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REPORT NO. RP-DR-0001

TO

SPACE NUCLEAR PROPULSION OFFICE - CLEVELAND EXTENSION

PHOEBUS-2 SIX MONTHS MATERIALS DEVELOPMENT REPORT
FOR 15 FEBRUARY THRU 15 AUGUST 1965 (U)

SEPTEMBER 1965

CONTRACT SNPC-35

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REPORT NO. RP-DR-0001

PHOEBUS -2 SIX MONTHS MATERIALS DEVELOPMENT REPORT
FOR 15 FEBRUARY THRU 15 AUGUST 1965 (U)

ROCKET ENGINE OPERATIONS - NUCLEAR

SEPTEMBER 1965

CONTRACT SNPC-35

SPECIAL REREVIEW FINAL DETERMINATION	Reviewer	Class.	Date
	KAW	U	3-1-82
Class: <u>U</u>			

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FOREWORD

This Materials Report is submitted in compliance with Clause No. 48 - Materials Report (September 1962), SNPO-Form 417 (Mod.), Contract SNPC-35 which states in part:

"The Contractor shall furnish to the Contracting Officer, within 30 days after the end of each six-month period of the contract . . . , a separate technical report . . . , describing in summary form those tests, experiments, developments, studies, and other efforts of the Contractor relating to materials and their processing for use in fulfilling the technical requirements of this contract, which are either conducted or planned during the six month . . . period of this contract to be covered by the report. . . . "

This report covers the period 15 February through 15 August 1965.

for J. J. Reereboom, Manager
Phoebus Division
Rocket Engine Operations - Nuclear

APPROVED:

for W. D. Stinnett
Program Manager
REON

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Sect. I

Page 1

SECTION I

INTRODUCTION AND SUMMARY

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I. INTRODUCTION AND SUMMARY

A. INTRODUCTION

This Materials Review Report covers the work performed on the Phoebus-2 program during the period 15 February through 15 August 1965. It documents and summarizes the results of materials work performed under the study and development phases of the contract. Although the primary effort by Materials personnel was expended on the Phoebus-2 nozzle, participation in design reviews and materials selections was also provided. In addition, technical engineering data and support were provided to the design activity on related component hardware such as injector and adapter assemblies.

The material presented in the Detailed Discussion is divided into three sections, as listed below:

- Properties Data
- Welding Development
- Brazing Development

B. SUMMARY

Materials procurement was begun in May and June for all forgings, plate, bar, and strip materials required for the design properties, the weld development, and the brazing development programs. Lack of experience on the part of sub-contractors in controlling or analyzing for boron content to levels below 50 ppm was a cause of procurement delay subsequent to initial orders. Analysis limitations were resolved by establishing independent laboratory sources capable of determining boron content down to 1 ppm. Placement of procurements was delayed until these capabilities were established.

Hastelloy X forgings purchased on AMS-5754 were available for preliminary weld development during June, July, and August. The first ring weldment was completed

20 July on 3-in.-thick ring forgings as described in Section II-B of this report. Evaluation of the tensile properties specimens from the same material (Heat #5-2718) was also initiated.

Accomplishments of the brazing development program include experimental planning, tooling development, experimental channel forming, and jacket grooving.

Process specifications are being prepared and the initial braze joints will be made after receipt of acceptable materials.

SECTION II
DETAILED DISCUSSION

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II. DETAILED DISCUSSION

A. PROPERTIES PROGRAM

1. Materials List

The materials selected for the Phoebus-2 nozzle and materials development program are listed in Table 1. Hastelloy X was selected as the reference structural material and Hastelloy N and Inconel 625 were included in the test program as backup jacket and cooling channel materials. The weld and braze filler materials are also listed. The properties data are discussed in detail in subsequent paragraphs.

2. Material Specifications

The program objectives defined that the materials specifications would incorporate the most recent technical advances which have an application to the materials and procedures indicated for the Phoebus-2 nozzle. Modification of current industrial or government specifications incurred delays and problems. However, the anticipated results of using the select quality, vacuum melted materials, and minimizing undesirable elements such as phosphorus and boron were considered as ample justification for such changes.

