JPL D-1268

SP-100 PROGRAM

Technical Information Report Multifoil Insulation Study for Weight Reduction

Stuart D. Glazer

January 1984

National Aeronautics and
Space Administration

JPL

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

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SP-100 PROGRAM

Department of Defense
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Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

MULTIFOIL INSULATION STUDY
FOR WEIGHT REDUCTION

Stuart D. Glazer

January 1984

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA

FOREWARD

This document reports the results of a study in which the means to improve multifoil insulation were determined. A test program for evaluating the study results is recommended and described.

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SECTION 1

INTRODUCTION

The purpose of the present task is to develop high temperature multi-foil insulation suitable for use in the SP-100 thermoelectric converter project. Part of this task involves careful examination of alternate foil and foil spacing materials with the goal of effecting significant weight savings over current state-of-the-art foil insulation. This task involved the determination of the state-of-the-art foils, ascertaining what data is available, what additional data is required, preliminary assessment of the suitability of alternate foil and spacer materials, and specific recommendations for additional tests required to qualify new and existing insulation designs for use in the SP-100.

SECTION 2

FOIL REQUIREMENTS AND OPERATING ENVIRONMENT

Maximum hot shoe temperatures up to 1550 K are anticipated which would lead to comparable foil temperatures. Thermal cycling occurs during earth orbit with possible variation in reactor power operation while thermal decay to lower temperatures will occur as the reactor power levels decrease. The SP-100 Converter will be stored and launched in the "cold" (reactor non-operating) condition, and will not attain operational temperatures until it is exposed to the hard vacuum of space after launch. The projected foil operating life is seven to ten years.

The foil must be chemically compatible with all other materials in immediate contact with it. The final design of the SP-100 converter subsystem and selection of materials is not finalized at this time. Table 2-1 lists some thermoelectric couple and related component materials used in similar applications or proposed for use in the present project. Since the foil is not a structural load bearing component, it need not exhibit high strength at the elevated temperatures. Where the insulation is to be supported by the thermoelectric components, reduction in the foil weight will reduce stresses in the thermoelectric elements. Thermal expansion of the foil must be compatible with those of the other components supporting it in the context of a particular design to prevent excessive stresses or alignment problems under operating conditions.

Table 2-1. Thermoelectric Module Materials

Materials	Applications
SiGe	Thermoelectric Element
SiC	Possible Sublimation Control Coating and Electrical Insulation
Si ₃ N ₄	Sublimation Control Coating
Be	Possible Radiator Material
Graphite	Possible Radiator/Heat Collector Material
SiO ₂	Astroquartz Foil Spacer/Thermoelectric Element Wrap
ZrO ₂	Potential Foil Spacer
ThO ₂	Potential Foil Spacer
Y ₂ O ₃	Potential Foil Spacer

Design of the foil must consider fabricability and installation procedures. Since one of the foil layer spacer concepts being studied involves the use of tiny (less than 15 micron diameter) particles, questions pertaining to cleanliness and contamination of other spacecraft components must also be addressed. The latter factor is of concern not only during ground assembly and deployment in space, but also during the launch phase. To this end, foil behavior during vibration simulating shuttle launch must be characterized and shown to be acceptable.

SECTION 3

CURRENT FOIL TECHNOLOGY

Much experience already exists with molybdenum foil used for space based radioisotope thermoelectric generators (RTG's). Two different foil spacer concepts are currently utilized. Section 3.1 discusses the molybdenum foil insulation, and section 3.2 presents applications involving the use of alternate foil materials.

3.1 MOLYBDENUM FOIL INSULATION

RTG's currently used in the Voyager spacecraft, and those being assembled for the Galileo spacecraft, use the molybdenum foil. General Electric Advanced Energy Products Division at King of Prussia, PA, fabricates this insulation. Sixty layers of 0.0003 inch thick foil are used in these applications. Foil layers are separated by very loose weave "astroquartz" (SiO_2) cloth layers ranging in thickness from 0.002 to 0.005 inches. The entire insulation package is 0.7 inches thick, and the weight of the cloth layers is approximately half of the total insulation weight. The present G.E. assembly procedure involves punching holes in the foil, to allow thermoelectric element insertion through the foil. This is done using especially prepared templates with very sharp, ultra low clearance dies. This procedure is manual, with only a few foil layers punched at any one time. Approximately 25% excess foil material must be purchased to yield the requisite assembled insulations. Each thermoelectric element is wrapped with a quartz yarn, which provides for foil thermal expansion and electrical isolation between the thermoelectric elements and the metal foil.

This foil design is based on a design proposed and studied by the Linde Corp., at Tonawanda, NY during the 1960's and early 1970's, as discussed in Ref. 3-1. At that time, a sixty layer molybdenum foil with zirconia (ZrO_2) cloth spacer was tested by Linde. A thermal conductivity of approximately 1.0×10^{-3} BTU/hr-ft. $^{\circ}\text{F}$ through the insulation package was measured at approximately 1273 K. A shift to quartz cloth spacer was recommended for ease of fabrication. Linde has a computer program to model the thermal performance of the insulation.

As a result of the Voyager and Galileo RTG applications, much operational experience has been gained with molybdenum foil using quartz cloth spacer. Molybdenum is still considered a prime choice for the foil material due to its high melting point of 2890 K and its very stable chemical properties. Weight reduction of the Mo foil is not feasible at present, as the principal suppliers of the Mo cannot produce foil thinner than 0.0003 inches with present technology of the Sendmir rolling equipment. One major supplier estimated that an investment of several million dollars is required to design and build new rolling presses for this application. Rolling molybdenum foil to 0.0003 inches thick is a very time consuming process, ultimately resulting in a customer cost of approximately \$1200/lb. of foil (approximately \$19.12/square foot).

