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DART Technology Development Final Report

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by

K. C. Cooper
W. C. Turner

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DART TECHNOLOGY DEVELOPMENT
FINAL REPORT

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K. C. Cooper and W. C. Turner

ABSTRACT

Technology development for a Decomposed Ammonia Radioisotope Thruster (DART) of 0.01 to 0.1 lb thrust was performed at the Los Alamos Scientific Laboratory (LASL). Design, materials and fabrication, and testing results are presented here.

Assembly of a rhenium heat exchanger, including nozzles and inlet tubes, was accomplished with electron beam welding. A useful series of molybdenum-rhenium alloys with from 20 to 46 wt % rhenium was prepared, using electron beam melting for purity. Molybdenum foam was brazed to molybdenum sheet. Diffusion couples were tested at operating conditions, and compatibility for 500 h was established between: carbon vs beryllia, carbon vs tungsten, iridium vs molybdenum-rhenium, zirconia vs tungsten, zirconia vs molybdenum, hafnia vs tungsten, and hafnia vs molybdenum.

Testing showed the adequacy of the graphite reentry ablation structure. Electrically-heated tests demonstrated adequacy of the multi-foil thermal insulation, and of the heat pipes for pre-launch cooling. Energy absorption data were obtained on foam and drilled molybdenum, which are promising as cushioning for the radioisotope fuel against impact. The original impact capsule, of threaded molybdenum, was inadequate against impact, but a welded molybdenum-rhenium capsule behaved satisfactorily. Rhenium was a satisfactory catalyst for decomposition of the propellant ammonia.

INTRODUCTION

Los Alamos Scientific Laboratory (LASL) has been developing the technology of a Decomposed-Ammonia Radioisotope Thruster (DART) for low-thrust application on spin-stabilized Air Force satellites. Development of the DART was started by Thompson Ramo Wooldridge (TRW) in 1966 under contract to the Air Force Rocket Propulsion Laboratory (AFRPL). The TRW contract was completed in 1968 with the demonstration of satisfactory operation of two radioisotope-heated DARTs in a vacuum chamber. In 1969, cognizance of DART development was transferred from the Air Force to the U. S. Atomic Energy Commission (AEC), which chose LASL to continue the development of DART technology.

LASL's work was funded by the AEC through June 1971. Subsequently, the project was jointly funded

through FY 1972 by the AFRPL and the Space and Missile Systems Organization (SAMSO).

This final report summarizes the work performed by, and under the cognizance of, LASL on the DART project.

The design specifications given by the Air Force to LASL are shown in Appendix A. The most significant of these specifications are:

- Three thrust levels, 0.01, 0.05, and 0.1 lb.
- Intermittent operation with maximum on-time of 0.2 s and equivalent full-time thrust from zero to 0.005 lb.
- Propellant exhaust temperature 2500°F at minimum heat removal, corresponding to a specific impulse of 310 s.
- Propellant exhaust temperature 2000°F at maximum heat removal, corresponding to a specific impulse of 280 s.

- Operating lifetime, seven years.

The specifications were substantially identical to those for the TRW DART design, except for the temperatures which had been increased for the LASL design by 500°F. These higher temperatures required LASL either to select or to develop materials with higher temperature resistance and to test them for compatibility.

Another important difference, though not contained in the original LASL specifications, was the change from PuO_2 microsphere fuel in the TRW design to solid PuO_2 disks, as developed for the TRANSIT and PIONEER space programs. These disks required a different approach to designing the impact capsule, which has to withstand high temperatures and must be compatible with a variety of materials for long periods of time, in addition to its primary function of protecting against the release of the highly toxic plutonium fuel in case of launch-pad accident or earth impact after atmospheric reentry. Extensive safety testing of the design was required.

LASL's work was concentrated on the incorporation of compatible materials in an impact-resistant design which would be proven in extensive testing. Neither the testing with radioisotope nor thrust-performance testing was part of LASL's assignment. The latter tests, performed with ammonia in an electrically-heated DART furnished by LASL, were conducted at Edwards Air Force Base, AFRPL, and will be reported by them.

DESIGN

LASL was responsible for the development of DART technology, not for the development of a flyable design. However, a reference design was needed on which to base the choice of materials and tests. General design features are described; details and alternative designs are discussed. Analyses performed in the course of design are referenced in this section.

General Design Features

The reference LASL DART design is shown in Fig. 1. The design is neither optimized nor complete; some modifications that could be made will be mentioned in subsequent sections of this report. The following description starts from the center line and proceeds radially outward.

Fig. 1. LASL DART - reference design - Thermo Electron insulation.

- Fuel, pressed plutonium oxide (PPO) in the form of disks.
- Fuel can, of molybdenum-rhenium, contains the fuel.
- Impact capsule, also made of molybdenum-rhenium, surrounds the fuel can.
- Ablation structure, of ATJS graphite, completely surrounds the impact capsule.
- Heat exchanger, of rhenium in the shape of a sleeve with internal flow passages, surrounds most of the cylindrical portion of the ablation structure.
- Standoffs, of tungsten wire, separate the rhenium from the graphite ablation structure.
- Inlet tubes for ammonia, made of rhenium, enter at one end of the heat exchanger; there is one tube for each of the three parallel flow passages.
- Nozzles of rhenium are attached to the other end of the flow passages, one nozzle to each flow passage.
- Multifoil insulation of molybdenum surrounds the heat exchanger and ablation structure, except for necessary penetrations.
- Outer can surrounds the insulation, again with necessary penetrations.
- Bolting flange is in the plane of the three nozzles.

The internal components of the DART are supported mainly axially, and the following description of additional design features will proceed from the fuel axially outward.

- Foam cushions of molybdenum are adjacent to the fuel can, just inside the impact capsule.
- Standoffs of beryllia support the impact capsule and, in turn, are supported by the graphite ablation structure.
- Standoffs of tantalum separate the graphite from the molybdenum insulation and the centering bushings.
- Centering bushings of zirconia penetrate the insulation and are supported by the outer can.
- Heat pipe holes penetrate the outer can and insulation and terminate inside the ablation structure.
- Pressure-relief device and
- Post-impact oxidation protection shell are not shown in this design.

Design Details

The design details mentioned above are discussed below. Extended discussions of materials, fabrication processes, and compatibility problems are presented elsewhere in this report.

Fuel. The AEC, in view of funding limitations, specified that LASL work on the DART would be limited to providing an electrically-powered test device for simulated operational tests. No radioisotope would be used in this testing. A decision was made to design around the TRANSIT fuel, which was being intensively developed at the time. This fuel was originally PuO_2 in solid solution with ZrO_2 or ThO_2 bound in a molybdenum matrix. It was later changed to pressed plutonium oxide (PPO). The fuel, in the form of circular 0.2-in.-thick wafers with a diameter of 2.2 in., has a heat-generating capacity of 40 W per wafer. The DART design was based on six wafers, for a possible power of 240 W. Later it was recognized that 150 W was closer to optimum; the design (if it were to be developed into a flight article) should therefore be changed by:

- Reducing the fuel to four disks, which would shorten the DART; or
- Reducing the diameter of the fuel and fuel can, and adding foam molybdenum inside the cylindrical portion of the impact capsule; or
- Downgrading the fuel thermal output by incorporating, e.g. zirconia or thorium.

Fuel Can. The fuel can prevents contamination from the highly toxic plutonium fuel during

fabrication, assembly, and handling of the DART. It also serves as a secondary capsule to help contain the fuel in case the impact capsule ruptures during an impact. The first material to be used was TZM molybdenum because of its compatibility with the fuel and because of its high-temperature properties. However, satisfactory welds could be obtained only with difficulty and the weldments were brittle at room-to-moderate temperatures; molybdenum-50 wt % rhenium was therefore substituted for the original TZM. Molybdenum-rhenium has all the desirable properties of TZM, plus good weldability and ductility at lower than room temperature. The LASL fuel can had no perforations; however, if the can were to contain radioisotope fuel fine holes should perforate the can to permit the escape of helium gas generated by the heat producing α -decay of the plutonium over the lifetime of the unit.

Impact Capsule. The impact capsule protects against the release of the toxic plutonium fuel in case the fuel can is damaged during earth impact after reentry or is damaged during a launch mishap. The capsule fulfills its function by absorbing kinetic energy (to minimize breaking up of the fuel), and remaining physically intact after impact (to prevent the escape of fine fuel particles produced during impact). Initially the LASL DART had a TZM molybdenum impact capsule with a threaded joint (Fig. 2). Safety tests, however, showed that the threaded joint was too weak, and that the ductile-to-brittle transition temperature was too high. Molybdenum-50 wt % rhenium was, therefore, chosen to replace the TZM, because it can be welded well and is ductile to below room temperature. Its compatibility problems are about the same as those for molybdenum. The welded molybdenum-rhenium capsule performed well in safety tests.

The optimum composition of the molybdenum-rhenium alloy, for both the impact capsule and the fuel can, was of some concern: a lower rhenium content results in lower costs and better machinability, whereas a higher rhenium content facilitates welding, results in lower ductile-to-brittle transition temperatures, and in better low-temperature impact resistance. Molybdenum-rhenium alloy of varying composition was therefore developed and tested, as discussed later.

Fig. 2. Impact capsule - threaded joint.

Ablation Structure. The prevention of isotope dispersal during reentry into the atmosphere (as a result of failure to obtain orbit during launch or because of orbital decay) or after earth impact was a primary objective. The DART consequently had to be protected against overheating and attendant loss of impact resistance during reentry. A graphite ablation structure was practically the only possible design choice, particularly because AEC safety personnel had advised to avoid any penetrations in the ablation structure to accommodate coolant tubes and nozzles. This meant that the ablation structure had to be between the impact capsule and the heat exchanger and insulation, exposed to high temperature for years of DART operation.

This design and ATJS graphite were chosen on advice from Sandia Laboratories, Albuquerque, NM, based on their experience in space isotope reentry protection. (This specific grade of graphite is not crucial to the design, and other grades may even offer advantages in performance). A thickness of 0.5 in. was chosen to allow for the possibility that some of the material would have to be removed on the inside and replaced with insulating graphite, e.g., pyrolytic graphite, to protect the impact capsule against overheating during reentry. This type of insulation should be considered for a flight DART.

The threaded end plugs alleviate thermal stresses at the intersection of the cylindrical section and the flat ends; these stresses would occur during reentry in a stable attitude (i.e., side-on) with attendant nonuniform heating.

Heat Exchanger. The heat-exchanger design is shown in Fig. 3. In arriving at this design, the following requirements were considered:

- Gaseous ammonia must be heated from 70°F to between 2000 and 2500°F.
- The ammonia must be catalytically decomposed in the heating process to N_2 and H_2 .
- The pressure drop must be reasonable, consistent with an inlet pressure of 50 psia.
- The volume of contained gas must be minimized, to permit a quick response to the opening and closing of the inlet valve.
- The design must be rugged and easily fabricated.
- Propellant leakage should be minimized, but need not be zero.

