Long Life Radioisotopic Power Sources Encapsulated in Platinum Metal Alloys

CASSINI MISSION TO STUDY SATURN AND ITS MOONS

By E. A. Franco-Ferreira and G. M. Goodwin
Oak Ridge National Laboratory, Tennessee, U.S.A.

and T. G. George and G. H. Rinehart
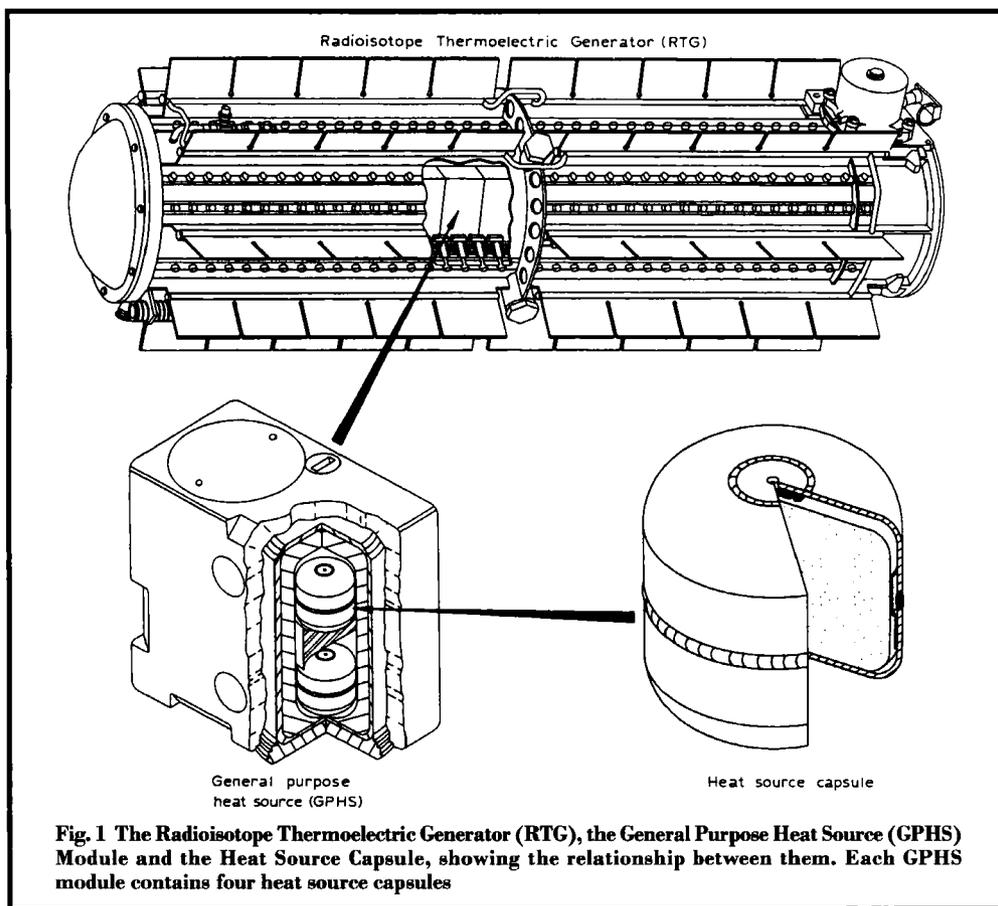Los Alamos National Laboratory, New Mexico, U.S.A.

The platinum metals alloys, DOP-26 iridium and platinum-30 per cent rhodium, have been successfully used to encapsulate plutonia fuel pellets for the Cassini Spacecraft. The iridium-encapsulated heat sources provide approximately 900 watts of electrical power for the spacecraft and its experiments, whereas the platinum alloy clad pellets will supply about 150 watts of heat to various parts of the spacecraft and its lunar probe, Huygens. The particular alloys used on this mission have been selected to fulfil the critical function of maintaining fuel containment during normal service and for projected malfunction or accident scenarios. Their ability to perform satisfactorily has been demonstrated through extensive testing of their mechanical, physical and impact properties. The Cassini heat source manufacturing yields were significantly higher than those obtained for previous missions.

The last NASA grand-scale interplanetary voyage of this century, the Cassini/Huygens Mission, is scheduled to be launched during a window which occurs between October 6th and 30th, 1997 (1–3). The mission is a joint U.S.-European close-up study of Saturn and its moons.

The four-year study will begin in July 2004 and will include sixty orbits of Saturn and about forty fly-bys of Titan, its largest moon, by the Cassini orbiter. Huygens, the European-built lunar probe, will have a parachute descent lasting for two and a half hours before landing on Titan. Among investigations will be Saturn's atmosphere, its rings and its magnetosphere and additional insights will be sought into questions raised during the twin Voyager fly-bys of the 1980s (4). The atmosphere and surface of Titan and other icy moons will also be characterised. Titan is the only known body in the solar system, other than Earth, with a thick nitrogen atmosphere, and it is thought to be the only other object in the solar system which has a liquid and solid surface with shorelines (5).