3.2 ALTERNATE FOIL MATERIALS

Alternate foil materials are used in a variety of applications with both the particle spacer and the astroquartz cloth. Principal incentives for the use of other foils are reduced weight, operation in different temperature

regimes, better matching of thermal expansion characteristics to the mountings, and to some extent, reduced cost.

Thermo Electron Corp. (TECO) of Waltham, MA has built and tested a zirconia coated nickel foil insulation system for use on the Ground Demonstration System (GDS) of the Brayton Isotope Power System (BIPS) (Ref. 3-2). This insulation was successfully tested at 1073 K. In addition to foil sheets and narrow one and three inch wide tapes, nickel was successfully electroplated over mandrels in very complex shapes. While this was an extremely expensive process, it was found that a vapor deposited, electroformed nickel based foil package over a complex shape was as effective as a conventional foil system which contained four times as many layers. This is primarily attributed to the facts that : 1) the electroformed pieces eliminated heat leaks at the edge joints which are present in foil systems, and 2) the vapor deposited nickel exhibited a mirror-like finish with an extremely low emissivity. The individual layers so formed for this application were 0.0007 inches thick and quite rigid.

In addition to multifoil insulation packages made of molybdenum foils which are rated to operate up to 1973 K in a vacuum furnace application, TECO also produces standard packages consisting of zirconia coated stainless steel foils which may operate to 1123 K, combination Mo foils and stainless steel foils for operation up to 1773 K, tantalum foils which may operate up to 1973 K, and combination tungsten/Mo foils for temperatures up to 2673 K.

A. Taylor of NASA at LaRC (Ref. 3-3) has a patent on a self spacing textured titanium foil system as an alternate space shuttle thermal protection system. Required foil mechanical strength in this application restricts use of the foil to 810 K. It also requires use of relatively thick foils, approximately 0.003 - 0.004 inches thick.

3.3 ALTERNATE SPACER MATERIAL

The concept of particle spacers was pioneered and developed by TECO during the late 1960's and early 1970's as discussed in Ref. 3-4. The spacer material most extensively studied and used is zirconia (ZrO_2). Particle size distribution and density have been optimized to provide the maximum thermal separation between foil layers, and thus the overall heat transfer is minimized. The particles are electrostatically deposited on foil sheets by a carefully controlled proprietary process. Weight of the zirconia spacer as applied is 0.00006 gm/cm^2 , or less than 1% of the weight of the corresponding area of 0.0003 inch thick Mo foil.

Thoria (ThO_2) has been proposed and studied as a spacer due to its exceptional stability at high temperature. Thoria is slightly radioactive, however, and as such requires special care in handling.

TECO has tested the compatibility of the zirconia spacer with niobium, nickel, aluminum and copper (Ref. 3-5). Testing indicated that a chemical reaction between ZrO_2 and niobium took place during a 168 hour test at 1173 K. It was felt this would cause long term insulation degradation in the previously discussed BIPS application. Clean nickel foils were found to be compatible with the ZrO_2 spacer during a 1000 hour test at 1273 K. Aluminum foils were compatible with ZrO_2 up to 770 K during the same test, and copper foils with ZrO_2 spacer produced acceptable results up to 1073 K. Early work

also included long term compatibility tests at high temperatures between thorium and yttria (Y_2O_3) spacer particles, and tungsten, molybdenum, tantalum, and tungsten 25% rhenium (Ref. 3-6). Fairchild has also proposed the use of aluminum and vanadium foils in the Modular Isotopic Thermoelectric Generator (MITG) Project, an advanced modular RTG design for use in space (Ref. 3-7). Use of any of the above materials in the present application is contingent on long term compatibility with thermoelectric element and other related materials. This testing has not yet been performed.

3.4 LATEST FOIL PACKAGES

TECO produces metal foil based insulation under the patented trade name Multi-Foil. The principal terrestrial application for Mo/ZrO₂ particle spacer multifoil is as thermal insulation in high temperature vacuum and inert gas furnaces (Ref. 3-8). It has also been proposed for use in a container of LiAl/FeS batteries for an electric car (Ref. 3-9). It is currently being studied at Fairchild Corp. at Germantown, MD, for use in the MITG project. Innovative insulation design and fabrication techniques are being developed for the MITG project. The thermoelectric and insulation components are undergoing extensive performance and long term materials compatibility testing at Fairchild. The Mo/ZrO₂ insulation package is generally providing satisfactory performance and operating characteristics.

SECTION 4

POSSIBLE FOIL MATERIALS

As discussed in Section 1, the primary goal of this study was to reduce weight over the present state-of-the-art foil insulation, the best of which is based on 0.0003 inch thick Mo foils with the TECO type particle spacer (being tested by Fairchild for the MITG project). Aside from reduction in the number of foil layers (which is a systems optimization consideration), weight reduction may take place from reducing either the weight of the foil layers or the spacer material. Since the foil is by far the heavier component, weight reduction efforts should practically center on development of alternate foil materials.

4.1 STUDY APPROACH

An independent study was undertaken of all elements to identify additional potential foil materials for use in the SP-100 converter application. It is important to recognize that the foil weight per unit area is the density-thickness product; thus, minimum produceable foil thickness is an important consideration. The very thinnest of foils is almost always rolled on Sendzimir type rolling mills. Even though many commercial firms own this equipment, production of metal foils less than 0.0005 inches thick is uncommon, and most suppliers will not guarantee uniform results much thinner than this. In general, factors such as metal ductility, annealing characteristics and availability of vacuum annealing facilities, and experience with the particular metal establishes fixed lower bounds on foil thickness. In the case of Mo, this physical limit is 0.0003 inches. The goal is thus to identify high temperature materials whose density times minimum foil thickness product is less than that for molybdenum.

