	27.2	19
2200	Stress Relieved 2200°F/1 hr	.04	54.6	47.7	10.6	14
			43.6	34.0	2.5	14
2280	Stress Relieved	.019	49.5	47.8	31.2	19
2400	Stress Relieved	.019	55	-	-	12
			39.3(L)	-	9.0(L)	11,14
	Stress Relieved	.054	46.6(T)	-	13.0(T)	11,14
			27.9(L)	-	26.5(L)	11,14
2500	Stress Relieved 2200°F/1 hr	.016	30.4(T)	-	18.0(T)	11,14
			47.8	46.3	18.0	13
	Stress Relieved 2200°F/1 hr	.04				
			21.8	13.6	48.8	14
2550	Stress Relieved	.019	31.8	26.6	15.0	14
			30.9	29.8	58.0	12
2820	Stress Relieved	.019	12.3	9.4	86.2	12
3000	Stress Relieved 2200°F/1 hr	.016	10.3	7.6	75.0	18

*L - Longitudinal (Parallel to rolling direction)

T - Traverse (Perpendicular to rolling direction)

Table 6. W-25Re

- A. Density - 0.714 lbs/in^3 (19.8 gm/cc) - (Ref. 22)
- B. Melting Point - 5650°F (Ref. 22)
- C. Thermal Conductivity - No data available
- D. Specific Heat - (Ref. 22)

$$\text{R.T.} = 0.035 \text{ Btu/lb-}^\circ\text{F}$$

Note: Data taken as a weight proportion for the specific heats of unalloyed Re and W.

- E. Thermal Expansion - No data available
- F. Emissivity - No data available
- G. Tensile Properties- Metallurgical Condition and Material form unknown except as stated

Ultimate Tensile Strength vs Temperature - Figure 5 (Ref. 22)

Yield Strength vs Temperature - Figure 7 (Ref. 22)

Modulus of Elasticity at R.T. - $50 \times 10^6 \text{ psi}$ - (Ref. 22)

Elongation at R.T. - 0.010 inch dia. wire - (Ref. 23)

Annealed 2910°F - 10%

Annealed 3630°F - 7%

As drawn - 2%

- H. Stress to Rupture - Annealed in H_2 for 2 hours at test temperature prior to test - Figure 12 (Ref. 17)

Table 7. Pt-20Rh

- A. Density - 0.677 lbs/in³ (18.74 gm/cc) - (Ref. 24)
- B. Melting Point - 3452°F (Ref. 24)
- C. Thermal Conductivity - (Ref. 25)

°F	Btu/ft-hr-°F
63	43.1
212	42.8

Note: Data taken as volumetric mean of thermal conductivities of platinum and rhodium

- D. Specific Heat - (Ref. 26)

$$C_p = 0.031 + 7.1766 \times 10^{-6}T + 5.4188 \times 10^{-9} T^2 - 3.5284 \times 10^{-12} T^3$$

where

$$C_p = \text{Btu/lb-°F}$$

$$T = \text{°F}$$

Note: Data taken as a weight proportion for the specific heat of platinum and rhodium

- E. Thermal Expansion - Figure 3 (Ref. 26)
- F. Emissivity - Figure 4 (Ref. 26)

Note: Data is for Pt-10Rh and should apply to Pt-20Rh

- G. Tensile Properties - Annealed sheet and rod

Ultimate Tensile Strength vs Temperature - Figure 13 (Refs. 27, 28)

Yield Strength vs Temperature - No data available

Modulus of Elasticity vs Temperature - No data available

Elongation vs Temperature - Figure 13 (Refs. 27, 28)

- H. Stress to Rupture - Figure 12 (Ref. 29)