Electrical power for the eighteen science instruments and forty-four on-board processors (4) will be supplied by three Radioisotope Thermoelectric Generators (RTGs) which are powered by plutonia ($^{238}PuO_2$)-fuelled General Purpose Heat Source (GPHS) modules. This source of power is necessary because of the great distance of Saturn from the sun and because of the duration of the mission. Alternative, solar power, would have required a pair of arrays measuring at least 35 m × 9 m (2). Also, because of the distance from the sun, instruments and equipment on both the orbiter and probe will require external heaters to maintain their temperature within normal operating ranges. This heat is provided by plutonia-fuelled Light Weight Radioisotope Heater Units (LWRHUs) which are strategically located on their respective vehicles. The $144 million spent on the RTGs and LWRHUs represents approximately 4.5 per cent

Radioisotope Thermoelectric Generator (RTG)

General purpose
heat source (GPHS)

Heat source capsule

Fig. 1 The Radioisotope Thermoelectric Generator (RTG), the General Purpose Heat Source (GPHS) Module and the Heat Source Capsule, showing the relationship between them. Each GPHS module contains four heat source capsules

of the total cost of $3.2 billion for the 18 year long lifetime of the programme (4).

The plutonia fuel pellets for both the RTGs and LWRHUs are completely contained in iridium alloy and platinum alloy claddings, respectively. This encapsulation is necessary both to maintain the integrity of the fuel forms during service and to prevent accidental release of fuel particles to the environment.

The two accident scenarios of most concern entail a launch accident or an inadvertent atmospheric re-entry as a result of an orbital abort or during the gravity-assisted Earth fly-by in 1999. Extensive impact testing of fuelled capsules has verified that the cladding materials can provide the requisite fuel containment under all credible accident conditions. The occasion of the launch presents an ideal opportunity to

review and update the use of platinum metals for the containment capsules, a topic that has been reported here previously (6).

Radioisotope Thermoelectric Generators

The electricity-producing RTGs are identical in design to those used on the Galileo Mission to Jupiter, launched in 1989, and the Solar-Polar Ulysses Mission, launched in 1990. The Cassini spacecraft will carry three RTGs, the largest number ever for a single launch. An RTG and its relationship to the fuelled capsules and the GPHS modules are shown in Figure 1.

The RTG is 1.13 m long, 426.7 mm in diameter from fin to fin and weighs 55.8 kg. It contains eighteen GPHS modules powered by a total of seventy-two heat source capsules. The

electrical output is approximately 300 watts and results from thermoelectric conversion of the heat from the capsules which is delivered at an operating temperature of 1287°C.

Each heat source capsule, producing about 60 watts of heat, consists of a pressed and sintered pellet of plutonia weighing 151 g contained in a 0.685 mm thick shell of DOP-26 iridium alloy. The capsule, shown in Figure 2, is 29.97 mm long and 29.72 mm in diameter. Capsule closure is accomplished by an autogenous gas tungsten arc weld at the equator. Each capsule has a sintered iridium frit-vent at one pole to release the helium which is produced by α-decay within the fuel pellet. The frit-vent, covered by its 0.127 mm thick iridium decontamination cover, is visible in Figure 2.

As shown in Figure 1, each GPHS module contains four heat source capsules. The modules are assembled from a number of nesting graphite components which provide the required heat transfer to the thermopiles in the RTG. The graphite shells are also responsible, along with the RTG structure, for significant impact attenuation in accident conditions. To activate the vents, just prior to assembly of the GPHS module, the decontamination covers on the capsule frit-vents are removed.

Light Weight Radioisotope Heater Units

LWRHUs of the present design (7) were also used to heat components in the Galileo spacecraft (8), and were launched aboard the Mars Pathfinder Mission in December 1996. A maximum of a hundred and fifty-seven of these heat sources will be used on the Cassini spacecraft and the Huygens probe (9). Figure 3 shows various components of a LWRHU.

