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MASTER

NUCLEAR ROCKET MATERIALS PROGRAM

by

J. J. LOMBARDO and C. W. FUNK

Aerojet-General Corporation
Sacramento, California

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J. J. Lombardo† and C. W. Funk‡

ABSTRACT

The Nuclear Rocket Program* has contributed a number of significant developments to materials technology. Irradiated ball-bearing retainers of polybenzimidazole bonded glass have been successfully operated in liquid hydrogen for three hours at 24,000 rpm with a 1200-pound thrust load. Solution-hardened iron and nickel-base alloys have demonstrated resistance to thermal-neutron radiation damage 10^{19} nvt without the ductility loss noted in precipitation-hardened alloy systems. Radiation damage of beryllium and graphite has decreased electrical and thermal conductivity at cryogenic temperature. Beryllium has been classified as a brittle alloy at cryogenic temperature when irradiated at 10^{18} nvt (fast); however, fracture mechanics data were generated to enable this material's application in design of reactor components. Nickel-base austenitic alloys have been cold-worked and age-hardened to exhibit 330,000 psi yield strength at -320°F without significant ductility loss. The recently developed high-strength weldable 7039 aluminum has been evaluated for radiation damage at cryogenic temperatures and found to provide elongation greater than 10% when exposed to 2×10^{17} nvt (fast). It has been demonstrated that P-03 graphite will not adhere in 10^{-10} torr vacuum. The largest Hastelloy X forgings in the United States and the welding of heavy sections of Hastelloy X constituted significant advances in the state of high-temperature alloy casting, forging, and fabricating. New management techniques to improve the reliable application of materials have been evolved from the joining of aerospace and nuclear engineering experience.

INTRODUCTION

A significant advancement in materials development has been achieved in the NERVA materials program. In the nuclear rocket engine, materials are exposed to severe environmental conditions which include both extremely elevated and cryogenic temperatures, nuclear and solar radiation, deep-space vacuum, corrosion, and intense thermal gradients.

The cryogenic temperature of the liquid-hydrogen propellant, for example, requires consideration of the ductile-brittle-transition temperature and alloy toughness. The nuclear radiation from the reactor, in turn, degrades material properties. The vacuum of space is conducive to the adhesion and cold welding of contacting surfaces. In addition, intense thermal gradients in certain reactor and engine components require determination of the thermal properties of materials.

* The Nuclear Engine for Rocket Vehicle Application (NERVA) program is administered by the Space Nuclear Propulsion Office, a joint agency of the U.S. Atomic Energy Commission and the National Aeronautics and Space Administration. Aerojet-General Corporation as prime contractor for the engine system and Westhouse Electric Company as principal subcontractor responsible for the nuclear subsystem, are developing a nuclear propulsion system.

† Space Nuclear Propulsion Office, Cleveland, Ohio

‡ Aerojet-General Corporation, Sacramento, California

For NERVA, an extensive materials program assessed the capability of each material in its total expected environment. Tables summarize the scope of the information generated in the program. Examples of significant contributions of the material technology are described in detail. The major emphasis of the NERVA Materials Program has been to extend materials technology to meet the unique requirements of the nuclear rocket (e.g., the influence of radiation, extremely high and low temperatures, and space vacuum). In addition, considerable advances have been made in fabrication processing for large and complex configurations.

CRYOGENIC AND RADIATION ENVIRONMENTS

To establish that NERVA materials will operate successfully in a cryogenic and radiation environment, it was necessary to extend available data to account for the synergistic influence of low temperature and radiation. The extension of new NERVA cryogenic and radiation data is illustrated in Figure 1. Examples of significant extensions of cryogenic data on materials are typified by the work on the NERVA pressure vessel, springs, and bolts.

Pressure Vessel Alloy

Aluminum type 7039 was selected for the NERVA pressure vessel as a result of superior ductility and toughness, coupled with high strength at cryogenic temperatures. Other candidate materials included Inconel 718 and annealed Ti 6Al-4V/ELI.