Table 8. Palladium

- A. Density - 0.434 lb/in^3 (12.02 gm/cc) - (Ref. 30)
- B. Melting Point - 2825°F - (Ref. 30)
- C. Thermal Conductivity - (Ref. 30)
 $32 - 212^\circ\text{F} - 35.5 \text{ Btu/ft-hr-}^\circ\text{F}$
- D. Specific Heat - (Ref. 30)
 $32^\circ\text{F} - 0.0584 \text{ Btu/lb-}^\circ\text{F}$
- E. Coefficient of Linear Thermal Expansion - (Ref. 30)
 $68 - 212^\circ\text{F} - 6.17 \times 10^{-6} \text{ in/in-}^\circ\text{F}$
- F. Spectral Emissivity at $0.65\mu = 0.33$ - (Ref. 30)
- G. Tensile Properties - Annealed Sheet and Rod
Ultimate Tensile Strength vs Temperature - Figure 13 (Refs. 27,30)
Yield Strength vs Temperature - No data available
Modulus of Elasticity (Ref. 30)
 $\text{RT} - 16.7 \times 10^6 \text{ psi}$
Elongation - Figure 13 - (Refs. 27 and 30)
- H. Stress to Rupture - Figure 12 (Ref. 29)

Table 9. Iridium

- A. Density - 0.818 lbs/in^3 (22.65 gm/cc) - (Ref. 31)
- B. Melting Point - 4430°F - (Ref. 31)
- C. Thermal Conductivity - (Ref. 31)
 $32 - 212^\circ\text{F} - 855 \text{ Btu/ft-hr-}^\circ\text{F}$
- D. Specific Heat - (Ref. 31)
 $32^\circ\text{F} - 0.0307 \text{ Btu/lb-}^\circ\text{F}$
- E. Thermal Expansion - (Ref. 31)
 $68 - 212^\circ\text{F} - 3.22 \times 10^{-6} \text{ in/in/}^\circ\text{F}$
- F. Emissivity - (Ref. 31)
Spectral at $0.65\mu - 0.33$
- G. Tensile Properties (Ref. 31)
Rod vacuum annealed as follows:
Tests up to $1830^\circ\text{F} - 1500^\circ\text{C}/15 \text{ minutes}$
Tests above $1830^\circ\text{F} - 1500^\circ\text{C}/\text{one hour}$
Ultimate Tensile Strength vs Temperature - Figure 13
Yield Strength vs Temperature - Figure 14
Modulus of Elasticity at RT - $75.0 \times 10^6 \text{ psi}$
Elongation vs Temperature - Figure 13
- H. Stress to Rupture - No data available

Table 10. Oak Ridge - Pt-Rh-W Alloys

A. Density - Pt-30Rh-10W - 0.592 lbs/in^3 (17.5 gm/cc) - (Ref. 32)

Pt-26Rh-8W - No data available

B. Melting Point - Pt-26Rh-8W - 3630°F - (Ref. 32)

Pt-30Rh-10W - 3740°F - (Ref. 32)

C. Thermal Conductivity - No data available

D. Specific Heat - No data available

E. Thermal Expansion - No data available

F. Emissivity - No data available

G. Tensile Properties - Stress Relieved and Recrystallized Sheet

Ultimate Tensile Strength vs Temperature - Figure 15

Pt-26Rh-8W - (Ref. 32)

Pt-30Rh-10W - Data being generated

Yield Strength vs Temperature - No data available

Modulus of Elasticity vs Temperature - No data available

Elongation vs Temperature - Figure 17

Pt-26Rh-8W - (Ref. 32)

Pt-30Rh-10W - Data being generated

H. Stress to Rupture - No data available

Table 11. Hafnalloys (Hf-3Pt)

- A. Density - 0.485 lbs/in³ (13.42 gm/cc)
- B. Melting Point - 3860°F
- C. Thermal Conductivity - No data available
- D. Specific Heat - No data available
- E. Emissivity - No data available
- F. Thermal Expansion - No data available
- G. Tensile Properties - Annealed Sheet
 - Ultimate Tensile Strength vs Temperature - Figure 15
 - Yield Strength vs Temperature - Figure 14
 - Modulus of Elasticity vs Temperature - Figure 16
 - Elongation vs Temperature - Figure 17
- H. Stress to Rupture - No data available