Fig. 3. Rhenium heat exchanger.

- The surface area for radiation heat transfer must be sizable.
- The heat capacity for intermittent high heat removal must be fairly large, to minimize temperature drop during pulsing.

The high temperature requirement limited the choice of material to the refractory and noble metals. Molybdenum was eliminated because of its tendency to nitride, and tantalum because of hydriding. Molybdenum-rhenium was considered of dubious value because of its molybdenum content, while platinum had to be rejected because it is not a good catalyst for ammonia decomposition.¹ Osmium, while a good catalyst,² was not considered because of scarcity and brittleness.

Tungsten and rhenium were the main contenders. Tungsten, although a good catalyst,¹ was not selected because it is too brittle for easy fabrication. Rhenium was chosen because it is compatible with ammonia and its decomposition products and because it showed promise of being a catalyst at DART operating temperatures;^{3,4,5} besides, it is ductile at low cryogenic temperatures. It was fabricated successfully, as detailed later, and its decomposition properties were found to be satisfactory.

To minimize both pressure drop and contained gas volume, tube-type gas passages were needed. However, because the heat exchanger operates in vacuum, heat input is primarily by thermal radiation (secondarily by conduction) and a large surface area is required to minimize the temperature drop for heat input to the exchanger. These requirements for a large surface area plus the need for relatively high heat capacity led to the concentric-sleeve design shown in Fig. 3. Flow passages were grooved into the inner sleeve before the sleeves were welded together.

Standoffs. These tungsten parts are necessary to separate the rhenium from the graphite for two reasons:

- On heating up, the rhenium expands away from the graphite, and spacers prevent localized line contact with anomalous temperatures.
- There is a chemical interaction between rhenium and graphite as discussed elsewhere in this report, and tungsten is compatible with both materials.

The geometry of the standoffs is not important. In the electrically heated LASL test unit, wires were placed in axial grooves in the graphite and were then captured by the heat exchanger.

Inlet Tubes. These tubes are made of rhenium because of its welding compatibility with the heat exchanger material. The tubes interface through fittings with the ammonia supply system from which the propellant is released, as required, through electrically operated valves to the proper inlet tube/flow passage/nozzle combination.

Nozzles. Three rhenium nozzles are welded to the heat exchanger. Rhenium was chosen because of its high temperature resistance and because of its welding compatibility. The internal profile was based on TRW's experience with low-thrust, low-Reynolds'-number nozzles. The thickness of the wall in the expanding section was minimized to reduce heat losses by conduction along the walls and subsequent radiation to space.

Multifoil Insulation. In the vacuum of space, reflective insulation by far surpasses in effectiveness any other kind. TRW's DART design used relatively few layers of separated self-supporting molybdenum sheets. Molybdenum was retained in LASL's design because of its high-temperature strength, low vapor pressure, relatively low emissivity, and compatibility with other DART materials. However, the design concept was changed to numerous thin foils of reflecting metal separated by oxide spacers and contained within cans. This approach was adopted for the following reasons:

- More effective insulation was needed to hold heat losses down, because the new design was to operate at higher temperatures, with resulting tendency toward higher heat losses both through the insulation and through joints, penetrations, and supports.
- Reflectivity decreases with increasing temperature, requiring more layers.
- The possibility existed that the effectiveness of the insulation would decrease with time, making it desirable to overdesign.
- The AEC had sponsored development of multifoil insulation by Thermo Electron Co. and by Linde Division of Union Carbide Co.; LASL took advantage of the findings.

Thermo Electron insulation is used in the design shown in Fig. 1. Linde insulation, shown in Fig. 4, is similar, but thicker. Both are compared in Table I. Further design and performance details are given in the Testing Section of this report and in Appendix B.

Outer Can. The DART assembly is contained within a stainless steel can. The can consists of two cup sections, joined with a bolting flange that is used for mounting the DART. Springs in the ends of the outer cans squeeze the cup sections of insulation together. The bolting flange was incorporated mainly to ease assembly and disassembly during testing. In a flight design, a different flange would allow insertion of the radioactive fuel after mounting the DART on the satellite.

Foam Cushions. These cushions should reduce fuel breakup during impact. Molybdenum foam of 35% theoretical density was chosen for compatibility reasons and because of its high-temperature properties.

A metallic bond is desirable between the cushions and the impact capsule, to minimize temperature gradients in some alternative designs. The brazing operation joining these components is described elsewhere in this report.

Standoffs. Standoffs are needed between the impact capsule and the graphite ablation structure as a load support. These standoffs must be compatible with both materials at high temperature. They can be thermal conductors. Beryllia was found to be the best material.

Fig. 4. LASL DART - Linde insulation.

TABLE I
COMPARISON OF THERMO ELECTRON AND LINDE INSULATION
FOR LASL DART

	Thermo Electron	Linde
Foil material	Mo	Mo (hot zone) Ni (cooler zone)
Foil thickness	0.0005 in.	0.0005 in.
Spacer material	ZrO ₂	ZrO ₂ (hot zone) SiO ₂ (cooler zone)
Spacer geometry	Powder, bonded to foil	Cloth
Spacer thickness	0.001 in.	0.005 in.
Joints	Butt	Stepped
Number of foils	90	78
Bulk thickness	0.12-0.18 in.	0.75 in.

Tantalum standoffs are shown in Fig. 1 between the graphite and the centering bushings. This material was used successfully in the testing program, but a change to beryllia may be desirable.

Centering Bushings. These bushings have to support a load and to insulate thermally, as they extend from the hot zone to the cool outer can. Zirconia and hafnia are the only known prospective materials under these conditions; zirconia was used for DART laboratory testing.

Heat Pipe Holes. Holes for insertion of heat pipes are provided for prelaunch cooling; the pipes would be removed before launch. Tests showed that two water heat pipes are adequate.

Pressure Relief Device. At the start of the LASL program, such a device was being developed for other space isotope applications; it was assumed that one would be available for the DART. Without such a device the pressure buildup in a sealed impact capsule due to helium production associated with α -decay would not be tolerable at the high operating temperatures. Release of all the helium to space is undesirable from the standpoints of heat transfer from the fuel to the impact capsule, and suppression of volatilization of fuel and other materials inside the impact capsule.

Post-Impact Oxidation Protection Shell. It was recognized that a special oxidation-resistant shell might be needed to prevent release of fuel from the fuel can at the high post-impact temperatures expected ($>1000^{\circ}\text{F}$). This problem was investigated in other space isotope projects and therefore was not considered in LASL's effort.

Design Analysis

Analytical work consisted of stress and thermal analyses. Stress analyses were performed principally on the outer can which was designed with a bolting flange to withstand axial accelerations of 10 g's. The can and attached insulation-positioning springs were calculated by standard methods and computer codes.

Most of the analytical effort was spent on thermal analyses, concerned with determining the temperature distribution throughout the DART, and with studying the heat transfer and pressure drop in the propellant flow passages, both for steady state and transient conditions.

Methods of Analysis. Temperature distributions were calculated conservatively by assuming radiation transfer only, neglecting conduction transfer from the fuel outward to the heat exchanger. However, from the heat exchanger to the outer can, heat losses through joints in the insulation and by conduction through inlet tubes, nozzles, and standoffs were calculated. It was felt that the assumptions for the temperature-distribution model contained large uncertainties so that the calculations did not merit refining. Reliance was rather placed on testing, which is covered later in this report.

The flow passages and nozzles were sized by conventional hand-calculations, aided by TRW's DART design. Because heat removal from the heat exchanger is intermittent whereas heat input is steady, the transient conditions were determined by more elaborate calculations. The flow of dissociating ammonia through the largest of the three flow channels was investigated by transient analysis.⁶ This channel provides the largest fluid heat requirement and the greatest heat drain on the channel walls. Results indicated that the ammonia dissociates completely, reaches 2500°F at the exit of the channel, and maintains that temperature as the flow becomes steady. In addition, the wall temperature drop in the heat exchanger is insignificant. The valve was assumed to open in 6 msec and the starting transient was essentially complete 15 msec later.

Several items were investigated further.⁷ Changes in the turbulent heat-transfer correlation had insignificant effect. Flow in the large channel is largely in the laminar-turbulent transition region where analytical representations of heat

transfer are subject to large errors. Experimental work is required to improve the degree of confidence.

Dissociation was assumed to occur at a fixed temperature of 1500°F in initial calculations. Using the method given in Ref. 8, continuous dissociation was assumed, corresponding more closely to the real physical case. The difference was insignificant.

The effect of reducing the heated length of the flow channel from 15.5 to 12.5 in. was also minor.

Thermal Properties. The thermodynamic properties of ammonia and dissociation products were presented in Ref. 9, which includes plots of enthalpy, specific heat, viscosity, and thermal conductivity vs temperature. Dissociation is assumed to occur at 1500°F, with the heat of dissociation being 1414.5 Btu/lb. Data were taken from Refs. 10, 11, and 12. Enthalpy vs temperature curves for ammonia and dissociation products are shown in Figs. 5 and 6.

FABRICATION

Once the preliminary geometry of the DART had been designed, the areas where fabrication and materials development was needed were chosen. The present section covers the fabrication work.

Outer cans, insulation systems, heat exchangers, ablation shields, impact capsules, fuel cans, and

Fig. 5. Enthalpy of ammonia vs temperature.

Fig. 6. Enthalpy of dissociated ammonia ($\text{NH}_3 + 1/2 \text{N}_2 + 3/2 \text{H}_2$) vs temperature.

incidental hardware were fabricated for test purposes. The only components requiring advances in fabrication technology were the heat exchanger, the impact capsule, and the fuel can. The heat exchangers to be tested were made of both molybdenum and rhenium, and the fuel cans and the impact capsules were made of molybdenum and molybdenum 50 wt % rhenium alloy. All of these parts were thus fabricated from just one binary series of materials. Fabrication problems were twofold: (1) production of sheet and forgings, and (2) conversion of such materials into actual parts.

Properties of Materials

Molybdenum. Molybdenum possesses only a few of the characteristics normally considered to adversely affect fabricability. Essentially, these are low elongation and poor solubility of non-metallic impurities. The disadvantage of low elongation can be overcome by processing at 300-800°F, i.e., above the ductile-to-brittle transformation temperature.

The solubility of impurities in molybdenum is:¹³

Hydrogen	0.1 ppm
Carbon	0.1-1
Nitrogen	1
Oxygen	1

Impurity atoms above this limit migrate into the grain boundaries, with the following effects:

- Absolute values of elongation in the brittle range vary inversely with impurity level.
- The ductile-to-brittle transition temperature at any particular strain rate is directly proportional to impurity level.
- Weldability varies inversely with impurity level. It is always beneficial to preheat the molybdenum into the ductile range prior to welding, but microcracking upon subsequent cooling can be avoided only if the level of impurities is low.

The above three effects are directly dependent on the thickness and concentration of the impurity atoms in the grain boundaries. Therefore, improvements can be made either by reducing the number of impurity atoms or by increasing the total grain boundary surface area. The latter approach is utilized by developing the smallest possible grain. This is achieved by coldworking and partially recrystallizing the metal while it is in bar or sheet form. Ideally, the final stress relief heat treatment would provide full recovery and partial recrystallization (to a 10-20% level).