Five materials procurement specifications were generated for nickel-chromium-molybdenum alloys applicable to the materials evaluation program. The initial issue of these specifications was based on ORNL documents. The Aerojet documents cover the alloys designated as Hastelloy X, Hastelloy N, INOR-8, Inconel 625, and ALLVAC 625. The chemistry and tensile properties were presented as jointly approved by SNPO and Aerojet. Aerojet nondestructive testing requirements were incorporated. The latest issues of referenced ASTM test methods were introduced.

A summary of the work done on these five specifications is as follows:

Forgings - AGC-90056B was based on ORNL Specification MET-RM-2. The initial issue date was 31 March 1965, with the "A" and "B" revisions issued on 9 April 1965 and 14 May 1965, respectively.

Plate, Sheet, and Strip - AGC-90057A - Amendment 1 was based on ORNL Specification MET-RM-B334. The initial issue date was 31 March 1965. The "A" revision was issued on 9 April 1965 with Amendment 1 issued on 14 May 1965.

NOZZLE

JACKET	HASTELLOY X HASTELLOY N	REFERENCE A ALTERNATE	AVAILABLE LARGE SIZE INGOT AND FORGING EXPERIENCE
CHANNEL	HASTELLOY X INCONEL 625 HASTELLOY N	REFERENCE ALTERNATE ALTERNATE	
WELD FILLER	HASTELLOY X *HASTELLOY N *INCONEL 625	UNDER STUDY OF COMPOSITIONS AND MERITS	
BRAZE ALLOY	82 Au-18 Ni 70 Au-8Pd-22 Ni 81.5 Au-16.5 Cu- 2 Ni		

INJECTOR

PLATE	347 SS		
RING & BAFFLES	COPPER - (OFHC)		
BRAZE ALLOYS	65 Cu 35 Au-3 Ni - (1925°F)		
1ST REBRAZE ALLOY	50 W/O (82 Au-18 Ni); 50 W/O (65 Cu 35 Au-3 Ni) (1850°F)		
ADAPTER	347 SS		
ADAPTER LEG	7039		

* Originally proposed in weld development effort

Table 1
 Materials List

Welding wire - AGC-90058A - Amendment 1 was based on ORNL Specification MET-RM-B304. The initial issue date was 31 March 1965. The "A" revision was issued on 9 April 1965 with Amendment 1 issued on 14 May 1965.

Weldability test - AGC-90059B was based on ORNL Specification MET-WR-4. The initial issue date was 31 March 1965 with the "A" and "B" revisions issued on 9 April 1965 and 14 May 1965, respectively.

Bar - AGC-90060 was based on ORNL Specification MET-RM-B336. The initial issue date was 9 April 1965. This specification was revised to reflect the changes already incorporated into the other specifications. This specification was released 2 June 1965.

3. Preliminary Processing Plan

The preliminary Materials Processing Plan, including integration of test and inspection operations previously reported in RP-P-0001, Phoebus-2 Nozzle Program Plan, was revised and is presented in Figure 1. Operational changes reflected in the revised plan include:

Annealing after forging per Specification AGC-90056B.

Cleaning after rough machining and upon completion of the welding.

Elimination of the stress relieving operation subsequent to rough machining and prior to welding of the jacket forgings. Haynes-Stellite indicates that stress relief at 1900°F will not help the mechanical or metallurgical properties but will adversely affect the ductility. Haynes-Stellite indicated that stresses resulting from rough machining are only a surface condition and therefore do not adversely affect weldability of the forging.

In the Legend of Figure 1, the symbol for hydrotest was changed from H to Y for clarity, and F was added for Penetrant Inspection.

Chemical analysis was changed to wipe test after cleaning and packaging of shim material.