The fuelled capsule consists of a pressed and sintered plutonia pellet weighing 2.7 g encapsulated in a 0.875 mm thick shell of platinum-30 per cent rhodium (Pt-30%Rh) alloy. This capsule, identified as the "clad" in Figure 3, is 12.85 mm long and 8.60 mm in diameter at its two ribs. Capsule closure is by means of an autogenous gas tungsten arc weld of a flat end cap at one end of the cylindrical capsule. Three
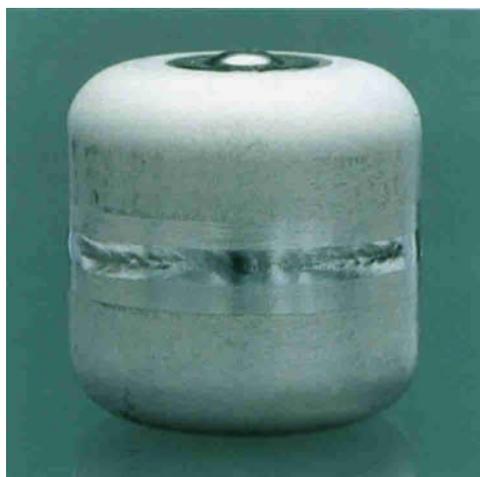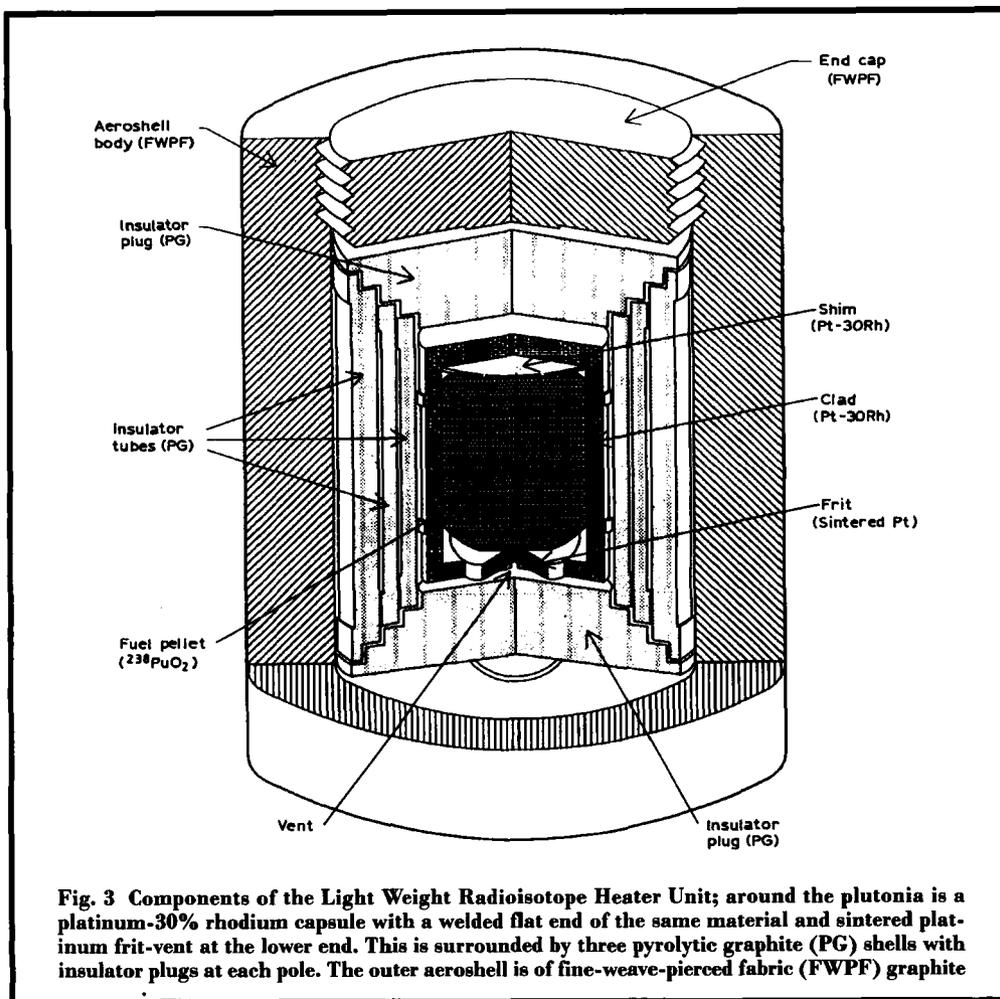

Fig. 2 A DOP-26 iridium General Purpose Heat Source capsule showing the equatorial tungsten arc weld and the iridium frit-vent at the pole to release the helium produced by the plutonia

capsules, with their final closure welds at the top, are shown in Figure 4. As in the GPHS modules, each LWRHU capsule is equipped with a frit-vent of sintered platinum in the centre of its lower end cap, which is activated prior to assembly. The remaining components of the LWRHU, shown in Figure 3, are three pyrolytic graphite shells and the outer fine-weave-pierced fabric graphite aeroshell, each with their respective end closures. These graphite components control the overall thermal balance of the heat source and enhance the performance of the fuel cladding in accident conditions.

Each LWRHU produces 1 watt of heat. The LWRHU units are mechanically attached to the spacecraft singly and in groups so as to maintain the temperature of critical instruments and mechanical devices. Some of the LWRHUs are mounted in thermostatically-controlled assemblies which are able to vary the amount of heat provided either to the spacecraft components or radiated into space.