The property investigation used material obtained by forging and ring rolling 7039 aluminum to a 60-in. outside diameter, 5.5-in. wall thickness, and a 4.8-in. length heat-treated to the T-61 temper condition. Specimens were taken in the axial circumference and short transverse directions of the final ring-rolled material for evaluation. Notched and unnotched tensile, as well as precrack Charpy, specimens were utilized.

The property data obtained at test temperatures of ambient, -100°F , and -320°F are summarized in Table I. Tensile testing was conducted on short transverse orientated specimens to investigate the least worked and most questionable orientation of the material. The results showed elongation and reduction of area-ductility to be adequate at the temperatures tested. Notch toughness was also sufficient as exemplified by the notched-to-unnotched tensile ratio greater than unity at each temperature, although precracked Charpy impact indicated marginal toughness at -320°F .

Welded test plates were prepared by subcontractors to demonstrate their capability of using the tungsten-inert-gas DC straight-polarity process in joining thick sections of aluminum alloys with two weld-joint configurations. The weldments were inspected radiographically and cross-sectioned for metallographic examination. Hardness traverses were employed to evaluate the response of the weldment to post-weld heat treatment. Transverse tensile tests of the 7039 aluminum weldments were performed at room and cryogenic temperature and compared to parent metal properties.

FIGURE 1 - EXTENSION OF MATERIAL PROPERTIES

High Strength Austenitic Alloys

Nickel-base alloys were hardened to R_c 55 with yield strengths to 285 ksi for cryogenic service as springs, bolts, and bearing components. The purpose was the replacement of brittle body-centered-cubic alloys (e.g., Types 440-C and 17-PH stainless or AISI 9254 alloy steels) with face-centered-cubic alloys for service at -300 to -420°F.

Udimet 630, Waspaloy, and Inconel 718 were evaluated because of the high strength attainable by precipitation hardening. Goals included: (1) high hardness of R_c 58 for bearing application; and (2) high yield-strengths of 250 ksi or greater for springs or bolts. Cold reduction varying from 53% to 64% was procured in readily available forms. Although standard recommended heat treatments did not develop the desired hardness, variations of double-aging temperatures increased the hardness by 4 to 17% above that produced by standard treatments

(see Table II). This greater hardness resulted from reducing the aging temperatures to produce a finer dispersion of gamma-prime, the principal aging constituent. Because R_c 58 hardness was not achieved, application to rolling-element bearings was not considered promising.

Having demonstrated improved hardness, the three alloys were procured to 0.5-inch diameter, with 36% cold reduction for the testing of mechanical properties at room and cryogenic temperatures. Alloys were first aged (using the optimum heat treatments developed during experimental studies) and then tensile tested. The cryogenic ductility accompanying high strength is shown in Table III. Room-temperature tensile properties of both Udimet 630 and Waspaloy exceeded the F_{tu} 225 ksi of AISI 9264 spring-alloy at R_c 47. Ductility of the three alloys improved slightly at -320°F (Table IIIa).

The study indicated that: (1) cold-worked,

SHORT TRANSVERSE PROPERTIES

TEMP °F	ULTI- MATE TENSILE KSI	0.2% YIELD KSI	% ELONG. 2 IN.	% RED AREA	NOTCH TENSILE K _T = 6.4 KSI	RATIO NOTCH/ UNNOTCH TENSILE	PRE-CRACK CHARPY W/A IN-LB/IN
RT	62	51	15	42	82	1.31	136
-100	—	—	—	—	—	—	104
-320	81	60	14	25	87	1.07	53