Figure 1
 Preliminary Materials Processing Plan

4. Design Properties Data

Jacket Forgings

The materials selected for the nozzle jacket are all solid solution strengthened nickel base alloys to assure good yield strength and ductility over the entire temperature range from -423° to 1200°F . While some other alloys may offer adequate strength and ductility in this temperature range, the service temperature requirement for nickel-base alloy channels and the attendant joining problems eliminated other alloys for candidate jacket materials.

The material design requirements of the nozzle jacket are summarized as follows: Cryogenic ductility over 10% elongation together with good values of tensile, yield, shear, and compressive strength and the modulus of elasticity for the temperature range of -423° through 1200°F .

Physical properties relative to thermal stress, coefficient of thermal expansion, thermal conductivity, elastic modulus (dynamic), Poissons ratio, and plastic strain effects.

The data obtained from industrial and government sources were useful to the ambient and elevated temperature design requirements, but are very limited in sub-zero properties. The actual properties to be obtained from the heavy section forging could differ from the tabulated values. The available data were taken from sheet, bar, and plate, but no data were available from heavy section forgings. The majority of the reported data are for air melted grades containing the commercial range of alloy constituents including the interstitial elements, hydrogen, oxygen, nitrogen, and normal levels of phosphorus, and sulphur. It is apparent that most of these elements may be significantly lower in the vacuum melted jacket forgings. The effect of the metallurgical thermal history including the brazing cycle on the mechanical properties may contribute a significant change which is not defined by any of the available references. The published values properties for the three jacket alloys for the 0.2% yield strength are shown in Figure 2. The figure includes weld metal properties and a design allowable curve (solid line) based on 80% of values for the 0.5-in. bar stock data. Figure 3 presents the ultimate strength data corresponding to the above yield data. The values for the elongation of the same materials are shown in Figure 4. The solid line represents the

DATE: Haynes - Oct 64 (Publ. date)
 AGC - 1965

Figure 2
 0.2% Yield Strength - Design Allowables

DATE: Haynes - Oct 1964
 AGC - 1965

Figure 3
 Ultimate Tensile Strength - Design Allowables

DATE: Haynes - Oct 1964
 AGC - 1965

Figure 4
 Elongation vs Temperature - Design Allowables

design allowable values based on 80% of values for Hastelloy X bar. The dashed lines in Figures 2, 3, and 4 present the first Aerojet samples (tested at LASL) of vacuum-melted Hastelloy X. The improved elongation values of the CEVM material compared with air melted heats illustrate the advantages of the vacuum approach.

Efforts to evaluate the full range of variation and tensile properties are represented by the vertical bands shown in Figures 2, 3, and 4. The data provided by Union Carbide, Stellite Division, represents a large number of tests (over 100) at room temperature, of which 1% to 99% of the data falls within the bands shown. Values for the modulus of elasticity, the thermal expansion and the thermal conductivity are shown in Figures 5, 6, and 7 respectively.

Specimen Preparation

The first Hastelloy X forging was heat treated in hydrogen simulating three braze furnace cycles with peak temperatures of 1840, 1800, and 1775°F, respectively. In each case, a slow furnace cool of $60 \pm 5^\circ\text{F}/\text{hour}$ was maintained. similar to full scale nozzle brazing. After heat treatment an average increased hardness of Rockwell B-91 compared to the original value of B-82 was observed. After heat treatment, the Hastelloy X ring forging was sectioned and prepared for machining of tensile specimens for design data evaluation.

By 31 July 1965, 110 tensile specimens representing Hastelloy X ring forging, Heat No. X5-2718EE, were machined, inspected, and accepted by AGC. The specimen types included the following:

- Forty Standard R-1 per Drawing 1114843.
- Twenty Standard R-3 per Drawing 1114844.
- Thirty Standard R-1-N (Notched) per Drawing 1114845.
- Sixteen Standard R-3-N (Notched) per Drawing 1114846.
- Four Standard R-4 per Drawing 1114842.

All specimens were serialized as to heat number, location, and orientation of each specimen.