Alloy Selection and Properties

The service environments experienced by space power isotopic heat sources may involve exposure at elevated temperatures to low pressure oxygen as well as to carbonaceous insulating

Fig. 3 Components of the Light Weight Radioisotope Heater Unit; around the plutonia is a platinum-30% rhodium capsule with a welded flat end of the same material and sintered platinum frit-vent at the lower end. This is surrounded by three pyrolytic graphite (PG) shells with insulator plugs at each pole. The outer aeroshell is of fine-weave-pierced fabric (FWPF) graphite

materials. Refractory metals and alloys based on tungsten, tantalum, molybdenum and niobium do not perform well in environments of this type. In contrast, however, platinum group metals and alloys exhibit a high degree of resistance to oxidation and to embrittlement by interstitial elements. They also have good high temperature strength and ductility, and the liquidus temperatures of their metal-carbon

Fig. 4 Three platinum-30% rhodium Light Weight Radioisotope Heater Unit capsules

compounds are high enough to pose no danger during atmospheric re-entry or accidents, such as a fuel fire.

Effects of Dopants

The development of iridium alloys doped with tungsten, thorium and aluminium (for use as containment materials and claddings) began at Oak Ridge National Laboratory more than twenty years ago (10). The addition of tungsten was found to improve alloy fabrication and increase yield strength, while additions of thorium and aluminium greatly improved ductility at very high strain rates by increasing grain boundary cohesion (11).

The optimised iridium alloy used for RTG fuel encapsulation, known as DOP-26, contains by weight 0.3 per cent tungsten, 60 ppm thorium and 50 ppm aluminium. The thorium dopant serves two important functions. At low levels, it appears that thorium segregates to the grain boundaries and improves grain boundary cohesion, while at higher dopant levels, the thorium combines with iridium in the form of Ir_5Th. These intermetallic precipitates pin the grain boundaries and inhibit grain coarsening while increasing alloy strength at high temperatures, with the optimum dopant level being 200 ppm thorium by weight (12).

Unfortunately, thorium can contribute to hot cracking in autogenous welds made in DOP-26. The iridium-rich end of the iridium-thorium binary phase diagram shows a eutectic reaction:

$$Liquid \leftrightarrow (Ir) + Ir_5Th$$
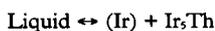
at 2080°C, and congruent melting of Ir_5Th at 2260°C, both of which occur significantly below 2447°C, the melting point of iridium (13). This behaviour would be expected to lead to hot cracking (14) and, in fact, significant problems of this nature were experienced during Galileo/Ulysses production (15, 16).

Liquation cracking was encountered in the Galileo/Ulysses capsule girth welds at the overlap region (tie-in), but was essentially eliminated for Cassini by careful control of alloy composition, melting practice (17) and welding procedures (18). The dependence of weldability on
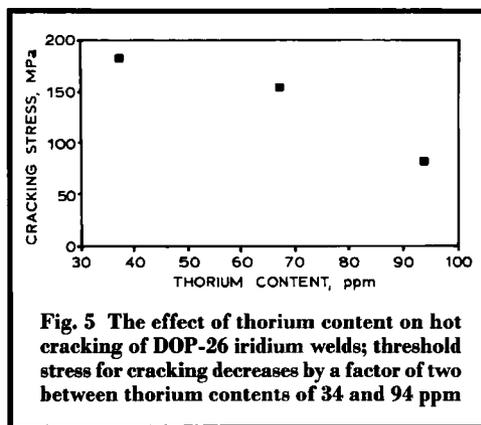

Fig. 5 The effect of thorium content on hot cracking of DOP-26 iridium welds; threshold stress for cracking decreases by a factor of two between thorium contents of 34 and 94 ppm

composition is graphically shown in Figure 5, which plots hot cracking sensitivity (cracking stress) versus the thorium level. Note that the threshold stress for cracking, as measured by the Sigmajig test (a weld cracking test) (19), decreases by a factor of two over the thorium content range of 37 to 94 ppm. Each heat of production material was subjected to multiple chemical analyses and Sigmajig tested for weldability prior to fabrication of the cladding shells. (A "heat" of material is a quantity of material melted as a single lot during production). Thorium concentrations were maintained in the 60 to 70 ppm range, which still resulted in acceptable mechanical properties. Throughout production of the Cassini GPHS heat source capsules, not a single instance of weld hot cracking was encountered (18).

Design of Radioisotope Heater Units

Radioisotope heater units used prior to the Galileo mission employed relatively heavy, multilayer tantalum alloy containment systems, loaded with 80 per cent enriched plutonia fuel in the form of shards or pellets. The primary drawbacks to these systems were the weight of the multilayer containment and the inherently poor oxidation resistance of tantalum alloys to external oxidising environments and to oxygen released by the fuel.

A design effort at Los Alamos National Laboratory on a new light-weight radioisotope heater unit focused on methods of increasing the power density of the unit and its high

temperature oxidation resistance. Because the weight and bulk of a multilayer containment system significantly reduced the power density, a decision was made early-on to utilise a vented capsule design. Although the proposed use of a frit-vent permitted the helium produced by α-decay of the $^{238}PuO_2$ to escape, and precluded the need for heavy pressure-vessel type containment, it increased the importance of the required oxidation resistance of the clad. As a result, the primary requirements for the LWRHU cladding were defined as:
• a melting or eutectic point at least 200°C above the maximum predicted temperature during atmospheric re-entry,
• sufficient strength and ductility to survive impact with the Earth with no loss of containment, and
• chemical compatibility with both carbon (present in the graphite aeroshell surrounding the capsule) and oxygen over the range of operating and re-entry temperatures.