The above factors make it desirable to obtain a fine grained, high purity metal and to maintain these characteristics through processing.

Some of the literature states that the two common alloys of molybdenum (0.5% Ti and TZM) are less subject to impurity effects than pure molybdenum in that the titanium acts as a getter. We do not so interpret the data and consider neither alloy of help in offsetting impurity effects, although proposed many years ago for such a purpose. TZM though remains as a useful alloy for another reason: the titanium and zirconium additions raise the recrystallization temperature, making it possible to forge molybdenum below the recrystallization temperature and thereby to readily produce cold worked shapes. Another advantage is the possibility of obtaining a very strong alloy by the controlled addition of carbon and very careful processing with the effect

of dispersing carbide through the matrix. Manufacture of such an alloy, however, requires such difficult techniques as water quench from very high temperatures.

Rhenium. Although no data on the solubility of gases in rhenium were found, apparently rhenium in this respect is equivalent to tantalum and niobium and has a solubility for oxygen, carbon, and nitrogen about 30-100 times that of molybdenum. Rhenium is thus a refractory metal whose fabricability is not impaired by impurity effects and which, moreover, is ductile at room temperature.

Molybdenum/Rhenium Alloys. Superficially it might appear that adding rhenium to molybdenum would increase the solubility of gases, at least in proportion to the amount of rhenium added. The manner of dispersion of the resultant nonmetallics, as long as they remain in the matrix, can be disregarded.

The literature generally states that this is not the case, at least not for alloys containing considerably less than 50 wt % rhenium. However, one has to keep in mind that most of these data have been obtained on powder metallurgy materials which have high levels of impurities. It was therefore decided to investigate molybdenum/rhenium alloys of vacuum-melt materials. Arbitrarily selected were additions of 20, 30, and 46 wt % rhenium. The latter amount of rhenium was chosen instead of the usual 50 wt % because of the proximity of the brittle sigma-phase boundary line. This investigation was performed by Oak Ridge National Laboratory and is discussed elsewhere in the report.

Fabrication of Parts

Three heat exchangers were made from molybdenum and three from rhenium, all to precisely the same design. The molybdenum items were made in the following manner:

- Concentric cylinders were spun at 700°F from flat sheet.
- The inner cylinder was machined by milling.
- Nozzles and fittings were machined with single-point tools.
- Welding was done by electron beam, with details preheated to 700°F.

No problems resulted.

The rhenium heat exchangers were expected to be as easily fabricated, but were not. The rhenium,

as received in platelets, contained the following admixtures:

Iron	10 ppm
Chromium	3
Silicon	30
Carbon	140
Oxygen	17

As converted to sheet by Cleveland Refractory Metals, the outer-cylinder stock contained:

Iron	30 ppm
Chromium	10
Silicon	70
Carbon	700
Oxygen	19

The following procedures were used to fabricate the parts:

- The cylinders were made by rolling and welding. The inner cylinder posed no problems, but the outer cylinder could not be butt-welded. Severe pitting resulted. Several platelets were rolled down by General Electric to obtain sheet with better composition, as indicated above. This sheet was subsequently worked and welded successfully. Mandrel sizing was used on the outer cylinder to obtain a good fit. The outer cylinder was then vacuum annealed, i.e., hopefully recrystallized, at 1260°C. (This was an estimate based on T_M . We have recently discovered in Ref. 14 that 1200°C is correct.)
- The inner cylinder was pierced and slotted by electric discharge-machining (EDM) and was trimmed by grinding.
- Nozzles and fittings were machined from fabricated blanks. Nozzles were ground on the outside diameter, pierced, and finished by EDM on the inside diameters, and trimmed by grinding. Only EDM gave problems. Obviously, EDM is accomplished by particles breaking free once the surface reaches the melting range. The combination of high melting point and a tiny part to be machined, necessitating a small electrode, caused problems. Probably EDM should not be used for parts smaller than the 0.016 in. nozzle.
- An attempt to make rhenium filler wire was unsuccessful. The rhenium platelets were melted by electron beam, but a drop casting developed microcracks before or during hot rolling. However, vacuum melting did purify the rhenium, as shown by wire analysis:

Iron	10 ppm
Chromium	3
Silicon	9
Carbon	14
Oxygen	11

The wire could probably have been successfully made by incorporating a hot extrusion stage after drop casting.

- The original tubing was made by forming two halves, welding, and drawing. Additional tubing was later made by vapor deposition on a mandrel by Ultramet. This latter technique was so successful that a similar approach should have been used for the nozzles.
- Electron-beam-welding was a marginal technique for repairing pinhole leaks. For a follow-on program, it is recommended that vacuum furnace brazing be developed for leak repair. This would be routine, but empirical. The braze alloy, of course, has to withstand service life in a vacuum.

Capsules and Fuel Cans. The fuel cans were made from both molybdenum and molybdenum-50 wt % rhenium alloy by rolling and welding, to use LASL capabilities. A better method would have been spinning. The GTA welded closures were apparently satisfactory.

Impact capsules were made from both plate and sheet. Sheet capsules were made only from molybdenum, by hydroforming the ends at 150-200°F and spinning the cylinders at 700°F. Closures by electron beam welding were routine. Plate capsules were routinely hogged from molybdenum bar or machined from die-formed molybdenum-50 wt % rhenium plate.

A method of brazing molybdenum foam to molybdenum was developed and is described elsewhere in this report.

MATERIALS DEVELOPMENT

The materials development comprised three separate activities:

- Testing of materials couples for diffusion.
- Development of molybdenum-rhenium alloys of varying compositions.
- Brazing of foam molybdenum.

Details of the work on these three subjects are presented in this section.

Diffusion Couples

The need for diffusion barriers can be easily visualized by examining Fig. 1, which shows the DART as originally designed at LASL. Differing requirements for the various details necessitate use of dissimilar materials. Because of the severe time and temperature conditions required of the DART, the compatibility or resistance to diffusion and reaction between adjacent materials is of concern. Therefore, an investigation was required as to mutual compatibility of any adjoining candidate materials. For cases where any choice of candidates is incompatible, a third material, one compatible with both of the candidates, has to be introduced at the interface, i.e., a diffusion barrier.

Investigation of compatibility between fuel and other materials in the thruster was outside the scope of LASL DART work. (See, for example, Ref. 15 for work on TRANSIT fuel.)

Referring still to Fig. 1, it can be seen that the molybdenum-rhenium fuel can is in occasional contact with the foam molybdenum and the TZM impact capsule. The TZM impact capsule is in constant contact with zirconium carbide (later changed to beryllia), and in occasional contact (if permitted) with graphite. The graphite ablation structure, in turn, is in contact with both rhenium (heat exchanger) and tantalum (standoff). Finally, the zirconia (center bushing) contacts tantalum (standoff) and molybdenum (foil insulation), and zirconia, at low temperature, contacts the stainless steel parts of the outer can.

Another diffusion problem, not apparent in the figure, may arise. It may be necessary to protect the fuel can within the impact capsule with an oxidation barrier. The oxidation barrier would be made from wrought sheet, in a manner very similar to the fuel can. Candidate materials for the oxidation barrier are iridium and a platinum-rhodium-tungsten alloy.

Test. Virtually no published data (as far as we could ascertain) exist as to diffusion characteristics of prospective DART materials over a long period of time at 1400°C. It was therefore decided to test in vacuum "buttons" (couples) of these materials, in contact with each other, at 1300, 1400, and 1500°C for 500 h. This work was done at ORNL. Each test capsule contained only one couple. All

graphite couples were tested in open graphite capsules, whereas non-graphite couples were tested in open metallic capsules, with the capsule made of one of the materials, or equivalent. Two capsules were sealed before test: one contained beryllia and molybdenum, the other beryllia and tungsten. The latter experienced major mass transfer from one end to the other end of the tungsten, and thereby justified the decision to use unsealed capsules.

The material couples run were:

Graphite vs	iridium tantalum rhenium molybdenum-50 wt % rhenium tungsten molybdenum platinum-rhodium-tungsten
Zirconia vs	molybdenum tungsten tantalum
Hafnia vs	molybdenum tungsten tantalum
Beryllia vs	rhenium molybdenum-50 wt % rhenium platinum-rhodium-tungsten tungsten molybdenum zirconia
Iridium vs	molybdenum-50 wt % rhenium
Molybdenum vs	rhenium platinum-rhodium-tungsten
Tungsten vs	rhenium molybdenum-50 wt % rhenium

The materials and their pairing, as listed above, were chosen because of the following reasons:

- Tungsten, tantalum, and beryllia were tested with graphite as prospective diffusion barrier materials.
- Rhenium, molybdenum-50 wt % rhenium alloy, and molybdenum were tested with graphite to verify that barriers are indeed necessary.
- Platinum alloy was tested with graphite in case an oxidation barrier outside the impact capsule should be desirable.
- Hafnia was run as an alternative to zirconia. Both materials are poor thermal conductors, as desired.
- Tungsten was run against beryllia, in case a dual diffusion barrier was proven necessary by the other tests.

Results. The results of tests made at 1400 and 1500°C are presented in Appendix C. Visual

examination of the couples tested at 1300°C confirmed results expected from the higher-temperature tests and no tabulation is therefore given.

Both beryllia and tungsten appear to be effective diffusion barriers for carbon, whereas tantalum is not. Both molybdenum and rhenium certainly need such a barrier when coupled with graphite. The platinum alloy may not require such an interface.

Iridium appears to be satisfactory in contact with a molybdenum-rhenium alloy. However, more data for both iridium and platinum are necessary as coupled with molybdenum, molybdenum-rhenium alloy, beryllia and tungsten.

Both zirconia and hafnia seem to be acceptable for the centering lushing, but the weight loss of both materials has to be investigated further. Both are compatible with tungsten and molybdenum. Compatibility of both materials with beryllia is well known and therefore was not tested. The weight loss was surprising, especially because it exceeds any loss that can be explained by routine phenomena such as outgassing. It might have been caused by some discrepancy in the testing procedure.

Molybdenum-Rhenium Alloy Development

Molybdenum is an obvious choice as an impact capsule material and as a fuel can material because of its high yield strength at high temperature, and because of its compatibility with PuO_2 fuel. However, molybdenum has two significant disadvantages:

- It has a low solubility for gaseous impurities and is therefore difficult to fabricate, and
- It is brittle below ~500°F at very high strain rates.

Alloys of molybdenum and rhenium can be prepared which retain the desirable properties of molybdenum without the disadvantages. However, rhenium is very expensive (~\$2000/lb), and also decreases the machinability of the alloy with increasing rhenium content. The usual alloy, which contains 46 wt % rhenium, did not seem to be of optimum composition for the intended application. LASL therefore awarded a contract to Oak Ridge National Laboratory (ORNL) to determine the ductility and strength of molybdenum-rhenium alloys over a range of lower rhenium contents. Their main conclusion was that a useful series of alloys exists within the range of 20 to 46 wt % rhenium, with the final choice of a

particular composition dependent on cost, machinability, temperature, and impact-rate requirements.