TABLE I - FORGED 7039-T61 ALUMINUM

Material	Heat Treatment Cycle		Resultant Hardness R _c
	First Aging	Second Aging	
Inconel 718 0.1875 in. dia. wire, 64% CR	1350°F/8 hr/FC - to -	1200°F/total 20 hr/AC	48.0
	1450°F/8 hr/FC - to -	1300°F/total 20 hr/AC	45.0
	1250°F/8 hr/FC - to -	1150°F/total 20 hr/AC	50.6
	1200°F/8 hr/FC - to -	1100°F/total 20 hr/AC	52.0
	1125°F/8 hr/FC - to -	1000°F/total 20 hr/AC	52.1
Waspaloy 0.1875 in. dia. wire, 55% CR	1550°F/24 hr/AC	1400°F/16 hr/AC	45.9
	1600°F/24 hr/AC	1475°F/16 hr/AC	44.2
	1375°F/24 hr/AC	1225°F/16 hr/AC	50.0
	1300°F/24 hr/AC	1175°F/16 hr/AC	50.7
	1225°F/24 hr/AC	1100°F/16 hr/AC	53.6
Udimet 630 0.25 in. thick plate, 53% CR	1400°F/8 hr/AC	1200°F/10 hr/AC	52.5
	1475°F/8 hr/AC	1300°F/10 hr/AC	45.0
	1300°F/8 hr/AC	1100°F/10 hr/AC	52.0
	1225°F/8 hr/AC	1100°F/10 hr/AC	55.4
	1150°F/8 hr/AC	1025°F/10 hr/AC	55.1

TABLE II - AGING CYCLES APPLIED FOLLOWING COLD WORK
OF BEARING ELEMENT MATERIALS

aged alloys Udimet 630, Waspaloy, and Inconel 718 were not embrittled by cryogenic temperature; (2) Udimet 630 was a primary candidate for replacing AISI 9264 in coil springs; and (3) all three alloys were suitable for high-strength bolt applications.

Radiation Resistant Cryogenic Bearing Retainer

A material has been developed which is capable of operating three hours in liquid hydrogen at high speeds and loads after being subjected to a total gamma dose of 2×10^{10} ergs/gm(C). Early NERVA requirements permitted use of Armalon (a glass-reinforced Teflon bearing retainer material) which was characterized by light weight and self-lubricity. However, this material suffered degradation at gamma dose-levels in excess of 3×10^8 ergs/gm(C). By evaluating materials with increased radiation resistance, the replacement of the Teflon laminate was accomplished without sacrificing strength or increasing weight. Additional requirements were equivalent toughness, compatibility with liquid hydrogen, and some degree of self-lubricity.

TABLE IIIa - LOW TEMPERATURE SPRING ALLOY PROPERTIES

ALLOY*	TEMP °F	ULTI- MATE TENSILE KSI	YIELD KSI	ELONG. %	RED AREA	PROPOR. LIMIT KSI	TENSILE MODULUS (DYNAM) 10 ⁶ PSI	TORSION MODULUS (COMP) 10 ⁶ PSI	POISSONS RATIO (TYPICAL)
UDIMET 630 (R _c 53)	RT	289	286	6	28	211	31.5	11.8	0.32
	-320	287	284	6	29	218	33.6		
		350	342	7	23	250			
		339	328	8	26	247			
WASPALOY (R _c 51)	RT	275	260	8	24		34.4	12.8	0.34
	-320	260	248	8	24				
		324	286	12	27	200			
		318	276	12	27	196			
INCONEL 718 (R _c 47)		295	275	12	34	162			
		292	274	11	36	183			

* ALL ALLOYS IN FORM OF 0.5 IN. DIA. BAR 36% COLD REDUCTION

TABLE III - MECHNAICAL PROPERTIES OF ALLOYS FOR CRYOGENIC APPLICATIONS

Materials with promising properties were evaluated in a liquid hydrogen wear test to determine wear rates of 0.375-inch diameter specimens at speeds and loads simulating a 50-mm bearing operating at 24,000 rpm. Similar specimens were irradiated and tested to determine the effect of nuclear exposures on mechanical properties. Subsequently, full-scale bearings with promising retainer materials were tested in liquid hydrogen. Some retainers had been exposed to a gamma dose of 2×10^{10} ergs/gm(C). The evaluated materials were classified as: (1) reinforced polymers; (2) filled polymers; (3) MoS₂-filled powder-metal compacts; and (4) reinforced graphite. Although not all materials have been fully evaluated, their operating performance in bearing applications are shown in Table IV.