On the basis of thermodynamic studies previously conducted at Los Alamos National Laboratory, three platinum-based alloys were selected as potential encapsulation materials: Pt-30%Rh, Pt-8%W and Pt-30%Rh-8%W (20). Subsequent testing of the three candidate alloys revealed that both Pt-8%W and Pt-30%Rh had sufficient ductility to warrant further investigation. In addition, these alloys were commercially available as sheet and tube. High strain-rate testing of Pt-30%Rh and Pt-8%W samples previously exposed to graphite at high temperatures (to simulate atmospheric re-entry) revealed that after a 1 minute exposure at 1700°C the ductility of the Pt-30%Rh was approximately 75 per cent greater than that of the Pt-8%W alloy (at a strain rate of 45 m s⁻¹). Consequently, the Pt-30%Rh alloy was selected as the encapsulation material for the LWRHU.

GPHS Encapsulation Hardware

The DOP-26 iridium alloy used to fabricate the shells for GPHS fuel encapsulation was produced at Oak Ridge National Laboratory. The production procedure, implemented in 1989, is described below (17). Iridium powder was first blended with tungsten powder to produce the Ir-0.3%W composition. The blended powder was compacted, hydrogen annealed and vacuum sintered. The sintered compacts were multiple electron beam melted into 500 g buttons. Final alloying with thorium and aluminum was performed by button arc melting under an argon partial pressure to minimise evaporative losses. The alloy buttons were drop-cast into 27 mm diameter ingots which were then joined end-to-end and vacuum arc remelted into 63 mm diameter ingots weighing about 10 kg. The ingots were hot extruded into rectangular bars at 1430°C and cold rolled to sheet. Circular blanks were cut from sheet that was surface ground to the final thickness, by electrical discharge machining. The iridium alloy blanks were then converted into shell halves as described below (21).

Iridium Alloy Blanks

The blanks were acid cleaned and each was sandwiched between two tantalum barrier discs. The sandwiches were encapsulated between two 304L stainless steel sheets with electron beam circumferential closure welds. This "blank assembly" was deep drawn at 925°C in a two-draw operation. After drawing, the stainless steel and tantalum were chemically stripped from the cups with warm acid mixtures. After stripping and acid cleaning, the cups were vacuum annealed at 1375°C for one hour to achieve full recrystallisation. One cup from each heat treatment run was destructively tested for metallurgical evaluation and chemistry verification.

The cups were sized to the required contour dimensions and a number of mechanical feature details were then added. Each cup was 100 per cent dye penetrant inspected. The outer surfaces of the cups, excluding the weld zones, were grit blasted to enhance their emissivity. A final acid clean removed any residual metallic contaminants prior to frit-vent installation and shipping to Los Alamos National Laboratory.

LWRHU Encapsulation Hardware

Capsule parts for the LWRHUs were fabricated from Pt-30%Rh (22). Sheet and tube of Pt-30%Rh alloy were first subjected to hardness
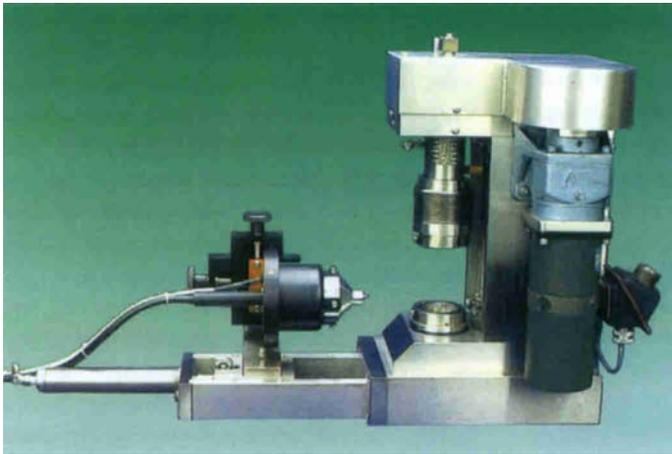
testing and confirmatory chemical analyses, and subjected to visual and ultrasonic flaw inspections. After verification of the material properties and chemical composition, capsule bodies were machined from the Pt-30%Rh tube-stock, and shims and end caps were machined from the sheet. The vent end-cap of the capsule was fabricated by cold-pressing and sintering 0.061 g of platinum powder into a porous frit, and then electron beam welding sheets of Pt-30%Rh around and over the platinum frit. A second electron beam weld was then used to attach the vent-cap assembly to the capsule body. The empty LWRHU shell was visually and dimensionally inspected and leak checked using pressurised helium.