Experimental Procedure. Molybdenum-rhenium alloys containing 20 and 33 wt % rhenium were electron beam melted and drop cast into small rolling billets using previously developed techniques. Rhenium plates and low-carbon arc cast molybdenum were used as starting materials. The rhenium was purified by electron beam melting before alloying with the molybdenum. Chemical analysis of the starting materials is given in Table II. Each alloy composition was electron beam melted three times to obtain homogeneity. The alloys were then drop cast into a 1/2 x 1 x 2-1/2 or 5-in. long water cooled copper mold. An alloy casting and mold are shown in Fig. 7.

Because a fine grain structure casting is produced by this technique the casting could be rolled to sheet. An initial breakdown temperature of 800°C

TABLE II
IMPURITY ANALYSES OF STARTING MATERIALS

Impurity	Analyzed Material, ppm		
	Re, as received	Re, EB melted	Mo, arc cast
Iron	10	6	5
Chromium	3	3	2
Silicon	30	9	5
Carbon	140	14	22
Oxygen	17	11	8

Fig. 7. Mold and drop casting used for electron beam melted molybdenum-rhenium alloys.

was used with final rolling at room temperature to 300°C. All of the sheet was rolled to 0.040 in. thickness. A total reduction of 92% was obtained with intermediate stress relief anneals at 800°C. The sheet material of each composition was recrystallized 1 h at 1400°C. Strips 1/2 in. wide and 6 to 8 in. long were cut from the sheets. Welding was performed both by electron beam and by gas-tungsten arc (GTA) with molybdenum-46 wt % rhenium filler metal. The ductility was evaluated by standard 4T bend tests at room temperature, 0, -78°C, and -196°C. Tests on the base metal were performed on 1/2 x 1-1/2 in. specimens. The butt welded specimens were cut to 1 x 1-1/2 in. and were tested transverse to the weld joint. A test speed of 1 in./min deflection rate was used for all tests.

Results and Discussion. The nominal alloy compositions and analyzed rhenium contents are given in Table III. Electron beam melting caused the preferential vaporization of molybdenum which increased the final rhenium content of the alloy by 3 to 4 wt %. The loss of molybdenum is not expected to be as great in larger heats of material, but some overcharge of molybdenum would be necessary to achieve the desired composition.

Rolling of the castings to 0.040 in. sheet was easily accomplished. Both alloys could be cold rolled after a 50% reduction at 800°C. The recrystallization anneal of 1 h at 1400°C produced an average grain diameter of 30 µm in both alloys (Fig. 8).

No difficulties were encountered in producing butt-welded joints by automatic electron beam welding. However, some cracking occurred during normal GTA welding with molybdenum-46 wt % rhenium filler wire. The cracks always initiated at the start or

TABLE III
COMPOSITION OF MOLYBDENUM-RHENIUM ALLOYS

Melt No.	Nominal Alloy Composition, wt %	Analyzed Rhenium Composition, wt %
5728	Mo-20% Re	22.5
5747	Mo-20% Re	23.2
5750	Mo-20% Re	22.9
5729	Mo-33% Re	36.5
5751	Mo-33% Re	37.4
5753	Mo-33% Re	37.2

Fig. 8. Microstructure of molybdenum-23 wt % rhenium sheet, recrystallized 1 h at 1400°C. 100X

interruption in the manual weld pass. In Fig. 9 centerline weld cracks in the area of an interruption are shown in the upper photograph. When a smooth weld pass was accomplished, no cracking was observed as shown in the lower photograph.

The bend test results on base metal and welded samples are given in Table IV. Ductility of the base metal down through -78°C was good, as expected. Ductility of the electron beam welded samples was also good, as anticipated. In fact, these samples are so promising that it is recommended that molybdenum-10-12 wt % rhenium materials be considered as a good engineering refractory. (ORNL is doing so for the Isotope Cladding Program.)

The ductility of the GTA welded samples is poor and not to be considered as inherent for the materials and processes. There was insufficient material to wring out the process. The alloy with 37 wt % rhenium should exceed the alloy with 23 wt % rhenium

Fig. 9. Gas-tungsten arc weld in molybdenum 37 wt % rhenium; molybdenum-46 wt % rhenium filler metal. The upper weld contains centerline cracks starting from craters.

TABLE IV
BEND TEST RESULTS ON MOLYBDENUM-RHENIUM ALLOYS

Rhenium Content, %	Conditions	Bend Angle at Indicated Test Temperature, °C			
		20	0	-78	-196
23	Base metal	90	90	90	0
	EB welded	90	90	80	0
	GTA filler	90	30	0	0
37	Base metal	90	90	90	0
	EB welded	90	90	90	0
	GTA filler	20	0	0	0

in low temperature ductility. Among the possible reasons for the poor results compared to electron beam welding are 1) larger heat affected zone in the GTA welds, 2) possibly impure filler wire, 3) greater impurity pickup with gas atmosphere, 4) rhenium enrichment due to volatilization and segregation resulting in sigma-phase formation in the weld zone. Metallographic examination of a weld in the 37% alloys supports item 4. A dendritic structure was observed in the weld zone (Fig. 10) and the hardness of the weld metal was 420 DPH vs 260 DPH for the base metal. Although a second phase could not be directly identified, the observed hardening could easily have caused the decreased ductility in this material. (Impurities can also harden a refractory.)

The results clearly demonstrate the ease with which electron beam welding can be accomplished as compared to GTA welding. In all probability, the

Fig. 10. Microstructure of GTA weld with molybdenum-46 wt % rhenium filler metal in molybdenum-37 wt % rhenium base metal. 50X

results also indicate that filler wire composition of 46 wt % rhenium is too high and that a composition of 35 wt % rhenium would be much better.

Conclusions. Molybdenum-rhenium alloys between 20 wt % rhenium and 46 wt % rhenium can readily be produced provided one attains the purity levels resulting from electron beam melting.

The exact weight percent of rhenium should be determined by the design strain rate and temperature required by the DART fuel can and impact capsule.

At least down through -78°C, electron beam welding results in little degradation of base metal properties.

Brazing of Molybdenum Foam

In one alternative design, incorporating a sheet metal impact capsule (Fig. 11), the foam cushions may have to be brazed to the capsule for good thermal contact. Although vacuum brazing of wrought molybdenum (or molybdenum-rhenium alloy) is done routinely, a brazing investigation was conducted to explore the effects of using foam as a parent metal. Problems could have resulted from either continuous outgassing of the foam during brazing or from failure to adequately clean the faying surface of the foam.

Fig. 11. Alternative impact capsule.

The braze alloy has to survive years of service at 1400°C in vacuum. To insure long life under such conditions, compositions are used that have low vapor pressures and will diffuse readily in both directions with the parent material. Partial or complete closing of the voids in the foam at the faying surface is not only acceptable, but preferable.

Results. The problem was attacked in two ways. Direct brazing was used first. However, the molybdenum foam contained too many extremely large voids. The brazing filler metals flowed by capillary action into the foam rather than along the joint.

The second method consisted of precoating the joint before brazing. An attempt to prepare the surface of the foam by applying a thin layer of a higher temperature brazing filler metal was tried, but the braze alloys were unable to sufficiently close the voids to permit an adequate, subsequent brazed joint. However, several successful brazes were made with molybdenum foam that had been precoated either by plasma spraying or by CVD techniques. The CVD technique appears to give the best surface for brazing.

Inverted T-joints of molybdenum foam to molybdenum sheet were made by using several ORNL-developed high temperature brazing filler metals. Data for three of the most promising filler metals are shown in Table V. The lowest temperature brazing alloy (Ti-54 wt % V-25 wt % Cr) was used to braze molybdenum foam that had been precoated by plasma spraying. As seen in Figs. 12 and 12A, the filler metal adequately wet and flowed along the joint. However, porosity and some cracking are present in the braze.

The other two brazing filler metals (V-20 wt % Ta-25 wt % Nb and Ti-42.5 wt % Zr-15 wt % Ta) were used to braze CVD-coated molybdenum foam. Figures 13, 13A, 14, and 14A show the excellent joints that were obtained. There were essentially no problems with porosity or cracking.

TABLE V
BRAZING MOLYBDENUM FOAM

<u>Filler Material</u>	<u>Brazing Temperature, °C</u>	<u>Technique for Coating Foam</u>
Ti-21 V-25 Cr	1550	Plasma sprayed
Ti-42.5 Zr-15 Ta	1800	CVD
V-20 Ta-25 Nb	1900	CVD

Fig. 12. Molybdenum foam brazed to molybdenum sheet with Ti-54 wt % V-25 wt % Cr filler.
7-1/2X

Fig. 12A. Detail of Fig. 12. 50X

Fig. 13. Molybdenum foam brazed to molybdenum sheet with V-20 wt % Ta-25 wt % Nb filler.
7-1/2X

Fig. 13A. Detail of Fig. 13. 50X

All three joints were tested in vacuum of 10^{-5} torr for 500 h at 1500°C and remained intact.

TESTING

The LASL DART development included testing to establish:

- Safety with respect to release of fuel.
- Heat flow and temperature distribution.
- Heat pipe performance.
- Energy absorption data on foam and drilled molybdenum.
- Catalytic activity of rhenium for ammonia decomposition.

Details of the work performed on these five subjects are presented in this section.

Safety Testing

The DART safety problem is the same as that of other radioisotope systems: How to ensure that the highly toxic radioactive fuel is not released to the biosphere. This is particularly important for respirable particles less than 3 μ in size.

Plutonium-238 fuel is not difficult to contain in fabrication, handling, or storage, but two situations exist for which containment must be demonstrated: (1) launch accidents, and (2) reentry

Fig. 14. Molybdenum foam brazed to molybdenum sheet with Ti-42.5 wt % Zr-15 wt % Ta filler. 7-1/2X

through the atmosphere followed by earth impact. The DART safety-testing program concentrated on these two situations.

Launch Accidents. The tests made under this category included:

- Shock-tube overpressure tests,
- Fragment impact tests, and
- Solid-propellant fire test.

Atmospheric Reentry and Earth Impact. These tests included:

- Arc-jet reentry heating simulation,
- Air-drop terminal-velocity determination,
- Simulated ground impact of TZM capsule,
- Overflight and reentry of molybdenum-rhenium capsule, and
- Simulated ground-impact comparison of designs.

Most of these tests were either conducted or supervised by Sandia Laboratories, Albuquerque, NM. Only the ground-impact comparison of designs was made at LASL. All Sandia Laboratories tests that involved impact capsules were made with TZM molybdenum, threaded-joint capsules, except for the overflight and reentry of the molybdenum-rhenium capsules.