One material, S-glass reinforced polybenzimidazole (PBI), met all original requirements. The undamaged condition of this retainer before and after testing is illustrated in Figure 2. This polymer has exhibited higher gamma-radiation tolerance than fluorocarbon polymers by two orders of magnitude. The higher strength of this system was achieved by requiring a higher proportion of reinforcement in the laminate (70 w/o) than that obtained with reinforced fluorocarbon. The lower

	ARMALON GLASS TFE	REIN- FORCED POLYMERS	FILLED POLYMERS	REIN- FORCED GRAPHITE	Mo S ₂ - POWDER METAL
FLEXURE STRENGTH, AVE KSI	15	25	12	10	10
DENSITY, LB/IN ³	.08	0.05	0.10	0.05	0.20
GAMMA RESISTANCE, ergs/gm(c)	10 ⁸	10 ¹¹	10 ¹¹	10 ¹¹	10 ¹¹
WEAR RATE 10 ⁻⁸ CC/IN	1.0	10	12	1.5	1.2
DESIGN	INTEGRAL	INTEGRAL	SHROUDED	INTEGRAL	SHROUDED

TABLE IV - BEARING RETAINER MATERIAL PROPERTIES

50 MM PBI RETAINER AFTER 2×10^{10} ERGS/GM (C)
IRRADIATION (LH₂ COOLED)

AFTER THREE HOUR BEARING TEST
24000 RPM (1.2×10^6 DN), 1200 LB THRUST LOAD

FIGURE 2 - IRRADIATED BEARING RETAINER

weight was the result of the higher volume of porosity (15%) obtained with PBI than with reinforced fluorocarbon. Several retainers of this system were machined from laminated and fully cured cylinders.

Radiation Resistance of High Temperature Alloys

Neutron irradiation, with fluences between 10^{15} and 5×10^{18} nvt (thermal), has been conducted on three nickel-base turbine alloys to establish the radiation damage threshold at the operating temperatures of each material. Irradiation was performed at 100°F, followed by tensile testing at elevated temperatures and at a low strain-rate of 0.0013 in./in./min, to magnify the expected ductility degradation. Several tests at a high strain-rate of 0.13 in./in./min were also performed to determine the strain-rate dependency of ductility following irradiation.

Material from a full-scale Rene' 41 rotor forging (22 inches in diameter and 2.5 inches thick) was irradiated to 1×10^{16} , 5×10^{17} , and 5×10^{18} nvt ($E < 0.48$ ev) and tested at 1200 and 1400°F at a strain rate of 0.0013 in./in./min. Two specimens exposed to 5×10^{18} nvt were tested at a high strain-rate of 0.13 in./in./min, and exhibited three times the elongation of those tested at the lower rate (see Figure 3).

Inconel 718 with a normal boron content (30-50 ppm) was tested with specimens from a large rotor forging. The damage threshold at or below 1×10^{16} nvt at 1050 and 1200°F is illustrated in Figure 4. This confirms that damage is greater at higher temperatures and a lower strain-rate.

Waspaloy tensile specimens obtained from a large rotor forging (22 inches in diameter and 2.5 inches thick) were irradiated and tested at 1100, 1200, and 1400°F. Data on elongation (see Figure 5) show that the controls are insensitive to strain rate. However, the low strain-rate enhances the ductility loss in the irradiated specimens. Unlike Rene' 41, the alloy is not susceptible to embrittlement. Unlike Inconel 718, it has a higher volume of solution-hardening elements, which may explain its better radiation resistance.

Influence of Radiation on Thermal Conductivity

Thermal conductivity was measured at cryogenic temperature during radiation for the first time. Thermal conductivity was significantly affected in beryllium and graphite but not in titanium and aluminum alloys.

Five reactor materials were irradiated to approximately 1×10^{18} nvt, $E > 1.0$ Mev, while maintaining material temperatures below 150°F. Post-irradiation thermal conductivity was measured between 77 and about 250°K. Significant conductivity decreases were observed in beryllium and graphite and these must be taken into account in design. Aluminum alloy 7039-T61 and titanium alloy Ti-5Al-2.5Sn ELI did not change beyond the experimental errors. The magnitude of thermal conductivity changes in beryllium or structural alloys because of cryogenic radiation was not previously known. In beryllium and graphite, low strength and ductility required that thermal-conductivity data be accurately known to properly design for thermal stresses.