Channel Strip Materials

The requirements for the channel wall material are very stringent as a result of the surface temperature of 1800°F (maximum) and the high heat flux which imposes

severe temperature gradients in the channel wall. Hastelloy X is the reference alloy for this application as a result of its high temperature properties and extensive use at high temperature. The alternative materials are of interest because of higher yield strength values above 1600°F.

The properties required by the design are the same as those listed for the jacket forgings, with the addition of a higher temperature (up to 1800°F). Since the channels are to be furnace-brazed, welding need only be evaluated for end-caps; however, the thermal expansion must be matched with that of the jacket material. The data shown (Ref. Figures 2, 3, and 4) for the three nickel-base alloys for the jacket will be used until more representative data for the thin channel strip are available.

Boron Analysis

Analysis for boron in Hastelloy X and N by several laboratories was reported by Haynes-Stellite Division, Union Carbide Corporation, indicating conflicting results. Samples of the first heat of Hastelloy X forging (No. X5-2718EE) were submitted for boron analysis to Anamet Laboratories, Berkeley, California. The original results indicated 23 ppm boron, but on re-checking a value of 4 ppm was reported. The problem was apparently due to abrasive particle contamination (possibly from emery cloth used to clean the sample). This concern for the prevention of boron contamination will be applied to grinding of weld joints. The re-run was done in triplicate with values of 4.2, 4.0, and 3.9 ppm measured.

To establish a laboratory which Haynes-Stellite and Aerojet can accept as satisfactory to all parties, additional samples were sent to Hales Testing Laboratory for boron analysis. Haynes-Stellite also sent samples from heats that have been analyzed previously to the same laboratory. The same alloy heats are also being checked at Oak Ridge. It is intended that this approach establish a degree of reproducibility and allow for greater confidence in the analysis of Hastelloys with low boron content. The procedure used is the wet chemical process employing a fluoremetric or colorimetric determination which is reported to have a $\pm 10\%$ accuracy at 5 ppm and about $\pm 20\%$ accuracy at 1 ppm boron levels. The lack of boron homogeneity within the alloy is an additional source of spread in the data which has not been defined.

Figure 5
 Modulus of Elasticity

Figure 6
Thermal Expansion

Figure 7
Thermal Conductivity

Additional samples of Hastelloy X and N previously analyzed by Anamet and Ledaux and Company Testing Laboratories were sent to Hales Testing Laboratory for boron analysis to establish a laboratory which Haynes-Stellite and Aerojet can mutually accept as being satisfactory. Some of the same alloy heats were checked at Oak Ridge National Laboratories (ORNL). Ledaux and Company or Hales Testing Laboratory were recommended as reliable sources for boron analysis by Aerojet. Union Carbide Corporation, Stellite Division, ALLVAC, and Aerojet were directed by SNPO to use Hales Testing Laboratory for future boron analysis.

BORON ANALYSIS PPM

<u>Heat No.</u>	<u>Material</u>	<u>Form</u>	<u>Anamet</u>		<u>Ledaux</u>	<u>Hales Testing</u>
			<u>Initial</u>	<u>Recheck</u>		
X4-4417	Hastelloy X	.021" thick sheet	35	23	4	5.1
X3-4097	Hastelloy X	.010" thick sheet	25	22	6	5.5
X4-4606	Hastelloy X	.016" thick sheet	11	--	3	6.0
N4-5110	Hastelloy N	.025" thick sheet	24	--	3	5.4
281-4-0143	Hastelloy N	3.50" diameter bar	31	--	7	5.0
284-5-0101	Hastelloy N	Billet	51	--	5	7.1
260-5-2757	Hastelloy X	Forging	61	--	5	6.2
260-5-2758	Hastelloy X	Forging	43	--	4	5.8
X5-2761	Hastelloy X	Forging	26	--	5	5.3
X5-2718EE	Hastelloy X	Forging Ring CEVM	23	4	--	7.0

Results of a chemical analysis performed at Hales Testing Laboratory on Hastelloy X ring forging Heat No. X5-2718EE indicated this material contained the following elements which were not previously reported: 0.90 Mn, 0.45 W, 0.13 Al, 0.01 Ti, 0.10 Cu, 0.02 V, and 0.002 Mg. These data confirm that this heat meets the chemical composition of AGC-90056B.