Figure 1. Thermal Conductivity Vs Temperature for Molybdenum and Tantalum Alloys

Figure 2. Specific Heat Vs Temperature for Molybdenum and Tantalum Alloys

Figure 3. Thermal Expansion Vs Temperature for Refractory Metal Alloys

Figure 4. Emissivity Vs Temperature for Refractory Metal Alloys

Figure 5. Ultimate Tensile Strength Vs Temperature for Molybdenum, Tantalum and Tungsten Alloys

Figure 6. Ultimate Tensile Strength of TZM Vs Temperature as a Result of Carbon Content and Stress Relief Treatment

Figure 7. Yield Tensile Strength Vs Temperature for Molybdenum, Tantalum, and Tungsten Alloys

Figure 8. Yield Tensile Strength of TZM Vs Temperature as a Result of Carbon Content and Stress Relief

Figure 9. Modulus of Elasticity Vs Temperature for Molybdenum and Tantalum Alloys

Figure 10. Elongation Vs Temperature for Molybdenum and Tantalum Alloys

Figure 12. Stress Rupture of Refractory Metal Alloys

Figure 13. Ultimate Tensile Strength and Elongation Vs Temperature for Noble Metal Alloys

Figure 14. Yield Tensile Strength Vs Temperature for Iridium and Hafnalloys

Figure 15. Ultimate Tensile Strength Vs Temperature for Advanced Alloys

Figure 16. Modulus of Elasticity Vs Temperature for Hafn alloy - Advanced Alloy

Figure 17. Elongation Vs Temperature for Advanced Alloys

REFERENCES

1. Wah Chang Product Data Book.
2. F. F. Schmidt and H. R. Ogden, "The Engineering Properties of Tantalum and Tantalum Alloys," DMIC Report 189, September 13, 1963.
3. J. C. Hedge, C. Kostenko, and J. I. Lang, "Thermal Properties of Refractory Alloys," ASD-TDR-63-597, June 1963.
4. Aerospace Structural Metals Handbook, Volume IIA, "Non-Ferrous Alloys," Syracuse University Press, Revised March, 1966.
5. M. L. Forti, "Physical Properties and Fabrication Techniques for Tantalum-10% Tungsten Alloy," AIME Technical Conference on High Temperature Materials, Cleveland, Ohio, April 1961.
6. Personal Communication with Bill Mauer, Wah Chang Corporation, Albany, Oregon.
7. Paul E. Moorhead and Phillip L. Stone, "Survey of Properties of T-111," NASA TN D-5873, Lewis Research Center, June 1970.

The following references provided the data in the above survey:

- a. P. E. Krieser, J. W. Toth, and R. C. McRae, "Bore Seal Technology Topical Report," WAED-64-54E, Westinghouse Electric Corporation (NASA CR-54093), December 1964.
- b. R. L. Amon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet," WANL-PR-M-006, Westinghouse Electric Corporation, October 15, 1963.
- c. R. L. Amon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet," WANL-PR-(M)-003, Westinghouse Electric Corporation, February 16, 1963.
- d. R. Begley, "T-111 Tantalum-Base Alloy Refractory Metal," Special Technical Data 52-365, Westinghouse Electric Corporation, March 1963.
- e. Sigferd Peterson, ed., "Metals and Ceramics Division Annual Progress Report for Period Ending June 30, 1964," ORNL-3670, Oak Ridge National Laboratory, October 1964.
- f. K. D. Sheffler, J. C. Sawyer and E. A. Steigerwald, "Mechanical Behavior of Tantalum-Base Alloy at Elevated Temperature," NASA CR-1436, 1969.
- g. Astronuclear Lab Staff, "Elastic Modulus and Thermal Expansion of Tantalum T-111 Alloy," Westinghouse Electric Corporation, February 1964.