All of the elements were considered in the present study. Eliminations were made for the following reasons:

- 1) Melting points less than 1273 K (to provide a margin above the estimated 900 K cold junction temperature for the SP-100 converter);
- 2) Rare earth metals, since these are usually reactive in air even at room temperatures.
- 3) Radioactive materials, due to handling/safety problems.
- 4) Extremely rare and precious metals, due to prohibitive cost.

In addition, beryllium was eliminated since it can only be produced in very small sizes in thicknesses of 0.0003 inches, at a cost of nearly \$100/square inch, and has a high vapor pressure and strength problems at temperatures approaching its melting point (1282°C).

Melting points and densities were compiled for the remaining elements. Extensive industrial contacts were made throughout the U.S. and with some foreign based material suppliers to determine the minimum foil thickness

which has been produced in the past, estimates (when available) of minimum thickness possibly attainable with a best effort, and foil cost. In addition a "figure of merit" was computed for each material. This consisted of the density-minimum foil thickness product divided by that for molybdenum, and as such, represents the fractional weight advantage per unit area over a Mo foil. Radiation heat transport through the foil insulation is emissivity dependent. Emittance effects were not included in the Figure of Merit, however, as the values are relatively similar for all metal foils considered. The grafoil and carbon have much higher emittances, and the use of reflective coatings will be considered.

4.2 FOIL MATERIAL EVALUATION

Results of this study are presented in Table 4-1. This table indicates that carbon, a graphitic material known by the trade name Grafoil (produced by Union Carbide), copper, niobium, titanium, vanadium, and zirconium either are or might be produced with significant weight per unit foil area savings versus molybdenum. Of these, copper was dropped from further study relative to the other six materials because of its much lower melting point and limited usefulness in the present application. Of the carbon and grafoil (which is also a form of carbon), the grafoil offers the greater weight reduction payoff of the two; thus, only the grafoil will be further considered. Finally, zirconium and vanadium exhibit virtually identical melting points and densities. Discussions with Teledyne Wah Chang Corp. of Albany, OR, (TWCA) indicates that the two metals are similar chemically, but zirconium forms an oxide layer only a few angstroms thick in air which protects the surface from further oxidation, whereas vanadium forms a thick greenish oxide layer in air at room temperature. Also, vanadium is approximately 10 times more expensive per pound than zirconium, and it has not as yet been rolled thinner than 0.0004 inches by Teledyne Wah Chang. The recommendation was made to primarily consider the plentiful, inexpensive zirconium which is currently available rolled to 0.00022 inch thicknesses in 12.0 inch width rolls. As a result of the above considerations, it is now recommended that four materials be chosen for further intensive study as potential foil replacements for molybdenum; grafoil, niobium, titanium and zirconium. The three metals possess melting points ranging from 1933 K to 2741 K, and offer potential weight reductions per unit area over molybdenum foil of from 44% to 85%. The grafoil is a non metallic alternative with extremely high temperature capabilities, albeit with a higher emittance.

A concern raised by R. Marsh of TWCA was the extent of grain growth in zirconium and other ultra thin foils at high temperature for long time periods. He postulated that thermal cycling, which will result from SP-100 nuclear reactor power variation, might cause microcracks to develop in the foil at grain boundaries. Even if this occurs, the effect on the characteristic thermal conductance of the foil insulation is unclear, since virtually no structural loads are placed on the insulation, once launched, in the SP-100 configuration.

Some key thermophysical properties of titanium, zirconium, niobium, and the special lightweight grafoil, originally presented in Ref. 4-1, are listed in Table 4-2. Vapor pressures and emittances contained in Ref. 4-2 and 4-3 are indicated in Figures 4-1 and 4-2. Emittances shown in Figure 4-1 are approximate, and should more accurately be determined for the as-produced thin foils. Other material characteristics are discussed below.

Table 4-1. Potential Mass Reductions of Various Foil Materials

Material	Melting Point (K)	Density (gm/cm ³)	Thinnest Foil Already Produced		Thinnest Foil Which May Be Produced	
			Thickness (in)	Figure of Merit*	Thickness (in)	Figure of Merit*
Carbon	3823	1.8-2.3	0.001	0.59		
Grafoil	3823	1.12-0.64	0.005	1.83	0.002	0.42
Copper	1356	8.96	0.0002	0.58		
Hafnium	2500	13.31	0.001	4.34		
Iron	1808	7.87	0.0005	1.28		
Molybdenum	2890	10.22	0.0003	1.0	0.0003	
Nickel	1726	8.90	0.0005	1.45		
Niobium	2741	8.57	0.00035	0.978	0.0002	0.56
Tantalum	3269	16.65	0.0002	1.08	0.0002	1.08
Titanium	1933	4.54	0.0001	0.15	0.0001	0.15
Tungsten	3683	19.3	0.002	12.59		
Vanadium	2113	6.11	0.0004	0.8	0.0002	0.40
Zirconium	2125	6.56	0.00022	0.47	0.00022	0.47

*Figure of Merit = $\frac{\text{New material density} \times \text{foil thickness}}{\text{Molybdenum density} \times \text{foil thickness}}$