Samples of Hastelloy X and N materials were submitted to K. E. Estes, SNPO-C, for analysis at ORNL. The following material forms and heats were included:

Chemical Analysis Sample Submitted to SNPO

<u>Material</u>	<u>Form</u>	<u>Heat Number</u>
Hastelloy X	Forging	X5-2718EE
Hastelloy X	Plate	X5-2752EE
Hastelloy N	Plate	284-5-0100
Hastelloy X	.062" dia. wire	X4-2941
Hastelloy X	.045" dia. wire	X4-4485
Hastelloy N	.045" dia. wire	N1-5059

Additional Properties

The service conditions for the Phoebus-2 nozzle indicated that the performance of materials in the region of plastic strain is a serious consideration. The stresses computed from the large temperature gradients superimposed upon the mechanical stresses exceed the normal stress allowances in the jacket, and thus present a special case. The use of controlled strain produced by bending a specimen while under a tensile load was proposed as a means of determining the effects of the service conditions on the candidate alloys.

While the similar nickel-alloy compositions demonstrate comparable short time tensile properties, the performance and relative plastic-strain endurance properties are unknown (such as those resulting from testing by repeated straining in the elastic and plastic regions). Such severe tests are believed to demonstrate more decisively the performance variation of the reference alloys in the temperature range of interest; namely, room temperature to 1200°F. The proposed approach for such testing is shown in Figure 8. However, consideration needs to be given to testing nearer coolant tube service temperature such as 1600-1800°F.

Design and fabrication of the elastic-plastic strain cycling test fixture was initiated in July. Tests were planned for September and October.

5. Radiation Effects on the Properties of Hastelloy X, Hastelloy N, and Inconel 625

Hastelloy X and Hastelloy N have been used in reactor service and benefit from sufficient radiation exposure to demonstrate their performance under varying conditions. Nuclear facilities which have reported irradiation testing or reactor applications include: Aerojet-General Nucleonics, San Ramon, California; BMI, Columbus, Ohio; General Atomics, San Diego; General Electric, Cincinnati, Ohio; ORNL, Oak Ridge, Tennessee; and Phillips Petroleum, Idaho Falls, Idaho.

Figure 8
 Technique for Plastic Strain Testing

No work has been reported on Inconel 625 irradiation because of its recent development. Inconel 625 alloy is a solid solution strengthened nickel base alloy, which helps predict its metallurgical behavior in service.

The effects of radiation can best be treated in two regions, low temperature and elevated temperature, although no sharp line of distinction is intended.

Low Temperature Exposure

The performance of Hastelloy X and similar ductile alloys when exposed to reactor neutron doses in excess of 1×10^{19} nvt ($E \geq 0.1$ MeV) of ambient reactor temps was consistently satisfactory. The yield strength increased by a factor of two after 5×10^{19} nvt with very little change in ductility as shown on Table 2. The threshold dose for the indicated hardening effect of fast neutrons is approximately 2×10^{17} nvt, but substantial changes are not generally observed below 10^{19} nvt, which is approximately the fast neutron dose anticipated at the throat of the Phoebus-2 nozzle after firing. Since the nozzle jacket wall will be at low temperature (below 600°F) the yield and tensile strength will improve progressively as shown in Figure 9. The hardening of Hastelloy N or Inconel 625 would presumably be very similar; however, the changes in percent elongation tend to differ between alloys, depending upon the micro-structure of each alloy and the heat treatment.

In general, it is concluded that the low temperature effects are not deleterious to the material performance of Hastelloy X.