FIGURE 3 - ELEVATED TEMPERATURE RADIATION DAMAGE

FIGURE 4 - ELEVATED TEMPERATURE RADIATION DAMAGE

FIGURE 5 - ELEVATED TEMPERATURE RADIATION DAMAGE

A standard measurement method was used but some difficulty was experienced by the need for radiation resistance of test-cell subcomponents and instrumentation. Extremely high reliability was required of the test assembly because maintenance requiring a warm-up after irradiation but prior to measurement would invalidate test results. The five test cells performed successfully after neutron irradiation as high as 1.2×10^{18} nvt, $E > 1.0$ Mev.

After annealing beryllium for four days at room temperature subsequent to post-radiation measurements, all damage had annealed out (see Figure 6). The same annealing cycle resulted in an insignificant amount of recovery in the graphite (see Figure 7). Concurrent measurements of electrical resistivity indicated that good extrapolations of thermal conductivity data can be made, based on increases in electrical resistivity caused by radiation.

FIGURE 6 - RADIATION DAMAGE OF THERMAL CONDUCTIVITY

FIGURE 7 - RADIATION EFFECTS ON THERMAL CONDUCTIVITY

Fracture Toughness of Irradiated Beryllium

The technical literature contained no fracture toughness data concerning beryllium at cryogenic temperature. Because of the inherent brittleness of beryllium, these data are essential for both the unirradiated and irradiated material in order to establish design allowable stresses. Considerable care was taken in selecting a suitable test specimen which could provide a valid plane-strain critical-stress-intensity value (K_{IC}) and strain energy release rate (G_{IC}). The wedge-opening-loading specimen, shown in Figure 8, was found to be of suitable size for neutron irradiation studies and to provide valid fracture-toughness data as verified by comparison with the more conventional double-cantilever-beam specimen. K_{IC} values before and after irradiation are shown also as a function of test temperature and annealing temperature. Specimens irradiated to approximately 4×10^{17} nvt $E > 1.0$ Mev at 140°R showed decreases from 15 to 9 ksi-in.^{1/2} Partial or complete recovery of this decrease occurred after annealing at room temperature. Similar large changes in ductility of irradiated tensile specimens occurred under the same conditions.

FIGURE 8 - RADIATION DAMAGE OF FRACTURE TOUGHNESS

Effect of Space Vacuum on Adhesion

A detailed analysis of the engine component functions led to the emphasis on sliding surfaces as the mechanism most vulnerable to adhesion in space vacuum. The components have been evaluated relative to their action, lubricant surface, contact stress, sliding velocity, and gaseous environment.

Adhesion testing of sliding-contact materials was required to assess environmental effects on the operation of the NERVA engine system in space. The University of Syracuse performed the evaluation by measuring the contact resistance between materials in an ultra-high vacuum. Test specimens consisted of duplicate 0.060-inch diameter cylindrical sections that made cross contact in mid-section. The test enclosure was evacuated to 10^{-10} torr vacuum and monitored for partial pressures of high-purity gas atmospheres. Thermal degassing and argon-ion bombardment were utilized to "ultra-clean" both the contact surface and test enclosure before and during testing. Electrical contact resistance, loading, and unloading forces were monitored to distinguish between adhesion and nonadhesion characteristics. Demonstration of adhesion under ultra-high vacuum

conditions was a prerequisite before investigation of gas-species effects.

Adhesion did not occur in high vacuum for reactor graphite or for 440C stainless steel. Adhesion of titanium did occur in vacuum but could be prevented by the presence of oxygen and hydrogen.

Fabrication Development

The Phoebus 2 nozzle required premium-quality welds in heavy sections of Hastelloy X (1 to 1-3/8 inches in thickness) for the first time under actual production conditions. Three important advances were made in the state-of-the-technology: (1) ingot sizes of 24-inches in diameter weighing six tons; (2) closed-die conical forgings prepared therefrom with unprecedented sizes of 72-inches maximum diameter with a 2.5- to 3-inch wall necessary for the large power required of the Phoebus system; and (3) weldability requiring the joining of sections 1 to 1.375 inches in thickness. A completed nozzle weldment is shown in Figure 9.