Shock-Tube Overpressure Tests.¹⁶ These tests were made to determine the response of the LASL DART to simulated launch-abort overpressure situations (i.e., to an explosion of the launch rocket). The TZM molybdenum capsules containing thorium-molybdenum cermet fuel simulant disks were tested in Sandia's 2-ft-diam shock tube at overpressures of

Fig. 14A. Detail of Fig. 14. 50X

~600 and 1200 psi. The atmospheric pressure at the test site was used as the pressure on the low-pressure side of the shock. The shocks were created by exploding shaped charges in the shock-tube installation. The test conditions are outlined in Table VI.

TABLE VI
SHOCK TUBE TEST CONDITIONS

Test	Peak Static Overpressure at Atmospheric Pressure of 12 psig	Equivalent Peak Overpressure at Sea Level	Impulse, psig-s
I	650	742	2.1
II	1170	1337	2.5

Test Details. Two capsules were used in each overpressure test. The capsules were identical except for the 0.020-in.-thick fuel cans. In one unit, the can was made of molybdenum-50 wt % rhenium, and in the other, of TZM molybdenum. The units were suspended freely in the shock tube, with the impact capsule dome facing the charge.

An electric furnace heated and soaked the capsules to 500°F. The furnace was moved out of the way a few seconds before the charge was fired. The test capsules were allowed to fly downrange until they impacted the earth. Table VII summarizes the test conditions and results.

Fragment Impact Test.¹⁶ The test objective was to determine the response of an impact capsule to the impact by an aluminum fragment at a velocity of 1500 ft/s. This simulated the case in which a piece

TABLE VII
OVERPRESSURE TEST RESULTS

	Overpressure, 650 psig		Overpressure, 1170 psig	
	TZM	Mo-Re	TZM	Mo-Re
Fuel can material	Shattered	Shattered	Shattered	Shattered
Graphite ablation structure	Intact	Intact	Shattered	Intact
Impact capsule condition	One longitudinal	Several radial both ends	---	Dome badly cracked
Impact capsule cracks	None apparent	None apparent	3 unbroken; 3 broken	All broken - 0.016 wt % less than 10 μ
Fuel simulant disk damage				

of launch rocket is driven edge-on against the DART by a rocket propellant explosion.

The unit selected for the test contained the molybdenum/rhenium fuel can that had been previously subjected to the 650 psig overpressure. The impact capsule had no graphite ablation structure around it.

Test Details. The fragment (2014-T6 aluminum sheet, 8 x 12 x 1/16 in.) was mounted on an outrigger on Sandia's utility sled. The DART test unit was placed on a stationary stand in the path of the fragment. The DART was recovered in a large steel tube filled with plastic foam and aluminum honeycomb for energy absorption. The unit was heated to 550°F by an electric furnace, which was removed 5 s before impact.

Test Results. The fragment, mounted on the rocket-driven sled, impacted edge-on into the impact dome at 1486 ft/s. The TZM strength member was shattered. The fuel can went into and through the recovery tube and came to rest a few feet from the end of the tube. There was no apparent damage as a result of secondary impact.

No fuel was released. The molybdenum-rhenium fuel can had no cracks under dye-penetrant inspection, but x-ray inspection revealed considerable breakup of the fuel. Postmortem investigation of the fuel at LASL showed 0.025 wt % less than 10 μ .

Solid Propellant Fire Test.¹⁶ One LASL DART capsule was subjected to a solid-propellant fire environment. The test was to simulate a launch explosion, with the capsule coming to rest on a burning chunk of solid propellant.

Test Details. The capsule used for the test contained a TZM fuel can that had been subjected previously to the 600 psi shock-tube overpressure. The capsule, which had a longitudinal crack in the cylindrical section, was bare. The capsule

was placed on a 12 x 12 x 18 in. block of TP-H-3062 propellant. The propellant was ignited by a thin layer of black powder spread over its surface.

Test Results. The propellant burned for 285 s. A copper calorimeter melted almost completely and formed a pool of copper, freezing the capsule into the melt on cooling.

Visual inspection revealed a large deposit of aluminum and aluminum oxide from the propellant, with some copper from the melted calorimeter, covering 70% of the capsule surface. The TZM strength member had disappeared almost completely at the end of the capsule that had been placed on the propellant. The fuel can was corroded through in a small area on the exposed end. The TZM had been severely attacked, and there was a gap 0.25 in. wide down the side of the strength member. Apparently, the damage had been caused by high-temperature corrosive action, with very little indication of melting.

X-ray inspection revealed no melting of the fuel-simulant disks, and no corrosion of the fuel disks was evident.

Arc Jet Reentry Heating Simulation.¹⁷ Five LASL DART ablation structures were tested to a programmed stagnation heating rate and stagnation pressure profile which simulated earth orbital-decay reentry. Surface temperature response, maximum surface recession, and mass losses were measured. The possibility that external grooves and heat-pipe holes would accelerate erosion was examined.

Procedure and Results. The test environment was an arc-heated, 8-in.-diam free jet of simulated air (80% N₂, 20% O₂), produced in Sandia's HEAT facility. The stagnation heating-rate and pressure profiles are shown in Fig. 15. The stream bulk stagnation enthalpy profile is shown in Fig. 16. The Mach number of the simulated jet stream was 3.5.

Test Units 1, 2, and 3 consisted of a DART ATJ-graphite heat shield with rods of molybdenum used to simulate the thermal capacity of the fuel capsule. These first three tests were made to check the design of the ablation structure and to study the effects of the heat-pipe holes which were drilled into one end (see Figs. 17-20). Unit 1 was tested in a side-on orientation with the heat

Fig. 15. Heating rate and stagnation pressure profile.

Fig. 16. Bulk stagnation enthalpy profile

pipe holes at $\pm 45^\circ$ to the stagnation line. Unit 2 was tested in the same orientation except that the heat-pipe hole was positioned at 0° . Although there was a burnthrough into the heat-pipe hole when tested with this hole at 0° , the graphite structure remained intact, with the presence of the hole having little or no effect on the ablation. Test Unit 3 was tested in an end-on orientation with the heat-pipe holes in the stagnation surface. Again, the holes had no adverse effect on the ablation characteristics.

The units which were to be used in later impact tests, Units 4 and 5, contained the TZM fuel capsule with thorium fuel simulant. Unit 4 was tested side-on with the heat pipe hole at 0° . Unit 5 was preheated in a furnace to 1550°F before the test. The test orientation was side-on, with the heat-pipe holes at $\pm 45^\circ$.

Fig. 17. DART Unit 1 after test, end view.

Fig. 18. DART Unit 1 after test, side view.

Fig. 19. DART Unit 2 after test, top view.

Fig. 21. Surface temperature response, Unit 2.

TABLE VIII

SUMMARY OF TEST RESULTS, SIMULATED REENTRY

Test Unit No.	Orientation	Maximum Surface Recession (in.)	Mass Loss (g)	Comments
1	Side-on, heat-pipe holes at $\pm 45^\circ$	0.183	71.0	Molybdenum thermal capacity simulant
2	Side-on, heat-pipe holes at 0°	0.181	72.0	Molybdenum thermal capacity simulant
3	End-on, heat-pipe holes leading	0.150	50.5	Molybdenum thermal capacity simulant
4	Side-on, heat-pipe hole at 0°	0.177	70.0	TZM fuel capsule with thorium SCC fuel simulant
5	Side-on, heat-pipe holes at $\pm 45^\circ$	0.183	75.3	TZM fuel capsule with thorium SCC fuel simulant; preheated to 1550°F before test

Discussion - One of the main objectives of the reentry heating tests was to determine whether external geometrical anomalies, e.g., standoff grooves and heat-pipe holes, increased the overall ablation effect or whether the effect was only local. The tests showed conclusively that the effects were only local. If, in broadside heating, the heat-pipe hole was penetrated by the plasma stream, erosion did not extend to the bottom of the hole opposite the penetration. The exposed cavity shielded the underlying graphite from the hot stream. This effect also occurred when the end of the shield containing the heat-pipe holes was exposed to the stream. The entrance to the holes eroded for a short distance, but no erosion occurred deep in the hole; neither did the grooves around the circumference show any intensifying effect.

Fig. 20. DART Unit 3 after test, top view.

A typical surface-temperature response (for Test Unit 2) is shown in Fig. 21. Test results are summarized in Table VIII.

The basic thickness of the graphite heat shield was 0.5 in., although only 0.250 in. of ablation was expected. Maximum ablation was 0.20 in.

Also, the testing was to determine whether the design incorporating end plugs that were not integral parts of the cylinder eliminated thermal cracking that occurred if the cylinder was heated to greatly different temperatures from the ends. The graphite did not crack during these reentry heating tests, and the design consequently was considered satisfactory in that respect.

Air Drop Terminal-Velocity Determination¹⁶

The primary objective of this test was to measure terminal velocities for the earth-impact simulation tests to be performed later. Four models each of three different DART configurations were dropped from a C-47 aircraft flying at an altitude of 15000 ft msl over the Tonopah Test Range.

Free fall was in a predominantly tumbling mode. The terminal velocities were measured by radar at an altitude of 16500 ft msl. A Mitchell camera was used to obtain documentary motion pictures.

Configuration 1 represented the entire DART, including capsule, graphite ablation structure, heat exchanger, insulation, and outer can. Configurations 2 and 3 represented the graphite ablation structure and everything within, with Configuration 2 having sharp corners and Configuration 3 rounded corners, representing the ablated condition. All models were constructed to simulate the size, shape, weight, center-of-gravity, and moments of inertia of the actual object.

Figure 22 shows the three DART configurations dropped. Table IX shows the average weight and average terminal velocity for each configuration.

Simulated Ground Impact - T2M Capsule¹⁶

Two LASL DART impact tests were performed. The impact target was a granite block; the desired velocity was 290 ft/s, which was 110% of the terminal velocity, corrected to sea level, of the rounded heat shield that had been previously tested at Tonopah. The impact capsules were of the threaded T2M design. The fuel can on one unit was T2M, and the other molybdenum-rhenium alloy. The tests were conducted on Sandia Laboratories' 5000-ft sled track.

Test Details - The test units were mounted on a stationary stand that held the unit at the desired angle of 45° with the face of the target.

CONFIGURATION 1

CONFIGURATION 2

CONFIGURATION 3

Fig. 22. Air drop test configurations.

TABLE IX
SUMMARY OF AIR DROP TEST RESULTS

Configuration	Weight (lb)	Terminal Velocity (ft/s)
1. Entire DART	11.062	311.2
2. Graphite - sharp corners	4.892	260.5
3. Graphite - rounded corners	4.873	284.7

The stand was in the path of the rocket-driven utility sled which carried the target (a 2-ft cube of granite) and the catcher box. A furnace heated the capsules to test temperature, and was removed 5 s before rocket ignition. Motion pictures were taken. Table X summarizes the test results.