Table 4-2. Key Thermophysical Properties of Alternate Foil Materials

	Mo	Ti	Nb	Zr	Grafoil
Density (gm/cm ³)	10.22	4.54	8.57	6.56	0.64 0.035*
Thermal Conductivity (W/cm-K)	1.39	0.22	0.53	0.232	0.43 **
Specific Heat (cal/gm-K)	0.058	0.123	0.064	0.0671	0.311***
Melting Point (K)	2890	1933	2741	2125	3823
Emittance at 1273 K	0.14-0.2	0.29	0.18	0.22	0.5
Coefficient of Thermal Expansion (K ⁻¹ x 10 ⁻⁶)	5.4	8.5	7.0	5.6	-0-

*For 1.12 g/cm³ grafoil, through thickness direction

**For 1.12 g/cm³ grafoil, in plane

***For 1.12 g/cm³ grafoil

Figure 4-1. Emittances of Selected Materials

Figure 4-2. Vapor Pressures of Selected Materials

4.2.1 Titanium Foil

Titanium foil has been produced by Titanium Industries of Fairfield, NJ, in continuous rolls, 0.0001 inches thick by 3.75 inches wide, 0.0002 inches thick by 4.0 inches wide, and 0.0005 inches thick by 12.0 inches wide. Edge trimming prevents production of the thinner foils in wider rolls.

Cost for the 0.0001 and 0.0002 inch thick foils is \$8.19 and \$3.81 per square foot, respectively. The extreme low density of titanium coupled with the thin foil gages offers the greatest potential of any of the candidate materials for weight reduction versus molybdenum. Availability of these foils from Titanium Industries is currently quoted as six weeks from the receipt of the order, with a required minimum order of approximately 10 lbs. (for threading through the rolling mill). An initial quantity of 10 lbs. of delivered foil would thus cost \$34K and \$8K for the 0.0001 and 0.0002 inch thick foils, respectively. Teledyne Wah Chang Corp. is also willing to attempt to roll titanium as thin as possible on a best effort basis.

Titanium is reactive at high temperatures. It will react to some extent with nearly any oxide, nitride, or carbide. The degree of chemical reaction depends upon the stability of the compound in contact with it and the temperature. It is relatively stable when in contact with zirconia particles at elevated temperatures in a vacuum. Titanium is highly pyrophoric; that is, it produces large amounts of heat during reactions with oxygen, and will thus burn in air. It is the only element that will also burn in pure nitrogen. The larger the surface area to volume ratio, the more easily the material may be ignited; thus, the extremely thin (0.0001 inch thick) foil may pose safety concerns and lead to special handling and processing precautions.

The crystal structure of titanium is hexagonal close packed (alpha phase). A transition to body centered cubic (beta phase) takes place at approximately 1153 K.

4.2.2 Zirconium Foil

Zirconium foil is currently available from Teledyne Wah Chang Corp. in 0.00022 inch thickness, up to 12 inches wide. A fixed price of \$62/lb. is quoted for the rolled foil (\$0.46/square foot) with a minimum order of 10 lbs. (\$620) if the width desired is not in stock. Foil production and delivery is currently 6-8 weeks. The minimum order for inventoried foil of 4 inch width is \$150.

There are several areas of concern which have been identified for zirconium foil.

- 1) Embrittlement will occur if excessive oxygen absorption occurs.
- 2) Zirconium will absorb oxygen from some oxides.
- 3) Zirconium foil may react with silicon nitride when they are in contact. (Note: Silicon nitride is the coating currently being used on the SiGe thermoelectric couples.)
- 4) Grain growth in foils may present a problem

5) Zirconium is pyrophoric.

These concerns are discussed below with possible solutions to the concerns.

The zirconium from which foil is rolled is designated "reactor grade," and corresponds to ASTM standard R60001. This grade is the purest produced. It typically contains approximately 700-800 ppm of oxygen. The oxygen content is critical, as zirconium will also react with most oxides, nitrides and carbides to some extent at elevated temperatures. It is most sensitive to oxygen, and is an excellent oxygen getter. Absorption of large amounts of oxygen, hydrogen, carbon or nitrogen will cause embrittlement. No special handling or storage is necessary in air at room temperature, as the extremely thin surface oxide layer prevents excessive oxidation. Care must be taken to insure that high temperature testing be performed only in a hard vacuum.

Zirconium will absorb the oxygen in zirconia particles and quartz yarn or cloth at high temperatures. W. Phillips of JPL has suggested that a different type of particle spacer, for example zirconium nitride, ZrN, might be much more stable in contact with the zirconium foil. TECO suggested that the slightly radioactive thoria particle spacer should be stable with the foil. It has also been suggested that yttria be used as a spacer.

The concern was also raised by M. Eck and A. Schock of Fairchild that zirconium foil would react if placed in physical contact with the silicon nitride (Si_3N_4) moelectric coating, impacting its effectiveness as a sublimation preventative. An alternate sublimation control coating, zirconium nitride (ZrN) was suggested by W. Phillips of the JPL Materials Laboratory. Zirconium nitride is a common, chemically stable, extremely hard material which is easily sputter or CVD coated onto surfaces. It is also an excellent electrical insulator at elevated temperatures. The difference in coefficient of thermal expansion between ZrN and SiGe is approximately the same as between silicon nitride and SiGe. It is known that the silicon nitride coating develops cracks at elevated temperatures over time. It is hoped that zirconium nitride will be less susceptible to cracking, and less affected when any of the more reactive alternate foil materials are used. Zirconium nitride is currently commercially applied by CVD onto tool bits.

Zirconium, like titanium, is pyrophoric. G. Kressman of GE has had much experience in dealing with 0.0008 inch thick Zr foil which is shredded into wire for use in flashbulbs. He indicates that it can be made to burn by the heat of a match. No special precautions are necessary during machining operations, other than good lubrication to maintain a low temperature, and safe storage of tiny chips. He also indicates that every effort should be made during punching and shearing operations to produce clean cuts free of sheared edges. It may be more practical to use electric discharge machining (EDM) to produce thermoelectric element penetrations, with subsequent degreasing, as currently done by TECO in production of molybdenum multi-foil insulation. L. Montgomery of the JPL flight safety office does not foresee major problems with its use in the SP-100 spacecraft application.