Fuel Pellet Fabrication

Plutonia powder was sampled for chemical and isotopic analyses and introduced into the glovebox processing line at Los Alamos National Laboratory. The raw plutonia powder was heated to 775°C for several hours in an argon atmosphere saturated with ^{16}O water vapour. This step was required to reduce the neutron emission rate from the plutonia (from the ^{17}O and ^{18}O α-n reactions) by replacing the susceptible oxygen isotopes in the fuel with ^{16}O. After the oxygen exchange treatment, the fuel powder was ball milled, slugged and screened into granules, and then seasoned at either 1100 or 1600°C. After seasoning, the high- and low-fired

granules were mixed in a ratio of 40:60, and vacuum hot pressed at approximately 1500°C into GPHS or LWRHU pellets. After hot-pressing, the pellets were dimensionally inspected and then sintered at 1527°C.

Fuel Encapsulation

The GPHS capsules were assembled and welded in a helium-filled glovebox at Los Alamos National Laboratory. Assembly entailed placing the two DOP-26 iridium half-shells in the welding fixture, loading the fuel pellet into the lower shell, and bringing the upper shell into contact with the lower one at the weld joint. The lower shells contained a 0.127 mm thick iridium foil weld shield which was positioned behind the joint and prevented the weld root from contacting the fuel pellet. The welding fixture, shown in Figure 6, rotated the capsule around a vertical axis in front of a horizontally-positioned welding torch. The entire welding operation was automatic, under computer control. The autogenous, full-penetration weld was made at an up-tapering current of 115 to 116 A and a travel speed of 12.5 mm s^{-1}. The entire welding cycle required 10.8 s of arc time, and the total cycle time for capsule assembly and welding averaged 15 minutes per unit (18).

The primary non-destructive examination used for inspection of the GPHS girth welds was an immersed ultrasonic test. Automated scanning equipment produced a graphical (C-scan)
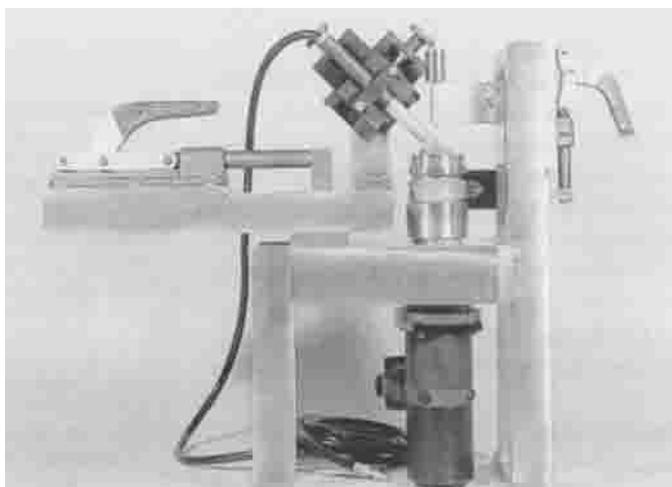
presentation of the ultrasonic signal data from each side of the weld during a two-minute test, the principal objective being to locate centre-line hot cracks at the weld tie-in. This type of defect was a significant problem during the Galileo/Ulysses production campaign. Occasional instances of weld root fusion to the weld shield and internal joint mismatch, both considered innocuous, produced ultrasonic indications which were resolved by auxiliary techniques, such as tangential radiography.

Assembly and welding of the LWRHU capsules were carried out in a helium-filled glovebox at Los Alamos National Laboratory. The Pt-30%Rh capsules were supplied with their bottom end caps welded in place by electron beam welding. The empty capsule was loaded into the welding fixture, see Figure 7, and the fuel pellet was placed into the capsule, followed by a 0.1 mm thick Pt-30%Rh weld shield disc and the 1.0 mm thick top end cap. The welding fixture rotated the capsule around a vertical axis in front of a welding torch held at an angle of 45° to the axis of rotation. The automatic welding operation was controlled by a computer system identical to that used for the GPHS capsules. The autogenous full penetration weld was made at a down-tapering current of 80 to 74 A and a travel speed of 16.9 mm s^{-1}. The entire welding cycle required 1.9 s of arc time and the total cycle time for capsule assembly and welding averaged 7 minutes per unit (9).

The completed closure welds were examined visually at a magnification of 30×, radiographed and tested for helium leaks. The vertical portion of the step had to be completely fused and the weld had to be free of linear defects exceeding 0.1 mm in length, as shown by radiography. Any pore or pores with an aggregate diameter in excess of 0.25 mm was a cause for rejection. The helium leak rate had to be less than 1×10^{-6} standard cm^3 s^{-1}. Because of product heat transfer requirements, the weld reinforcement on each flat end of the capsule had to be at least 0.076 mm at the lowest point and at a minimum of two out of three points at 90° intervals away from that location.