- h. K. D. Sheffler, "Generation of Long Time Creep Data on Refractory Alloys at Elevated Temperatures," 12th Quarterly Report, TRW Equipment Labs, NASA CR-72632, July 7, 1969.
8. K. D. Sheffler, J. C. Sawyer and E. A. Steigerwald, "Creep Behavior of Refractory Alloys in Ultrahigh Vacuum," NASA SP-245, Recent Advances in Refractory Alloys for Space Power Systems Symposium, June 26, 1969.
 9. K. D. Sheffler, "Generation of Long Time Creep Data on Refractory Alloys at Elevated Temperatures," 14th Quarterly Report, TRW Equipment Labs, NAS CR-72185, January 17, 1967.
 10. "Molybdenum Metal," Climax Molybdenum Co., 1960.
 11. J. A. Houck, "Physical and Mechanical Properties of Commercial Molybdenum-Base Alloys," DMIC Report 140, November 30, 1960.
 12. T. E. Tietz and J. W. Wilson, "Behavior and Properties of Refractory Metals," Stanford University Press, 1965.
 13. R. A. Barr and M. Semchyshen, "Stress-Strain Curves for Wrought Molybdenum and Three Molybdenum-Base Alloys," Research Laboratory of Climax Molybdenum Co., December 1959.
 14. F. F. Schmidt and H. R. Ogden, "Engineering Properties of Molybdenum and Molybdenum Alloys," DMIC Report 190, September 20, 1963.
 15. "Chase Rhenium-Molybdenum 50/50 Seamless Tubing," Rhenium Bulletin No. 1, Chase Brass and Copper, Inc.
 16. "Chase Rhenium-Molybdenum Alloys," Rhenium Bulletin No. 2, Chase Brass and Copper, Inc.
 17. P. N. Flagella and C. O. Tarr, "Creep Rupture Properties of Rhenium and Some Alloys of Rhenium at Elevated Temperature," GE-TM65-9-12, AIME Technical Meeting, French Lick, Indiana, October 1965.
 18. J. Van Orsdel, D. J. Maykuth, and V. D. Barth, "Refractory Metals (Cb, Ta, Mo, W)," DMIC Review of Recent Developments, April 16, 1969.
 19. B. A. Wilcox, A. Gilbert and B. C. Allen, "Intermediate-Temperature Ductility and Strength of Tungsten and Molybdenum TZM," Battelle Memorial Institute, AFML-TR-66-89, April 1966.
 20. "Development Data Climelt TZM," Climax Molybdenum Co., January 1962.
 21. E. O. Müller and W. Jakobeit, "'Super' Superalloys for Temperatures Over 900°C," Braun, Bovari, and Cie, Gas Turbine International, March-April 1970.

22. Materials Engineering - '70 Materials Selector Issue. Volume 70, No. 5, October, 1969. Reinhold Publishing Co.
23. Chase Rhenium, Rhenium-Molybdenum, Rhenium-Tungsten Alloys Data Sheet Publication RE-3, Chase Brass and Copper Co., Inc.
24. "Platinum, The Metal, Its Properties and Applications," International Nickel Co., Inc., Copyright 1968.
25. R. F. Vines and E. M. Wise, "The Platinum Metals and Their Alloys," International Nickel Co., Inc., 1941.
26. Johnson Matthey Research Laboratories.
27. Personal Communication with Joe Aliotto, Engelhard Industries, Carteret, N.J.
28. Mound Laboratory Material Specification MD-1-13457, "Material Specification for Platinum 20% Rhodium Alloy, Plate, Sheet, Bar and Seamless Tubing," June 30, 1970.
29. M. Semchyshen and J. J. Harwood (Editors), "Stress Rupture Properties of Some Platinum and Palladium Alloys" Presented at Refractory Metals and Alloys AIME Conference, Detroit, Michigan, May 1960.
 - a. E. P. Sadowski, "Properties at 927°C," Part I.
 - b. H. J. Albert, D. J. Accinno, and J. S. Hill, "Properties at 1300-1350°C, Part II.
30. "Palladium, The Metal, Its Properties and Applications," International Nickel Co.
31. "Iridium, The Metal, Its Alloys, Chemical Compounds and Catalytic Properties," International Nickel Co.
32. R. G. Donnelly, "ORNL Cladding Materials Program Briefing," at AEC Headquarters, February 11, 1971.