Zirconium, like titanium, has a hexagonal close packed crystal structure below 1138 K and becomes body centered cubic at higher temperatures. R. Sutherland, a metallurgist at Teledyne Wah Chang, does not expect this transition to cause irreversible detrimental material property changes, even with

frequent thermal cycling.

4.2.3 Niobium Foil

Niobium is also available from Teledyne Wah Chang. Although they have only rolled the foil to 0.0004 inch thickness in a 4.0 inch width, they are willing to attempt to roll it down to 0.0002 inches, or the lowest practical limit, in a 12 inch width with a best effort type of contract. Cost of the rolled foil would be in the range of \$120 - \$160 per lb. The higher figure results in a cost per square foot of \$1.43 for 0.0002 inch foil. A ten pound minimum order for niobium foil might thus cost \$1200 - \$1600, and take 6-8 weeks.

Niobium is another highly reactive metal. It reacts at elevated temperatures with oxygen, nitrogen, chlorine, and hydrogen. It is particularly sensitive to oxygen embrittlement. As already discussed in Section 3, TECO has found that chemical interactions between niobium and zirconia take place at 1173 K (Ref. 3-5). Thoria, yttria or some other particle spacer may prove acceptable. Other corrosion resistance properties are similar to tantalum. It possesses a body centered cubic structure at all temperatures.

4.2.4 Grafoil

Grafoil is manufactured by Union Carbide Carbon Products Division, Cleveland, OH. It is a calendered form of natural graphite. The purest grade, GTA, is 99.9% graphite, with the remainder ash. Commercially produced grafoil has a nominal density of 1.1 gm/cm³, or approximately half that of pure carbon. It is manufactured in a pliant sheet form down to 0.005 inches thick in rolls up to 2 feet wide by up to 100 feet long.

P. Petrunich of Union Carbide has produced samples of grafoil manufactured in small quantities in thicknesses of 0.002 inches. This appears to be the lower limit on foil thickness. As a calendered product, however, its density may be varied. It was indicated that a density of approximately 0.64 gm/cm³ may be achievable. This thickness-density product produces a sizeable weight benefit per foil layer versus molybdenum. Cost to produce several hundred square feet of this special grafoil product in 1 foot x 2 foot sheets was estimated at between \$5000 - \$10000. Larger production runs might cost less per square foot, since most of the cost is in the initial set up.

It is interesting to note that Union Carbide already markets a grafoil heat shield for furnace applications. It consists of alternating layers of flat and corrugated grafoil sheets. A 21 layer foil with a graphite felt backup has been successfully tested at temperatures to 2673 K.

Vapor pressure considerations restrict continuous grafoil vacuum operation to approximately 2000 K. In the SP-100 application, weight reduction would probably necessitate use of only the flat grafoil sheets separated by an appropriate spacer. It has been found that reactions between zirconia and carbon in vacuum begin at nearly 1570 K. Other materials may be used as spacers at higher temperatures.

The three metal foils exhibit two major advantages over the grafoil:

1) Their emittances are low, similar to that of molybdenum (0.2 - 0.3), whereas the emittance of grafoil is relatively high (0.5 - 0.6). Even though foil emittance is of secondary importance in restricting radiant heat transfer through foil insulation, the high grafoil emittance might require more foil layers to achieve the same insulating characteristics as metal foil insulation. Thus, the lower weight per foil layer of grafoil versus molybdenum might be partially offset by the need for more layers.

2) Chemical reactions at high temperatures between the grafoil and the other thermoelectric materials might be excessive and undesirable.

Note that both grafoil disadvantages may be overcome by a thin coating of a low emittance, less reactive metal, such as, for example, rhenium. A coated grafoil should be reserved for expanded investigations due to possible cost and schedule impacts.

SECTION 5

PROPOSED TEST PROGRAM

The only metal foil which has been successfully and completely tested at 1408 K in a thermoelectric application is the Mo/SiO₂ weave combination used on the Voyager and Galileo spacecraft RTG's. The Mo/ZrO₂ particle spacer combination has been tested by TECO for the BIPS application, and is being tested by Fairchild at 1270 K for the MITG project for extended times. Additional information is required to validate the other foil/coatings combinations listed in this report. As discussed in Section 4 the grafoil is a higher risk alternative foil than the previously discussed metal foils. As a result it is recommended that grafoil not be tested in the initial test series.

TECO has run tests with thoria (ThO₂) and yttria (Y₂O₃) particle spacers (Ref. 3-6). These were tested with molybdenum, tungsten, tantalum, and tungsten 25% rhenium foils at 1973 K and 2173 K in vacuum for up to 2,000 hours. While no gross chemical reactions occurred between any foil/oxide combination, there was some sticking of foil layers. Thoria was considered superior to yttria because slightly less sticking resulted at the test temperatures. Thoria is slightly radioactive, however, so that personnel safety considerations require special precautions during handling and testing. By contrast, yttria is not radioactive and is nearly as stable chemically as thoria. Simple chemical stability calculations based on free energy of formation indicate that yttria should be stable with all three alternative metal foils, although the elevated temperature at which significant reaction begins to occur is best determined experimentally. General consensus of Reference 3-6, J. Hagarth of Teledyne Wah Chang Albany, F. Huffman of TECO, and W. Phillips of JPL, indicate that yttria is an excellent choice for particle spacer in the present test series, which will not exceed 1573 K. Thus, yttria will be used exclusively in the proposed tests. Future use of thoria will only be considered if yttria reacts excessively with any of the alternative foil materials.