Production Results

A total of three hundred and nineteen GPHS capsules were built during the Cassini production campaign. The three RTGs required two hundred and sixteen capsules, the remainder being used for safety and performance testing, and as mission spares. There were thirty-six rejections for various reasons for an overall process yield of 88.7 per cent. Seven weld-related rejects were identified by non-destructive examination giving a welding process yield of 97.8 per cent. By comparison, the Galileo/Ulysses program showed a net yield of 72.7 per cent

over a total production run of six hundred and fifteen capsules.

For LWRHU capsule production, one hundred and eighty-one heat sources were fabricated for the Cassini program, with at least twenty-three units being designated as mission spares or for safety testing. Only one capsule was rejected by non-destructive examination, for a radiographic indication interpreted as a weld crack, giving a net yield of 99.5 per cent. For Galileo the yield was 89.3 per cent on a production run of one hundred and fifty capsules.

Conclusions

Because of their unique properties, two platinum metals alloys, DOP-26 iridium and Pt-30%Rh, have been used to encapsulate $^{238}PuO_2$ fuel pellets for the Cassini Mission heat sources. Extensive physical, mechanical and impact property testing have shown that these alloys are capable of providing the requisite fuel containment during all credible accident/malfunction conditions. Significant refinements in the production protocols for the DOP-26 alloy has led to improved manufacturing and enhanced weldability for those components. Overall process yields were higher than ever achieved previously, and programme costs were accordingly lower.

Multi-Agency Effort

The fabrication and supply of the RTGs and LWRHUs for use on the Cassini Mission was a multi-agency effort under the overall management of the U.S. Department of Energy's Office of Engineering and Technology Development, Space and National Security Programs. Los Alamos National Laboratory (LANL) was responsible for GPHS and LWRHU design, fuel pelletising, encapsulation welding, and final assembly of the LWRHU. Westinghouse Savannah River Company supplied the plutonia fuel powder to LANL. Lockheed Martin Energy Systems operates Oak Ridge National Laboratory and fabricated the DOP-26 iridium shells for the GPHS fuel encapsulation. EG&G-Mound Advanced Technologies was responsible for the graphite components of the GPHS and LWRHU, procurement and fabrication of the Pt-30%Rh hardware for the LWRHU capsules, final assembly of the GPHS modules, and loading, testing and shipping the three RTGs. Lockheed Martin Missiles and Space provided the housings and thermoelectrics for the RTGs, and was responsible for RTG spacecraft integration. Personnel from the Jet Propulsion Laboratory and Kennedy Space Center handled LWRHU spacecraft integration.

References

1 M. A. Dornheim, *Aviat. Week & Space Technol.*, 1996, 145, (24), 71

2 M. A. Taverna, *Aviat. Week & Space Technol.*, 1997, 146, (14), 44

3 B. A. Smith, *Aviat. Week & Space Technol.*, 1997, 146, (18), 66

4 B. A. Smith, *Aviat. Week & Space Technol.*, 1997, 146, (19), 42

5 B. A. Smith, *Aviat. Week & Space Technol.*, 1997, 146, (19), 95

6 *Platinum Metals Rev.*, 1985, 29, (1), 11; *op. cit.*, H. Inouye, 1979, 23, (3), 100; *op. cit.*, 1979, 23, (1), 16

7 R. E. Tate, "The Lightweight Radioisotope Heater Unit (LWRHU): A Technical Description of the Reference Design", LA-9078-MS, Los Alamos National Laboratory, January 1982

8 G. H. Rinehart, "Lightweight Radioisotopic Heater Unit (LWRHU) Production for the Galileo Mission", LA-11166-MS, Los Alamos National Laboratory, April 1988

9 E. A. Franco-Ferreira and G. H. Rinehart, "Welding One-Watt Heater Units for the Cassini Spacecraft", LA-UR-97-1003, Los Alamos National Laboratory, March 1997

10 C. T. Liu and H. Inouye, "Development and Characterization of an Improved Ir-0.3% W Alloy for Space Radioisotopic Heat Sources", ORNL-5290, Oak Ridge National Laboratory, 1977

11 T. G. George and M. F. Stevens, "The High-Temperature Impact Properties of DOP-26 Iridium", *J. Met.*, 1988, 40, (10), 32

12 C. T. Liu, H. Inouye and A. C. Schaffhauser, *Metall. Trans.*, 1981, 12A, (6), 993

13 "Binary Alloy Phase Diagrams", Vol. 3, ed. T. B. Massalski, ASM International, 1990

14 S. A. David and C. T. Liu, *Weld. J.*, 1982, 61, (5), 157-s

15 W. R. Kanne, Jr., *Weld. J.*, 1983, 62, (8), 17

16 W. R. Kanne, Jr., "Weldability of General Purpose Heat Source Iridium Capsules", Proc. Fifth Symp. on Space Nuclear Power Systems, eds. M. S. El-Genk and M. D. Hoover, Am. Inst. Phys., 1988, p. 279