Elevated Temperature Radiation Effects

Neutron exposures at elevated temperatures result in progressively less hardening and strength changes as the temperatures are increased. At temperatures about 1000°F the exposure to 10^{19} nvt would cause the material to suffer only a 5 to 15% loss in strength and ductility.

At higher temperatures (1200°F and above) a number of nickel-base alloys and stainless steels have demonstrated a loss of ductility, primarily noted during stress rupture testing after irradiation. Most nickel base alloys which have been exposed to thermal neutrons at about 10^{16} nvt, or more, will show a decrease in the rupture life and elongation when tested at 1200°F and above as shown for

PROPERTIES	UNITS	PRE-IRRADIATION VALUE	(*) AFTER IRRADIATION, AT INTEGRATED NEUTRON FLUX, IN n/cm^2					
			5×10^{19}	CHANGE, %	1.8×10^{20}	CHANGE, %	2.5×10^{20}	CHANGE, %
1. ULTIMATE TENSILE STRENGTH	1000 psi	112.5	129.3	+15.0	130.7	+16.2	131.0	+17.3
2. YIELD STRENGTH 0.2% OFFSET	1000 psi	49.4	100.2	+103	104.0	+110	106.3	+115
3. ELONGATION	%	52.0	50.0	-3.8	43.0	-17.3	42.0	-19.2
4. REDUCTION IN AREA	%	52.0	62.0	+19.3	64.0	+23.1	59.0	+13.5

(*) Irradiated at $120^{\circ}F$, tested at R. T.

Refs: Bartz, M. H., "Performance of Metals During Six Year Service in the MTR."
Peaceful uses of Atomic Energy, Geneva, (1958) 5 P. 1878

Table 2
Effects of Irradiation on the Properties of Hastelloy X (Annealed)

Figure 9
Hastelloy X Irradiation

Hastelloy X in Figure 10. The elongation measured by short time tensile testing was less affected than stress rupture elongation values after low neutron doses, but the former was reduced progressively above 10^{17} nvt (thermal). Low strain rates show greater losses than high strain rates.

Short time tensile test data on Hastelloy N were obtained at ORNL (see Ref. 1) for temperatures ranging from room temperature to 1650°F after irradiation at 1300°F to doses of 8×10^{20} . The extent of the changes in properties is shown in Figure 11. The uniform elongation values were found to fall more seriously than the total or true fracture strain, which had a value of 4.9% at 800°C as compared to 1.8% for the uniform strain values. It was observed (Reference 2) that the loss of ductility was affected by strain rate at the higher temperatures. At a strain rate of 0.2 in./in. minimum the uniform elongation at 700°C (1292°F) was 13.5% as compared with a value of 5.6% at the low strain rate 0.002 in./in. minimum. The corresponding unirradiated values were 32% and 20%, respectively. Although the above ORNL data represents a case where the neutron exposure is about 40 times as great as Phoebus-2, it does contribute to evaluating the problem.

The generally accepted cause of the severe loss of ductility and stress rupture life for nickel-base alloys (as well as stainless steels) when stressed at 1200°F or above is the formation of helium by the reaction $B^{10}(n, \gamma) Li^7$, and to a lesser extent by other gas forming reactions. The boron content of the Hastelloy N tested at ORNL was listed as 40 ppm, and that for the Hastelloy X was 5 ppm for data shown in Figure 10.

Martin and Weir (Reference 2) indicate further evidence that boron is involved by comparing cadmium shielded and unshielded exposures which show that thermal neutron reactions are the principal cause of the ductility loss.