The present test series also will seek to prove the feasibility of zirconium nitride as an alternate sublimation control coating on thermoelectric elements. This coating may be especially useful when used in conjunction with zirconium, titanium, or niobium based foil insulation systems.

5.1 INITIAL PROPOSED TEST SERIES

Tests to be performed in the proposed test program are as follows.

1) Long term chemical compatibility tests are required between titanium, niobium, and zirconium foil with yttria spacer, in conjunction with silicon nitride coated silicon-germanium thermoelectric elements wrapped in quartz yarn. It is recognized that a complete thermoelectric converter for the SP-100 will include other materials, such as, for example, graphite heat collectors, and carbon-carbon or beryllium radiators. Compatibility tests between the alternate foils and these materials would require a far more extensive (and expensive) testing matrix. Further, the extent of reactions may be highly dependent on final converter panel design configuration. This is considered beyond the scope of the proposed program, and will not be tested at this time. Each test article will include a single nonfunctional SiGe

thermocouple coated and wrapped with quartz yarn in a configuration similar to that used in the Voyager and Galileo RTG's. This thermocouple will be placed in a close fitting cut-out in a 20-30 layer foil blanket of approximately 15-20 square inches in size. This size is considered the minimum necessary to obtain accurate thermal conductance measurements (see test #2, below). The foil in each test article will be wholly comprised of either zirconium, titanium, or niobium foils separated by yttria particles. One of each type of foil assembly will be tested at three operating temperatures and for test durations of 2,000 and 6,000 hours. Since detailed post test examination is destructive, a total of 18 test article assemblies are required for these tests. The multiple operating conditions are necessary to permit quantitative extrapolation of chemical reaction rates at other temperatures and longer durations. All test articles may be tested simultaneously in a single large vacuum chamber. Assemblies being tested at the same temperature will be placed in common temperature controlled furnace chambers within the vacuum chamber. Testing of all samples will begin simultaneously. A chamber break will occur at 2,000 hours to remove and evaluate appropriate test assemblies. Testing will then continue for an additional 4,000 hours on the remaining articles.

2) Effective thermal conductance tests on the alternate foil material samples is required at the beginning and end of chemical compatibility tests, and comparison with a molybdenum foil sample with zirconia spacer used as a reference. A single test station will be designed and built to measure heat flux through the foil assemblies. Prior to initiation of the long term compatibility tests, and also just prior to final destructive evaluation, the thermocouple assembly in each test article will be replaced with a plug of insulation (to prevent excessive heat loss) during heat flux measurements. This testing will be conducted in a separate vacuum chamber, and at temperatures not to exceed those of the long term compatibility tests. Effective foil insulation thermal conductance changes will be plotted for each sample as a function of hot side temperature and test duration. Results will permit comparison of the effectiveness of various foil/spacer combinations.

3) Long term stability tests of zirconium nitride coated thermoelectric elements should be performed. Bare SiGe thermocouple units will be sputter coated with zirconium nitride. These test articles will be tested at three temperatures for 2,000 and 6,000 hours. Testing will take place in the same furnaces concurrently with the compatibility test. Six assemblies are required for this test, with three additional units reserved for initial determination of coating parameters. Destructive post test analysis will determine the effectiveness of the zirconium nitride as a sublimation control coating.

4) Long term chemical compatibility tests are necessary between zirconium foil with yttria spacer, in conjunction with zirconium nitride coated thermoelectric elements wrapped with zirconia yarn. Test assemblies will be similar in design to those used in the long term chemical compatibility tests described above. Zirconium foil/yttria spacer patches will undergo testing at three temperatures for 2,000 and 6,000 hours. These samples will be placed in the same furnaces used for test #1 and will be destructively examined in the same way. Chemical reactions and reaction rates occurring in these samples will be compared with results of the silicon nitride coated thermocouples of test #1. No thermal heat flux measurements are planned for these six test articles.

5) A thermal cycling test should be done on selected thermally aged foil samples. This test will make use of some individual layers of zirconium,

niobium, and titanium foils spared from destructive evaluation at the conclusion of the high temperature, 6,000 hour, compatibility tests. Samples will be cycled 10, 20 and 50 times between ambient and the highest temperature reached during the compatibility tests. Qualitative observations will be made of crack propagation at grain boundaries and foil brittleness.

A test matrix, summarizing the test article make-up and test conditions, is presented in Table 5-1.

5.2 TEST DATA

Specific data to be determined from the above tests are listed below.

1) Determine the extent of all chemical reactions occurring. This will be accomplished by extensive destructive testing of aged thermoelectric element samples. Test methods will include the following.

- a. Scanning electron microscopy (SEM), which will identify most chemical species that have been formed as a result of thermal aging due to initial impurities, mass diffusion, etc., and also determine diffusion penetration and coating thicknesses.
- b. X-ray diffraction, which will identify chemical compound formations on the surfaces.
- c. Energy dispersion for chemical analysis and X-ray diffraction (EDAX) as part of the SEM test. Samples are bombarded with electrons, and the resulting X-ray spectra produced identify individual elements present. Since EDAX testing applies only to very small sample areas, it will be used selectively based on SEM results and statistical considerations.
- d. Photomicrography, which will yield data on gross material configuration changes, which are visually observable.

2) Get chemical reaction rates. This data will be obtained by plotting the extent of reactions as functions of test temperatures and test duration. This will permit prediction of chemical reactions occurring at temperatures and for time periods somewhat beyond the test ranges.