17 E. K. Ohriner, "Improvement in Manufacture of Iridium Alloy Materials", Proc. Tenth Symp. on Space Nuclear Power Systems, eds. M. S. El-Genk and M. D. Hoover, Am. Inst. Phys., 1993, p.1093

18 E. A. Franco-Ferreira and T. G. George, *Weld. J.*, 1996, 75, (4), 69

19 G. M. Goodwin, *Weld. J.*, 1987, **66**, (2), 33-s

20 T. G. George and T. A. Cull, "The Lightweight Radioisotope Heater Unit (LWRHU): Development and Application", eds. M. S. El-Genk and M. D. Hoover, Space Nuclear Power Systems, Am. Inst. Phys., 1988, Vol. VII, p. 578

21 G. B. Ulrich, "Metallurgical Evaluation of Iridium Clad Vent Set Cups", Proc. First Int. Conf. on Heat-Resistant Materials, eds. K. Natescum and D. J. Tilack, 1991, p. 187

22 D. McNeil, Private communication, 1997, EG&G-Mound Applied Technologies, Miamisburg, Ohio

Platinum Metals Involvement in the Hydrogen Economy

International interest towards developments in various academic and technological aspects of the hydrogen economy is actively continuing and was demonstrated recently at the second conference in the Hydrogen Power Series, HYPOTHESIS-II, held from 18th to 22nd August 1997 at Agder College, Grimstad, Norway. This conference fills the intermediate years between the World Hydrogen Energy Conferences and has the same objectives. Almost 200 participants enjoyed excellent facilities and a combined lecture and poster display programme approaching 100 contributions.

Contributed papers were programmed under the subdivisions: Production, Utilisation, Distribution, Transportation and Safety, with the latter topic including some analysis of liquid hydrogen technology. Platinum metals involvement in areas of electrolytic hydrogen methodology and fuel cell technology were included in a substantial paper presented by M. M. Jaksic and N. V. Krstajic of the University of Belgrade, which dealt comprehensively with alternative catalyst compositions including both alloys and intermetallic compositions of platinum and palladium.

The advantageous inclusion of platinum in a catalytic packing composition involved in studies of the H_2-O_2 recombination reaction at low temperature was reported by G. Ioneta and I. Stefanescu of the Institute of Cryogenics and Isotope Separation, Rm. Valcea, Romania.

Incorporation of platinum into the anodes of fuel cells developed for improved resistance to carbon monoxide inhibition were reported by F. Lufrano, E. Passalacqua, G. Squadrito and A. Patti, C.N.R. Institute for Storage of Energy, S. Lucia-Messina, Italy, and the inclusion of platinum in fuel cells for vehicular transport was discussed by V. Kazarinov, F. Pekhota, V. Rusanov and V. Fateev of the Hydrogen Energy and Technology Council on Fuel Cells, Moscow. Needs for conjoint considerations of hydrogen concentration and lattice expansion strain gradients on processes of hydrogen permeation and on estimations of hydrogen diffusion coefficients in palladium and palladium alloys were summarised by F. A. Lewis, Queen's University, Belfast, R. V. Bucur, University of Uppsala, X. Q. Tong, University of Southampton, Y. Sakamoto, University of Nagasaki, and K. Kandasamy, University of Jaffna.

Selected papers will be published as a Proceedings Volume of HYPOTHESIS-II by Kluwer Press, Dordrecht. The next conference, HYPOTHESIS-III, will be held from July 5th to 8th, 1999, in Saint Petersburg State University, Russia; Fax: +7(812)428-7189, E-mail: egorov@efa.apmath.spb.su.

F. A. LEWIS

Palladium/Porous Glass Catalysts for Heck Reactions

Palladium catalysts are often used for the activation of C-H bonds, and in particular, there has been much work on palladium-catalysed Heck reactions. However, commercial development has so far been hampered by the low turnover numbers and turnover frequencies obtained. Palladium catalysts are also susceptible to poisoning, and therefore, relatively large amounts (1–5 mol%) of metal are required.

In order to overcome these problems, researchers from the CSIRO Division of Molecular Sciences, Australia, have used ~ 0.18 per cent palladium metal on porous glass tubing as a catalyst for liquid phase organic coupling reactions (J. Li, A. W.-H. Mau and C. R. Strauss, *Chem. Commun.*, 1997, (14), 1275–1276).

This catalyst, which could be used in continuous or batchwise reactions, is resistant to oxidative deterioration and can be reused several times; in most cases, the reactions can be performed in air.

The regioselectivity observed is mostly consistent with previously reported Heck reactions with terminal alkenes yielding about 80:20 mixtures of 1- and 2-arylated alkenes. This catalyst also gave good yields for the coupling of terminal acetylenes with aryl iodides and bromides to give internal alkynes without the need for solubilising or activating ligands.

It is suggested that this system could find uses in other palladium catalysed reactions, such as hydrogenations and dehydrogenations.