The test at higher temperature represented the condition after reentry through the atmosphere, whereas the test at lower temperature represented an aborted launch, in which the capsule had not reached operating temperature. The T2M was brittle, as

TABLE X
SIMULATED GROUND IMPACT TEST RESULTS

Test capsule temperature, °F	2000	500
Fuel can material	TZM	Mo-Re
Impact velocity, ft/s	275	275
Graphite ablation-structure	Shattered	Shattered
TZM strength-member	Separated at thread	Shattered
Fuel-can condition	Ruptured	Ruptured
Fuel-simulant condition	Very small amount released	Shattered

expected, and fractured at 500°F; whereas, impact at 2000°F showed that the TZM was ductile, again as expected, but the threaded joint was not satisfactory. These tests were partially responsible for the change in capsule design from threaded TZM molybdenum to welded molybdenum-rhenium.

Overflight and Reentry - Molybdenum-Rhenium Capsule - An impact capsule of the design shown in Fig. 23 was fabricated from molybdenum-46 wt % rhenium. It was similar to the TZM capsules tested previously, Fig. 2, but had a welded rather than a threaded joint. Thorium fuel simulant was placed inside the capsule before welding, and the capsule was then encased in a graphite ablation structure. This

Fig. 23. Molybdenum-rhenium impact capsule.

assembly was flight-tested in August 1971 on a rocket launched from Green River, Utah, was released after a driven return toward earth, and impacted at White Sands Missile Range, New Mexico. The graphite ablation structure broke on impact, as expected, but the impact capsule remained unharmed. X-rays showed that the fuel simulant also withstood the impact. This test of the improved capsule design was a complete success.

Simulated Ground Impact - Comparison of Designs -

The reentry-impact simulation tests at Sandia Laboratories used a single design, the threaded, TZM-molybdenum capsule. As discussed, these tests showed that the design and the material were not adequate. Alternative designs were then explored.

A series of impact tests was made at LASL to compare the survivability of three different designs. There were five capsules tested:

- The original threaded TZM molybdenum design (Fig. 2): Capsules 1 and 4.
- An alternative design (Fig. 11) which depended more on the cushioning effect of foam molybdenum than on the thickness of the casing to maintain fuel integrity on impact. These units were made of 0.020 in. high purity molybdenum sheet, with foam molybdenum surrounding the fuel simulant on all sides: Capsules 2 and 5.
- A welded molybdenum-rhenium capsule, shown in Fig. 23. Except for the material and the welded joint, it is basically identical to the original design: Capsule 3.

Each capsule contained thorium fuel simulant. All tests were conducted as follows: The capsule was supported on ceramic rods in front of a 6.5-in.-diam gas gun and was mounted broadside to the impact, for maximum damage. A portable electric furnace was lowered over the capsule, and heated it in an argon atmosphere to 900°F, well above the ductile-to-brittle transition temperature of molybdenum. A few seconds before the shot, the furnace was lifted out of the way. The aluminum missile shot from the gun weighed 40 lb, and had a steel facing which contacted the capsule. Timing pins at the gun muzzle were used to measure the missile velocity, which was 300 ft/s to simulate earth impact after reentry from space.

A heavy-walled steel tube was mounted coaxially with the gun to catch the impacted capsule and to

stop the missile at the tube face. Aluminum honeycomb or foam plastic inside the tube decelerated the capsule. The missile velocity was determined by the cross-sectional area of a steel rod which held the missile as gas pressure built up, until the rod broke in tension.

Capsules 1, 3, and 4 were of the "conventional" design for SNAP capsules, having a strong metal strength member to contain the fuel in impact situations, whereas the design used for Capsules 2 and 5 depended on a foam molybdenum structure to absorb the impact energy by crushing. With the correct crush strength and thickness, this latter technique could allow the inner container to decelerate without being exposed to forces strong enough to cause it to deform.

The impact conditions and results are summarized in Table XI.

Tests 4 and 5 were essentially repeats of 1 and 2, respectively, with impact velocity reduced 10%.

TABLE XI
DART CAPSULE IMPACT TEST SUMMARY
TEMPERATURE, 900°F

Capsule	Type	Impact Velocity (ft/s)	Results
1	TZM threaded strength member	325	Strength member and liner failed. Separation of strength member at joint. Severe distortion of parts. Liner severely failed on impact face, in welds, and in thinned regions of end caps. Some fuel simulant escaped. See Fig. 24.
2	Sheet molybdenum, foam molybdenum	325	Outer can, foam molybdenum, and liner failed. Initial foam thickness reduced from 3/8 in. to 1/16 in. See Figs. 25 and 26.
3	Molybdenum-rhenium strength member	325	Strength member deformed, did not fail. Liner failed. Strength member contained the simulant. Liner cracked longitudinally on impact face; weld opened up. See Figs. 27 and 28.
4	TZM threaded strength member	295	Similar to Capsule 1.
5	Sheet molybdenum, foam molybdenum	295	Similar to Capsule 2

Fig. 24. DART capsule No. 1, TZM threaded strength member.

Fig. 25. DART capsule No. 2, foam protection.

Fig. 26. DART capsule No. 2, showing foam deformation and failure.

Fig. 27. DART capsule No. 3, molybdenum-rhenium welded strength member.

Fig. 28. DART capsule No. 3, liner impact face.

Discussion¹⁸ - Of the three designs tested, the T2M threaded capsule exhibited the least satisfactory impact response. Because side-on impacts were chosen, to maximize the damage to all three designs, the foam molybdenum in the ends of the T2M (and welded molybdenum-rhenium) capsules contributed nothing to the survival of the capsule.

If fuel containment is used as a criterion, the molybdenum-rhenium welded capsule was the most satisfactory, because it still had a layer of containment, the strength member, intact after the impact. If, however, limitation of fuel damage is the evaluation criterion, the sheet metal-foam molybdenum capsule would be the most satisfactory, because its liner deformed the least and the fuel sustained

the least damage. (An optimum design might combine a strength member with more cushioning.)

The liners used in all capsules were unsatisfactory. The welds used for their assembly were particularly weak points (welds were not reinforced).

Thermal Performance Testing¹⁹

This section describes the thermal-performance testing at LASL, which did not involve flowing ammonia propellant. (Measurements of thrust and specific impulse, with ammonia flowing, were performed at Edwards Air Force Base Rocket Propulsion Laboratories and will be reported by them.)

Heat losses were compared in thrusters with multifoil insulation supplied by (1) Thermo Electron Co. and (2) Linde Division, Union Carbide Corp. Temperatures were obtained throughout the system. A 500-h run at the heat-exchanger design temperature of 2500°F was made. Heat-exchanger temperature vs electrical heat input was obtained for both insulation systems.

Results - Measured and calculated heat losses are shown in Table XII for Thermo Electron and Linde insulation assemblies. The heat-exchanger temperature was 2500°F at steady state. The heat losses of the two types of insulation must be examined in detail rather than taken at face value (see Discussion, below), because conditions were not comparable in several respects.

Most testing was done with the Thermo Electron insulation. With the rhenium heat exchanger at 2500°F, the temperature drop from the core of the

TABLE XII

HEAT LOSSES OF MULTIFOIL INSULATION

	<u>Heat Losses of Insulation, W</u>	
	<u>Thermo Electron</u>	<u>Linde</u>
Insulation	35	40
Joints in insulation	46	15
Centering bushings	21	15
Heat pipe holes	30	5
Inlet tubes	15	0
		(none in place)
Nozzles	29*	17*
Electrical leads	7	7
Thermocouples	<u>12</u>	<u>5</u>
Total	195*	104*

* Measured directly.

electrical heater to the rhenium was 200°F, and from the graphite to the rhenium 20° to 30°F. The 500-h run at a heat-exchanger temperature of 2500°F required 195 W throughout, indicating no degradation of insulation in this time.

The heat-exchanger temperatures were obtained as a function of electrical-heater input for the two insulation systems, and are shown in Fig. 29. The heat input was proportional to the absolute temperature to the 3.5 power, for both systems.

Outer skin temperatures were obtained at an input of 175 W and a heat-exchanger temperature of 2350°F. Typical temperatures were:

Component	Temperature, °F
Bolting flange	580
Middle of long cylindrical section	630
Flat end - outer radius	710
Flat end - near center	890

Test Specimens - The basic LASL DART design is shown in Figs. 1 and 4, Fig. 1 showing the insulation from Thermo Electron Co. and Fig. 4 the Linde insulation; the two are basically alike except that the Linde insulation is thicker.

Fig. 29. Steady state heat exchanger temperature vs input power.

Unassembled insulation halves are shown in Fig. 30 (Thermo Electron after test) and Figs. 31 and 32 (Linde before and after test).

The differences between the test specimens and an operating DART were:

- An electrical heater replaced the fuel.
- Thermocouples were led into the heater, the graphite ablation structure, and in some runs, the impact capsule and outer skin.
- The inlet tubes were not attached to valves. In the Linde insulation specimen, inlet tubes were not present.

Fig. 30. Thermo Electron insulation after test.

Fig. 31. Linde insulation before test.

Experimental Setup - Because the DART is designed to function in the vacuum of space, thermal testing had to be performed in a high vacuum, both to simulate the heat-transfer performance and to prevent oxidation of the refractory metals. The tests were conducted in a bell jar, at a vacuum of between 10^{-4} and 10^{-5} torr. Figure 33 shows the Thermo Electron insulated unit at operating temperature. The large nozzle with the bright spot looking through the throat to the heat exchanger is visible.

Fig. 32. Linde insulation after test.

Electrical leads and thermocouple wires were led into the vacuum chamber through seals in the base plate. Thermocouples were the primary temperature measuring device. An optical pyrometer was used to sight on the nozzles and on the heat exchanger, but the pyrometer was considered less precise than the thermocouples, because of uncertainties in emissivities and absorption in the glass bell jar.

Radiation heat losses were measured by heat-capacity type disk calorimeters. The disks were of copper, painted with a flat black enamel and were

Fig. 33. Thermo Electron insulated DART at operating temperature.

insulated from the housings by aluminum-oxide beads.

Discussion - The original Air Force specification for DART called for a maximum temperature of 2500°F at steady state, no propellant flow, and a minimum temperature of 2000°F at a duty cycle equivalent to 53 W of steady heat removal. Taking account of the heat loss-vs-temperature relationship of the insulation, the minimum allowable heat production in the fuel was 150 W. If the insulation system had a lower heat loss than 150 W at 2500°F, no propellant flow, there would be too much insulation, and the minimum temperature at full heat removal would go below 2000°F.

Design calculations put the required isotope heat output in the range of 150 to 180 W. However, because this heat requirement could go even higher, the basic design was sized to include as many as six fuel disks, each with a thermal output of 40 W, for a total of 240 W. These electrical heating tests were made to reduce the uncertainty in heat losses, and to obtain detailed information on performance of the two competing insulating systems.