3) Obtain measurements of the heat flux through foil samples which will permit determination of effective thermal conductance.

4) Evaluate coating parameters for effective sputter coating of zirconium nitride onto thermoelectric materials.

5) Observe by photomicrography the thermal cycling induced cracks along the (enlarged) grain boundaries as a function of number of thermal cycles.

Many of the above tests are inter-related within the overall test plan. A breakdown of individual tasks required for the entire proposed test program is shown on Fig. 5-1.

Table 5-1. Text Matrix

Test	Sample No.	Test Article				Test Duration (Hrs.)	Test Temperature (°C)
		Insulation Foil Material	Insulation Spacer Material	Thermocouple Coating	Thermocouple Yarn Wrap		
1. Material compatibility, & thermal conductance	1.	Ti	Y ₂ O ₃	Si ₃ N ₄	SiO ₂	2,000	700
	2.	"	"	"	"	"	900
	3.	"	"	"	"	"	1100
	4.	"	"	"	"	6,000	700
	5.	"	"	"	"	"	900
	6.	"	"	"	"	"	1100
	7.	Zr	"	"	"	2,000	700
	8.	"	"	"	"	"	950
	9.	"	"	"	"	"	1200
	10.	"	"	"	"	6,000	700
	11.	"	"	"	"	"	950
	12.	"	"	"	"	"	1200
	13.	Nb	"	"	"	2,000	900
	14.	"	"	"	"	"	1100
	15.	"	"	"	"	"	1300
	16.	"	"	"	"	6,000	900
	17.	"	"	"	"	"	1100
	18.	"	"	"	"	"	1300
	19.	Mo	ZrO ₂	None	None	0	-
2. ZrN coating effectiveness	20.	None	None	ZrN	None	2,000	900
	21.	"	"	"	"	"	1100
	22.	"	"	"	"	"	1300
	23.	"	"	"	"	6,000	900
	24.	"	"	"	"	"	1100
	25.	"	"	"	"	"	1300
3. ZrN coating effectiveness & material compatibility	26.	Zr	Y ₂ O ₃	ZrN	ZrO ₂	2,000	700
	27.	"	"	"	"	"	950
	28.	"	"	"	"	"	1200
	29.	"	"	"	"	6,000	700
	30.	"	"	"	"	"	950
	31.	"	"	"	"	"	1200

Figure 5-1. TEST TASKS

Task
1. Prepare detailed test plan.
2. Purchase Ti, Zr, Nb foils.
3. Purchase thermocouples
4. Fabricate foil samples.
5. Sputter coat ZrN onto thermocouples.
6. Prepare furnaces, vacuum chamber.
7. Fabricate foil sample thermal conductivity test apparatus.
8. Foil sample thermal conductance tests.
9. Long term testing.
10. Destructive sample testing.
11. Thermal cycling testing.
12. Reporting.

5.3 TASK DESCRIPTION

A brief description of each of the tasks shown in Figure 5-1 is now given.

1) Prepare a detailed test plan. This task includes:

- a. Design of test articles (dimensions of foil samples to be fabricated, based on timely availability of thermoelectric couples, test fixtures, etc.
- b. Specification of furnace, vacuum chamber, and instrumentation requirements.
- c. Definition of detailed test procedure, including the test matrix and detailed schedule.

2) Purchase Ti, Zr, Nb foils. TWCA has indicated that a 10 pound minimum order of 0.00022 inch thick Zr foil will cost \$62/pound = \$0.62K. They also estimated that a 10 pound minimum order of niobium foil, rolled to 0.00020 inch thickness, or as thin as possible on a best effort basis, would cost approximately \$160/pound = \$1.6K. Titanium Industries estimated that a 10 pound minimum order of titanium foil of 0.00010 inch thickness would cost approximately \$3.4K/pound = \$34K.

3) Purchase thermocouples. It is anticipated that reject thermocouples will be purchased from the Advanced Energy Products Division of General Electric Corp. (Valley Forge, PA) from the existing Galileo RTG flight thermocouple production line. Table 5-1 indicates that 18 coated couples, and 15 uncoated ones, are required.

4) Fabricate foil samples with a yttria spacer. Table 5-1 indicates that six niobium, six titanium, and 12 zirconium foil insulation packages with removable center insulation plugs are required as discussed in Section 5.1, test number 2. A single molybdenum foil/zirconia spacer sample will also be needed to be used as a test reference.

5) Sputter coat zirconium nitride onto thermocouples. Twelve thermoelectric elements must be coated, with initial setup, experimentation and evaluation.

6) Prepare furnaces and vacuum chamber. This task involves initial furnace and chamber cleaning and set-up, and instrumentation set-up. Also included is special preparation of one selected furnace for high temperature (1573 K) operation.

7) Fabricate foil thermal conductivity test apparatus. Design, fabrication, testing, and calibration of the test rig are included in this task.

8) Foil thermal conductance testing. Thermal conductance tests will be performed at the beginning and end of the long term material compatibility tests. In the interest of cost reduction, only the zirconium, titanium, and niobium foil packages with the silicon nitride coated thermocouples will be tested, along with the single molybdenum foil package as a reference.

9) Long term testing. Once the samples have been installed in the furnaces and the vacuum chamber pumped down, daily checks will be made to insure proper operation of all equipment.

10) Destructive sample testing. The SEM, X-ray diffraction, EDAX, and photomicrography examinations are performed in this task.

11) Thermal cycling testing. Photomicrography of these samples is assumed to be included within task #10.

12) Reporting.