A systematic evaluation (Reference 3) using varying amounts of boron in the alloy was performed with Inconel 600. However, a similar evaluation has not yet been made with the Hastelloys. The allowable concentration of boron in the alloy cannot be established without further work because of the many factors which affect the results. Alloy properties are affected by radiation damage of boron by the following conditions:

Figure 10
 Hastelloy X Irradiation at 1200° F

Figure 11
 Thermal Neutron Effects on Hastelloy N

Thermal flux
Temperature of exposure
Time of radiation exposure
Temperature during applied stress
Boron content
Metallurgical history of alloy
Grain size
Strain rate

While all similar boron-containing alloys have shown neutron induced changes when tested at elevated temperatures, the extent of damage cannot be predicted from the boron analysis above. Stress rupture tests (Reference 4) on A-286 heats having 8.5 ppm and 100 ppm boron contents demonstrated that irradiation of 5×10^{19} nvt (fast) was more damaging to the low boron than to the high boron analysis. The stress rupture life and elongation at 1200°F of the high boron heat was substantially better than those properties of the low boron analysis under the same test conditions. While the effects on A-286 are not applicable to other alloys they serve to illustrate the complex nature of the problem which is primarily dependent upon the micro-structure, grain size, and grain boundary conditions.

No quantitative relationship for the influence of boron on Hastelloy X is apparent since the only available data relating to high temperature testing was on heats containing less than 10 ppm boron. The 40 ppm boron contained in Hastelloy N was apparently too high for Phoebus applications, but data are not available on low boron containing heats as yet.

Note that all tangible evidence of serious ductility losses as a result of neutron doses less than 10^{20} nvt were at temperatures below 1000°F where grain boundary fracture was evident. Lower temperature exposures and stressing irradiated material at lower temperatures will allow the use of established data with normal safety factors.

At least two approaches are under consideration as a means of reducing the degree of degradation of ductility and strength. They are the reduction of the boron content in the original melt, and the controlling of boron in stable precipitates

not primarily at the grain boundaries. A fine grain size is also desirable, and may be helpful in the channel strip, but is not considered feasible in heavy forgings for the jacket.

Twelve Hastelloy X tensile specimens (R-3) were submitted for irradiation during the first Phoebus (321) reactor test at NTS. A suitable location was available on the roof of the privy, providing high thermal neutron flux (1.26×10^{14}) and access to forced air cooling. A dose of about 2×10^{17} nvt (thermal) was desirable to evaluate the effect of boron on the high temperature ductility of Hastelloy X under Phoebus conditions. Temperature control and monitoring was a problem, but heat transfer calculations indicated that forced air cooling was sufficient to maintain the desired range (below 1000°F). Temperature indicating paints were applied to the specimens to show the peak temperatures during exposure. The reactor test series was terminated after 11 minutes at full power, so that about 50% of the desired dose was obtained. (Originally two or more runs were scheduled.) Evaluation of the 12 irradiated Hastelloy X tensile specimens were postponed to determine if any further side-by-side exposures on the same material are feasible. Dosimetry data indicated that the specimens received 8.3×10^{16} nvt (thermal) and about 3×10^{17} nvt (total) dosage.

B. WELDING DEVELOPMENT

Fabrication of the Phoebus-2 jacket in three segments will require definition of the welding process to provide optimum and reproducible joint quality.

The objective of the welding program is to establish the weldability of Hastelloy X and two alternate heat resistant nickel base alloys, in thick-section sizes (in excess of 1-1/2-in.). Current literature and industrial experience with regard to welding of these candidate jacket materials is limited to smaller section sizes.

The welding is to be performed on three vacuum-melted heats of Hastelloy X and Hastelloy N in forgings and plate form and in three vacuum-melted heats of Inconel 625 in plate form. In addition three vacuum-melted heats of filler wire are to be employed in the welding of each of three candidate materials in forgings and plate form.

1. Forging Joints

The initial weld joint is located in the cylindrical section approximately 2-in. forward of the pressure vessel flange on a 59-in. diameter. The section thickness in this area is approximately 1.375-in. as seen in Figure 12. The proposed weld joint preparation is also shown. A double U joint preparation having a root radius of 0.180-in., a 45° included angle, and a 0.125-in. abutting land was selected to accomplish a balance of the welding shrinkage stresses. Approximately 40 automatic weld passes will be required to complete this weld.