Cracking associated with welding such high-nickel alloys as Hastelloy X has long been a problem in the fabrication of components from these alloys. These problems were compounded in the Phoebus Program because of the massive size and thick sections involved in the construction of the nuclear rocket nozzle. Multipass welds were required to join the three heavy Hastelloy X nozzle sections. The repeated thermal stressing imposed by many weld passes increased the alloy's inherent susceptibility to hot-cracking and micro-fissuring.

Since data were not available regarding the welding of thick sections of Hastelloy X, effort was directed toward the development of semi-automatic TIG welding procedures designed to render ineffective the inherent material characteristics which induce cracking during welding. Utilizing heavy Hastelloy X plate and ring forgings, a highly disciplined weld program was conducted,

FIGURE 9 - PHOEBUS II HASTELLOY X NOZZLE

selected welders were trained, and detailed procedures evolved which produced premium quality welds in the thick nozzle-section weld joints.

Six Hastelloy X nozzles were welded during the program. The weld quality improved with each nozzle produced. When inspected by visual, penetrant, radiographic, and ultrasonic methods, the last two weldments contained no rejectable weld defects in their 1200 linear feet of weld. Improvements in weld quality and production efficiency decreased fabrication costs by 50% when the last nozzle was produced.

The tensile and yield strength of welded specimens were superior to the corresponding parent metal. For example, the yield strength at 800 and 1200°F for the ring welds were 32.2 and 28.8 ksi respectively, which compared with the combined averages of the unwelded parent metal of 30.8 ksi at 800°F and 26.8 ksi at 1200°F.

Electrical Discharge Machining

A method to improve alignment and positioning of the coolant channels during machining was required. Electrical discharge machining (EDM) was used to expedite groove-machining the Hastelloy X Phoebus nozzle jackets. (Figure 10)

The channel grooves, which were conventionally machined in NERVA technology nozzles of 347 stainless-steel, presented a problem with Hastelloy X because of the material's lower machinability rating. Development technology

would be required in either conventional or electrical discharge methods to machine the 0.038 - 0.040-inch wide and 0.100 ± 0.010 -inch deep grooves required in the Hastelloy X material.

To machine such a narrow groove, slitting saws without side-cutting teeth were compared with a full-length carbon electrode which did not contact the material. A decision was made to develop the EDM method for the Phoebus Program. After flat samples had proven the EDM method possible, the tooling required for nozzle grooving was developed through the use of contoured specimens which simulated the nozzle. The 96 linear-inch length of the groove required the electrode to be made of several pieces of carbon. Initially, the joints caused a problem because of the alignment and vibration of the electrode during operation. This problem was corrected by cementing the electrode butt joints. Two contoured electrodes were used, one for a rough cut at higher feeds and one for a finish cut at a reduced feed. A dielectric fluid was continuously circulated around the electrode. Although it took 1.5 hours to machine a groove at first, the time was reduced to one hour after 640 grooves had been machined. Cases of human error during electrode contour trimming were the only causes for discrepancy.

Management Techniques

Materials management techniques have been evolved from the joining of the materials engineering backgrounds of the aerospace and nuclear industries. Problem prevention has been emphasized with detailed review of designs, final drawings, and specifications. The NERVA Materials Properties Data Book has been published to standardize allowable material properties of special interest to the Nuclear Rocket Program. This handbook includes physical, mechanical, nuclear, chemical, and fabrication characteristics of materials and provides the allowable stress for design use. Design properties are established on one of three criteria in the following order of preference: (1) statistically established - 3σ ; (2) specification guaranteed values; and (3) 80% of the average of experimental data.

A NERVA Materials Verification List is being prepared to validate the ability of each material to meet its predicted total environmental exposure. An estimate of the total material environment includes stress, radiation, corrosive environment, space vacuum, and vibration. Structural design specifications utilizing the material allowables defined in the NERVA Materials Properties Data Book also are being prepared to assure an orderly and consistent approach to the structural design of critical nuclear rocket engine and facility components.

FIGURE 10 - ELECTRIC DISCHARGE MACHINING