5.4 FOLLOW-ON TESTING

Data obtained from some of the tests proposed in Section 5.1 is considered necessary to prove initial feasibility of lightweight zirconium, titanium and niobium foil insulation packages in thermoelectric applications. The use of quartz yarn wrapped, silicon nitride coated SiGe thermocouples in the tests is based on proven Voyager and Galileo RTG designs. Similar configurations are considered possible in future SP-100 thermoelectric converter designs. Materials and the final design configuration of the SP-100 converter are not yet defined. Full validation of any foil insulation will require extensive material compatibility, thermal performance, and other tests with complete prototypical units. This paragraph describes some of the additional data requirements.

In the event that the alternate foil material/yttria particle spacer combinations evaluated in the proposed tests are not chemically compatible, testing with thorium may be considered. Additionally, feasibility tests utilizing coated or uncoated grafoil layers may be undertaken, with a concomitant determination of how thin grafoil may be produced. These are believed to be higher risk foil alternatives than the metal foils with yttria spacer, however. If conducted in the future, the long term material compatibility and thermal conductance tests described in the proposed test program should be repeated with these materials.

Once viable lightweight foil insulation systems are identified, further data is required to support design, fabrication, and detailed performance analysis activities. This includes:

1) Data on edge heat loss versus number of foils. This will improve analytical heat transfer performance prediction capabilities and permit better converter design optimization. This data may be provided by heat flux measurements through foil insulation samples composed of, for example, 20, 40, and 60 foil layers. Test articles might be fabricated of single or double overlapped foil sections to simulate foil discontinuities present in large converter panels. Peripheral edge length should also be varied.

2) Study of insulation fabrication procedures. Ultra thin metal foils, of the type used in foil insulation, are typically not available in widths greater than 12 inches. Titanium foil, 0.0001 inch thick, is only presently known to be available in 3.75 inch width rolls. Fabrication and assembly procedures must be developed.

3) Determination of foil handling and fabrication procedures to

ensure personnel/spacecraft safety. Foil materials identified in the proposed tests are nontoxic and noncarcinogenic. The thin foils are, however, pyrophoric. Fabrication/handling/storage procedures must be identified to minimize the flammability hazards. In addition, if thoria spacer is found to be necessary, a study should be made to assess its radiological safety impact. Special fabrication/handling/storage procedures would then have to be developed.

4) Assessment of prototypical foil/spacer particle assembly behavior during launch. This may be accomplished by subjecting a prototypical converter assembly panel to simulated launch loads. Pre- and post shake test thermal performance data should be taken to assure an adequate design. In addition, ceramic spacer particle movement should be studied to identify possible problems with particle containment and component/spacecraft contamination. A related study should focus on the impact of clean room requirements when foil insulation with ceramic particle spacer is used.

5) Long term, high temperature materials compatibility data for a prototypical converter assembly panel for a minimum of three different test durations, possibly up to 10,000 hours, at higher than anticipated operating temperatures. This accelerated testing is necessary to assure converter panel operation for the 7-10 year design life of the SP-100 spacecraft.

6) Long term, high temperature thermal performance data for a prototypical converter assembly panel. Foil insulation thermal performance degradation over time, possibly resulting from emittance changes or chemical reactions with other components, must be assessed.

SECTION 6

SUMMARY

Presented herein are results of a study to:

- 1) Assess the present state-of-the-art of Multi-Layer Foil Insulation of the type which may be used in the SP-100 Thermoelectric Converter.
- 2) Determine deficiencies in the data base.
- 3) Evaluate alternate foil and spacer materials.
- 4) Propose a series of tests to be performed to prove initial feasibility of some alternate foil materials.

The current state-of-the-art of foil insulation consists of the insulation used in space based thermoelectric applications in the Voyager and the Galileo Radioisotope Thermoelectric Generators (RTG's). This foil has sixty layers of 0.0003 inch thick molybdenum foil, separated by loosely woven quartz (SiO_2) cloth. Weight of the quartz cloth is approximately equal to that of the metal foil.

An alternative spacer technique, pioneered by Thermo Electron Corp., involves foil separation by application of tiny particles of a low thermal conductivity material. Spacer materials recommended include zirconia (ZrO_2), the slightly radioactive thorium (ThO_2) and yttria (Y_2O_3). This method of foil separation reduced the weight of the spacer to less than 1% of the foil weight.

Further weight savings in the range of 44% to 85% appear possible through use of alternate lower density, thinner foil materials. Titanium, zirconium, niobium, and a graphitic product have been identified as possible alternative foil materials. Substantial foil testing is required to prove feasibility of the recommended alternate foil materials and to characterize performance. Initial data required to establish feasibility of the lightweight insulation approach includes:

- 1) Determination of how thin niobium foil may be rolled.
- 2) Determination of the chemical compatibility of niobium, zirconium, and titanium foils separated by yttria particle spacer as applied by a proprietary process developed by Thermo Electron Corp. (TECO). Also to be determined is the chemical compatibility of these foil systems with a silicon germanium thermocouple, coated with silicon nitride sublimation control coating, and wrapped with quartz yarn.
- 3) Determination of feasibility of an alternate, possibly superior, sublimation control coating, zirconium nitride, for use on silicon germanium thermocouples. Also to be examined is the chemical compatibility of yttria coated zirconium foil with zirconium nitride coated thermocouples.
- 4) Determination of effective thermal conductivities of the foil samples, and evaluation of test induced degradation.

- 5) Evaluation of thermal cycling effects on the ultrathin thermally "aged" foils.

These proposed tests are included as part of a larger framework of test data required to eventually validate a particular foil design. Data obtained from these tests will indicate directions for future testing, and may lead to eventual development of a very lightweight foil insulation tailored for use in the SP-100 and other space based thermoelectric systems.

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