The data in Table XII clearly show that the losses through Linde insulation were considerably less than through Thermo Electron insulation. This result had been anticipated, for the following reasons:

- While both insulations have comparable numbers of 0.5-mil-thick molybdenum foils, the Linde insulation has foil spacers of bulky zirconia cloth as compared with Thermo Electron's zirconia powder. The bulkier insulation resulted in lower losses in penetrations for supports, nozzles, and inlet tubes, because the temperature gradient was smaller and more hot area was shielded by the insulation from direct radiation to the outside.
- The Linde insulation had been designed after the Thermo Electron insulation and incorporated several refinements, some of which were made possible by the greater bulk: (a) joints were stepped to cut down on heat leakage, (b) zirconia centering bushings were optimized to cut down conduction losses, (c) nozzles were sunk deeper into the insulation, (d) nozzles were fitted with insulation blocks of foam zirconia, and (e) heat pipe holes, instead of being open to radiation losses, had slit but not punched holes in every third layer of foil.

In fact, the Linde insulation was too effective -- heat losses were less than 150 W and it should be reduced in thickness.

To optimize the insulation design and to minimize weight, the heat losses by conduction along, and radiation from, the inlet tubes could be halved by reducing the wall thickness (14 mil in the tubes tested) and by appropriate insulation, which would make the temperature gradient along the tubes from the heat exchanger to the outside fitting more nearly linear.

The temperature drops from the heater core to the rhenium heat exchanger, and from the graphite to the rhenium, which were measured at 200°F and 20° to 30°F, respectively, were about one-half the predictions. There are two known reasons for this discrepancy, involving overly conservative assumptions in the calculations, i.e., (1) that all heat was transferred between components by radiation, and (2) that heat was radiated to the heat exchanger only from the graphite ablation structure.

Assumption 1 neglected the heat conducted out through contacting surfaces, and assumption 2 neglected the fact that in the no-ammonia flow, steady-state condition, heat removal from the rhenium heat exchanger by conduction to the inlet tubes and nozzles is relatively small, and the molybdenum inner can of the multifoil insulation conducts heat from the ends and radiates inward to the rhenium heat exchanger.

From Fig. 29 it was determined that, at steady state and no propellant flow, the heat input is proportional to the absolute temperature of the heat exchanger to the 3.5th power. Simple thermal-radiation theory predicts a 4th-power dependence on temperature, but some heat is conducted rather than radiated out through the insulation, and emissivities change with temperature.

By comparing Figs. 31 and 32, it is seen that the Linde multifoil insulation undergoes permanent deformation on being heated and cooled. This is not necessarily harmful, but has to be considered if disassembly and reassembly are necessary after high-temperature operation.

Conclusions -

- Heat losses through the basic insulation, whether Thermo Electron or Linde, are not large. The major losses are through joints and penetrations in the insulation, plus conduction losses through inlet tubes, nozzles, and supports, all of which have to penetrate the insulation.
- With careful attention to design, insulation is not a bottleneck in achieving the maximum and minimum temperature requirements.
- The thicker Linde insulation has lower heat losses through joints and penetrations than the thinner and lighter Thermo Electron insulation. The thickness of the Linde insulation also reduces the heat losses conducted out by inlet tubes, nozzles, and supports.
- The Linde insulation was more effective than desired.
- Conduction (in addition to radiation) is appreciable in the transfer of heat from the electrically heated core to the heat exchanger.
- Heat losses by conduction through heating wires and thermocouples are not negligible.

Heat Pipe Performance Testing

The heat pipe performance testing consisted of:

- Simulation of thruster temperature response after heat pipe removal, and
- Determination of steady state thruster temperature with various numbers of heat pipes in place.

Simulation of thruster temperature response after heat pipe removal²⁰ - The LASL DART was designed to use two to four water heat pipes to cool the thruster from the time of incorporation of radioactive fuel until the time of launch into orbit. If the heat pipes are removed on the launch pad and no other active cooling system is available, the thruster temperature will rise. Performance tests were therefore conducted to obtain temperature-vs-time data under simulated heat-pipe removal conditions.

The test was conducted in an argon atmosphere. The heat transfer system was heated electrically to 230°F, which simulated the condition of steady-state temperature with heat pipes installed and operating to cool the radioisotope-heated thruster. Next, the input power was raised to 170 W, simulating the condition when the heat pipes are removed and the thruster temperature is permitted to rise, the thruster being cooled only by conduction to ambient air. A plot of thruster ablation shield temperature vs elapsed time after heat-pipe removal is shown in Fig. 34. The asymptotic temperature is ~1100°F.

Thruster temperature vs number of heat pipes -

With an electrical heat input of 160 W, four water heat pipes maintained the thruster ablation shield

Fig. 34. Simulation of DART heating after heat pipe removal.

temperature at 230°F in air. Two heat pipes maintained 285°F, and one heat pipe 320°F.

Conclusion. The tests indicate that heat pipes can easily control the DART temperature before launch, and show the temperature rise after launch as a function of time. Whether heat pipes are compatible with the installation of the DART depends on detailed design of the DART-satellite system.

Energy Absorption Data on Foam and Drilled Molybdenum^{21,22}

Foam molybdenum of 35% density is used in the LASL DART design to cushion the fuel during impact. To determine quantitatively the energy-absorbing capabilities of this material, compressive tests were made at room temperature and elevated temperatures on foam with densities from 25 to 65%.

In compressive behavior, the foam molybdenum is effectively isotropic. However, because some design advantages might be gained if a nonisotropic, i.e., directionally oriented, energy absorber were available, a 33% dense material was made by drilling holes through the molybdenum, on a triangular grid.

Specimens were tested under axial compressive loading, at temperatures up to 2370°F. Loads were applied slowly, and no attempt was made to determine the effect of strain rate. An inert atmosphere was provided for the tests above room temperature.

Both on a weight and volume basis, the drilled specimens were superior to foam molybdenum by up to 50%. Foam of intermediate densities (35 and 45%) were superior to foams of low or high densities, particularly at room temperature. The tests indicate that the foam or drilled molybdenum, because of its large compressive strain at moderate compressive stresses, would be an efficient cushioning material absorbing impact energy, provided that this characteristic is preserved at high strain rate. (Simulated reentry test results indicate that this is indeed the case; see Safety Testing Section).

The energy absorbed per unit initial volume varied between 10000 and 12000 in. lb/in.³ (max.) over the temperature range tested, for foam molybdenum as shown in Fig. 35. On a weight basis, the energy absorbed was 75000 to 90000 in. lb/lb for foam (Fig. 36). Figure 37 presents load-vs-deflection data. The drilled specimen shows the desirable characteristic of an almost constant load with increasing deflection until it approaches collapse and the load rises rapidly.

Fig. 35. Volume specific energy absorption vs temperature.

Fig. 36. Weight specific energy absorption vs temperature.

Fig. 37. Load vs deflection, 1000°C.

Molybdenum foam is brittle at room temperature and might be expected to have poorer energy absorption characteristics at low temperature. Figures 36 and 37 show that this is not generally the case, although high-density foam does behave poorly at low temperatures.

Ammonia Decomposition

Much information on ammonia decomposition was obtained from a report by a French research team. Experimental data on ammonia decomposition in rhenium tubing was obtained within LASL.

Investigation by French Research Team⁶ - A comprehensive experimental and analytical study of ammonia heat transfer, decomposition, pressure drop, and heat fluxes was conducted by French researchers, with results that were of great interest to the DART effort.

The heat-transfer coefficients and pressure drops of gaseous ammonia in forced convection were measured experimentally. The fluid flowed at a rate of 0.0013 to 0.0053 lb/s through a long electrically heated tungsten tube (i.d., 0.11 in.; o.d., 0.20 in.; length, 27.1 in.).

The temperature of the wall reached 4940°F and that of the fluid 4040°F. The ammonia underwent complete dissociation, and the power required for this decomposition amounted to 30% of the total power exchanged. The inlet pressure varied between 6 and 16 atm, and the maximum pressure drop in the tube was 15 atm.

Two regimes of dissociation were identified (catalytic and homogeneous), and the variations in the rate of dissociation along the tube were studied.

Correlations of experimental results are presented in the study, using both enthalpy and temperature as the driving force for the transmission of heat. The pressure drops may be calculated with the aid of the classical friction factor.

Some pertinent conclusions reported in this work are:

- Heat exchange with catalytic dissociation (the superposition method) is presented as a first approximation. It is based on the fact that ammonia decomposition takes place in molecules adsorbed by the heating wall, leading to the following conclusions: (1) the heat of dissociation is transmitted directly to the fluid at the temperature of the wall, and (2) there is a

superposition both of the heat flux from the reaction and of the heat flux corresponding to forced convection of a mixture of gases of fixed composition.

- The catalytic power of a tungsten tube could vary over a factor of four, depending on its previous history of heating, cooling, and ammonia flow. The catalytic power could not be predicted a priori.
- A graphite tube had one-tenth the catalytic power of a tungsten tube.
- Homogeneous (gas-phase) decomposition becomes significant at temperatures above 2500°F.
- The convective-heat-transfer coefficient (excluding decomposition) is fairly well represented by the relationship:

$$N_u = 0.0025 R_e \times P_r^{0.37}.$$

There is also extensive discussion of experimental methods and problems, data obtained during the tests, and discussion, tables, and curves of physical properties of ammonia and dissociated ammonia (0 to 100% dissociation).

Decomposition of Ammonia in Rhenium Tubing²³

A test was made to determine whether rhenium surfaces would catalyze the decomposition of ammonia at DART temperatures. A 12-in.-long rhenium tube with an o.d. of 0.125 in. and an i.d. of 0.097 in., was coiled into a helix with a diameter of 1.0 in. The coil was induction-heated, with 4.0 in. of tubing at operating temperature and the ends visibly cooler. Test conditions and results are summarized in Table XIII. The amount of NH_3 decomposition was measured by absorption of effluent gas.

The flow rates were somewhat lower than the worst DART condition, which is in the large nozzle flow passage. On the basis of lb/s/in.² rhenium surface/inch length, the test was at two-thirds design rate. However, this is more than compensated by the longer effective passage length in the DART (almost three times). The test therefore shows essentially complete decomposition of NH_3 above 1500°F in a rhenium heat exchanger.

Additional References

Additional background material on ammonia decomposition is found in references 1-4 and 24 through 29.

TABLE XIII

AMMONIA DECOMPOSITION IN RHENIUM TUBING

Average Rhenium Temperature in Test Section (°F)	Duration of Run (min)	NH ₃ Flow Rate (mlb/s)	NH ₃ Decomposed (%)
1990	0.5	0.17	100
1820	0.5	0.17	100
1720	0.5	0.18	99.5
1620	0.5	0.16	97.4
1510	0.5	0.15	96.7

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APPENDIX A
LASL DART SPECIFICATIONS

Nozzle	A	B	C	Feed System Characteristics Assumed	
Thrust, lb	0.010	0.050	0.100	Propellant (at valve inlet)	
Maximum number of pulses	5×10^6	5×10^6	5×10^6	Type	Gaseous ammonia
Total NH_3 flow, lb	100	100	345	Temperature, °F	70
NH_3 flowrate, lb/s	3.25×10^{-3}	1.62×10^{-4}	3.25×10^{-4}	Quality	Mil Spec. MIL-P-27406
Maximum on-time, s	0.200	0.200	0.200	Propellant valve	
Minimum on-time, s	0.020	0.020	0.020	Voltage	24 V ac
Minimum off-time, s	0.800	2	4	Opening response, msec	6
Maximum equivalent continuous thrust, lb	0.002	0.00454	0.00476	Closing response, msec	3
				Inlet pressure	50 psia (nominal)

APPENDIX B

EXCERPTS FROM "THERMAL INSULATION SYSTEM FOR THE LASL MINITHRUSTER",
UNION CARBIDE CORPORATION, LINDE DIVISION, TONAWANDA, NEW YORK 14150

GENERAL DESCRIPTION

Multilayer insulation was applied directly to the inner insulation cup halves. The cylindrical sections are spirally-wrapped and incorporate a six-step joint which mates with the end sections. End sections are constructed using pre-cut disks of foil and spacer material. The two insulated cup halves meet at a one-step circumferential joint which passes through the thrust nozzles. Penetrations exist at this joint to accommodate three thrust nozzles and nozzle insulation.

End sections of the insulated cup halves possess penetrations to accept the centering bushings, ammonia feed tubes, and heat pipes. Each thrust nozzle and cone is enclosed with a close-fitting zirconia fiber block sleeve. The two-part sleeves extend from the heat exchanger assembly to the outer jacket and form an insulating interface between the nozzles and multilayer foils.

The outermost foil layer of each insulated cup half is spot-welded to maintain the insulated configuration. Each half is protected by an outer stainless steel jacket which carries the axial holding force exerted by the insulation springs.

The following construction materials were utilized in the insulation assembly:

- Molybdenum foil (0.0005 in.) and zirconia cloth (0.005 in.), 52 layers, 2500-1600°F operating temperature.
- Nickel foil (0.0005 in.) and quartz cloth, 26 layers, 1600-300°F operating temperature.
- Zirconia fiber block, 40 pcf, 2500-300°F operating temperature.
- Stainless steel, type 304, 0.024 in. thick, 300°F operating temperature.

THERMAL PERFORMANCE

An approximate analysis was made to determine system thermal losses. This study was based, in part, on extrapolated mean apparent thermal conductivity data for an idealized Mo-ZrO₂ sample operating in vacuum at a layer density of 120 layers/in. and at temperatures up to 2250°F.

Computed losses for steady state, no-load operation, between hot and cold boundary temperatures of 25°C and 300°F are listed below.

Components	Thermal Loss, W
Multilayer insulation	55
Ammonia feed tubes (3)	25
Thrust nozzles (3)	25
Heat pipe openings (4)	20
Circumferential joint	20
Centering bushings (3)	15
Total thermal loss	160

APPENDIX C
SUMMARY OF COMPATIBILITY TESTS - 500 HOUR, HIGH VACUUM

TABLE C-I
COMPATIBILITY OF METALS AND BERYLLIA WITH CARBON AT 1400°C

<u>Couple</u>	<u>Figure</u>	<u>Visual Observation</u>	<u>Metallographic Observation</u>	<u>Chemical Analysis wt% Carbon in Metal</u>	<u>Hardness</u>
C-Ta	C-1	Stuck together, some evidence of reaction.	Two layers on TA to depth of 0.003 in.	0.024	Matrix 112 DPH Layer 1512 DPH
C-W	C-2	No reaction.	No reaction.	0.0017	Matrix 375 DPH
C-Mo	C-3	Couple stuck together. Considerable evidence of reaction. Specimens discolored.	Reaction zone in Mo to 0.027 in. Needle-like precipitate reaction zone.	0.050	Matrix 151 DPH Layer 1100 DPH
C-BeO	C-4	No reaction.	No reaction.	0.020	
C-Re	C-5	Specimens stuck together	No reaction zone. Re cracked. Re heavily twinned at surface or contains needle-like precipitate.		Matrix 381 DPH
C-(Mo-50% Re)	C-6	Specimens stuck together. Metal discolored and some evidence of a reaction.	Reaction zone to 0.056 in. deep. Lamellar structure of precipitate in reaction zone. Precipitate in grain boundary of metal throughout.	0.02-1.03	Matrix 235 DPH
C-(Pt-Rh-W)	C-7	Specimens stuck together. Some evidence of reaction. Discolored.	Very slight surface reaction only.		Matrix 235 DPH

TABLE C-II
COMPATIBILITY OF METALS WITH OXIDES AT 1400°C

<u>Couple</u>	<u>Figure</u>	<u>Visual Observation</u>	<u>Metallographic Observation</u>	<u>Chemical Analysis wt% Oxygen in Metal</u>
Mo-ZrO ₂	C-8	Ceramic discolored. No reaction.	No reaction.	0.0059
W-ZrO ₂	C-9	Ceramic discolored. No reaction.	No reaction.	0.0008
Ta-ZrO ₂	C-10	Ceramic discolored. No reaction.	No reaction.	0.0280
Mo-HfO ₂	C-11	Same as above. Appears to be slight surface reaction.	No reaction.	0.0130
W-HfO ₂	C-12	Ceramic discolored.	No reaction.	0.0014
Ta-HfO ₂	C-13	Ceramic discolored.	Very slight (<0.0001) surface reaction.	0.0300
Mo-BeO	C-14	No reaction.	No reaction.	0.0067
W-BeO	C-15	No reaction.	Slight surface roughness.	0.0006
Re-BeO	C-16	No reaction.	No reaction. Re surface irregular structure either shows Widmanstatten second phase precipitate or is very highly twinned.	<0.0001
(Mo-50Re)-BeO	C-17	No reaction.	Chain-like grain boundary precipitate throughout metal, probably sigma phase. No reaction.	0.0040

TABLE C-III
COMPATIBILITY BETWEEN METALS AT 1400°C

Couple	Figure	Visual Observation	Metallographic Observation
Mo-Re	C-18	Couple bonded together.	Mo is etched structure. Three layers in diffusion zone. Some voids in Mo rich layer.
W-Re	C-19	Couple stuck together.	W is etched. Very little reaction. Re surface slightly roughened.
W-(Mo-50 Re)	C-20	No reaction.	Mo-50 Re shows grain boundary precipitate. No reaction. Flaws due to metallography.
Ir-(Mo-50 Re)	C-21	No reaction. Ir discolored in contact area.	No reaction.

TABLE C-IV
COMPATIBILITY OF SAME COUPLES AS IN TABLE C-I AT 1500°C
(Metallography only performed where there are comments)

Couple	Figure	Visual Observation	Metallographic Observation	Carbon Analysis, wt%
C-Ta		Couple stuck together. Visible reaction.		
C-W	C-22	Couple stuck together. Slight reaction.	No visible reaction.	
C-Re		Couple stuck together.	No reaction.	0.380
C-BeO		No reaction.	No reaction.	0.044
C-(Mo-50 Re)		Couple stuck together. Slight reaction.		
C-(Pt-Rh-W)		Couple stuck together. Visible reaction.	Reaction zone to depth of 0.001 in.	0.056

ADDITIONAL COMMENTS ON 1500°C TESTS

There still was no evident reaction between tungsten and either hafnia or zirconia. Molybdenum showed no reaction with zirconia and very slight reaction with hafnia. Tantalum showed a very slight reaction with both hafnia and zirconia.

Beryllia showed no reaction with any of the metals. The metals were molybdenum, tungsten, rhenium, and molybdenum-50 rhenium alloy.

For the molybdenum-rhenium couple the diffusion zone thickened, of course. At this temperature tungsten bonded to the rhenium. Similarly, iridium bonded to the molybdenum-50 rhenium alloy.

Very little attention should be paid to the metallography of the rhenium and molybdenum-rhenium alloy, except insofar as the compatibility effects are concerned. The alloy probably shows sigma phase and demonstrates advisability of a lower rhenium content. Twinning of the rhenium is far too evident. The processing history of the rhenium and molybdenum-rhenium samples is not known adequately, particularly the retained cold work and impurity levels.

TABLE C-V
SELECTIVE WEIGHT CHANGES (IN MG) OF COUPLES EXPOSED TO 1400°C

Couple	Figure	Carbon or Oxide	Metal
C-W	C-2	- 0.7	0
C-(Mo-50 Re)	C-6	- 19.	+38.7
Mo-ZrO ₂	C-8	- 10.6	0
W-ZrO ₂	C-9	- 70.5	+ 3.0
Ta-ZrO ₂	C-10	- 41.8	+ 1.4
Mo-HfO ₂	C-11	-116.	+ 8.3
W-HfO ₂	C-12	- 11.9	+ 5.0
Ta-HfO ₂	C-13	- 60.0	+ 2.5
Mo-BeO	C-14	- 5.5	0
W-BeO	C-15	- 4.6	+ 7.0
Re-BeO	C-16	- 4.1	+ 1.5
(Mo-50 Re)-BeO	C-17	- 4.1	+ 1.0
W-(Mo-50 Re)	C-20	0 (W)	+1.2 (alloy)
(Mo-50 Re)-Ir	C-21	+4.8 (alloy)	-6.8 (Ir)

Fig. C-1. C-Ta at 1400°C. 100X

Fig. C-4. C-BeO at 1400°C. 100X

Fig. C-2. C-W at 1400°C. 100X

Fig. C-5. C-Re at 1400°C. 100X

Fig. C-3. C-Mo at 1400°C. 100X

Fig. C-6. C-Mo 50 Re at 1400°C. 100X

Fig. C-7. C-(Pt-Rh-W) at 1400°C. 100X

Fig. C-10. Ta-ZrO₂ at 1400°C. 100X

Fig. C-8. Mo-ZrO₂ at 1400°C. 100X

Fig. C-11. Mo-HfO₂ at 1400°C. 100X

Fig. C-9. W-ZrO₂ at 1400°C. 100X

Fig. C-12. W-HfO₂ at 1400°C. 100X

Fig. C-13. Ta-HfO₂ at 1400°C. 100X

Fig. C-16. Re-BeO at 1400°C. 100X

Fig. C-14. Mo-BeO at 1400°C. 100X

Fig. C-17. (Mo-50 Re)-BeO at 1400°C. 100X

Fig. C-15. W-BeO at 1400°C. 100X

Fig. C-18. Mo-Re at 1400°C. 100X

Fig. C-19. W-Re at 1400°C. 100X

Fig. C-21. Ir-(Mo-50 Re) at 1400°C. 100X

Fig. C-20. W-(Mo-50 Re) at 1400°C. 100X

Fig. C-22. C-W at 1500°C. 200X

APPENDIX D

MOLYBDENUM-RHENIUM PHASE DIAGRAM AND DUCTILE-BRITTLE TRANSITION TEMPERATURE

Fig. D-1. Molybdenum-rhenium phase diagram. Taken from R. P. Elliott, Constitution of Binary Alloys, First Supplement, McGraw-Hill, 1965.

Fig. D-2. The ductile-brittle transition temperature of cast molybdenum-rhenium. From E. M. Savitskii, M. A. Tylkina, and K. B. Fovarova, Rhenium Alloys (1965) - translated from Russian, Israel Program for Scientific Translations, Jerusalem, 1970.