2. Development Plan

The welding evaluation program plan is illustrated in Table 3. The first task was to establish the initial welding parameters which were formulated into a preliminary welding procedure. The weld quality is to be monitored by nondestructive tests and metallography. It is planned to evaluate the TIG process on Hastelloy X plate material. Plate material was selected primarily because of lower costs involved. These plate weldments are to be submitted for nondestructive engineering evaluation and correlated with metallographic examination.

The weldability of forgings will be established by welding ring rolled forgings simulating the jacket throat diameter. The degree of restraint encountered in the automatic welding of these ring forgings will approximate that encountered in jacket fabrication. The extent of the distortion throughout the welding operation will be closely monitored. In addition to in-process weld inspection, the rings will be submitted for NDT engineering evaluation. Limited weld-process qualification tests will be performed to verify the application of sound welding techniques. The applicable filler wire will be selected and plates will be welded by the automatic process. The plates will be submitted to NDT engineering evaluations. Weld qualification tests will be performed as in the two previous studies. Qualified weldments will be submitted for the design materials test program.

The Hastelloy N welding evaluation will likewise be performed on plate and ring rolled forgings. The flow diagram is essentially the same as that of the Hastelloy X. It is assumed that weld process can be completely defined in the Hastelloy X evaluation.

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Figure 12
Jacket Weldment

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Table 3
 Welding Evaluation Program Plan

The Inconel 625 welding evaluation will be confined to plate material. This is because of the small size and weight of forgings produced in this alloy.

Completion of the projected program will have established suitable welding procedures to be applied to the jacket fabrication.

The type and quantity of weldments which will be fabricated during the course of the development program are summarized in Table 4. On the left, Table 4, are types and heats of each filler wire to be employed. Type 347SS listed was welded with Hastelloy X filler wire to establish the initial welding parameters. The vertical column of figures illustrates the number of weldments to be fabricated during the course of the test program in the three heats of each material in forging and plate forms. A total of 19 weldments will be produced during the course of this program.

The minimum weld process qualification tests which will be required to develop quality weldments are illustrated in Table 5. It is planned to obtain the weld chemistry and perform limited tensile tests at room temperature in both the axial and transverse directions. Metallographic samples which encompass the weld, heat affected zone, and parent metal will be sectioned for examination. These sections will be utilized for hardness transverse. The vertical columns of Table 5 indicate the minimum number of tests planned on each heat of material. A total of 144 tests are planned.

3. Welding Specification

A welding specification (AGC-90067, dated 3 August 1965) was prepared and published; copies are available on request. The weldability program began with the procurement of commercially available Hastelloy X forging stock. This first heat (#X5-2718EE) of CEVM ring forgings procured to AMS5754 specification meets the AGC-90056 specification. Air melt Hastelloy X filler wire was procured to AMS 5798 specification. These ring forgings, 17-in. ID x 23-in. OD x 5.25-in. thick were machined, ultrasonically inspected, and found to be sound.

Two of the rings (heat #X5-2718EE, SN-003 and-004) intended for the welding studies were machined with the joint preparation shown in Figure 13, while the third ring forging of this heat (SN-002) was parted into two rings each measuring 2.5-in. thick. The details of the third ring forging likewise received the weld joint

<u>FILLER WIRE</u>	<u>PARENT METAL</u>					
	<u>347 SS</u> <u>PLATE</u>	<u>HASTELLOY X</u> <u>FORGED RINGS</u> <u>(3 HEATS)</u>	<u>PLATE</u> <u>(3 HEATS)</u>	<u>HASTELLOY N</u> <u>FORGED RINGS</u> <u>(3 HEATS)</u>	<u>PLATE</u> <u>(3 HEATS)</u>	<u>INCONEL 625</u> <u>PLATE</u> <u>(3 HEATS)</u>
AMS 5798						
HEAT A (AIR MELT)	3	1				
AGC 90058A						
HEAT A (CEVM)		1	1	1	1	1
HEAT B (CEVM)		1	1	1	1	1
HEAT C (CEVM)	<u> </u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>
SUB-TOTAL WELDMENTS	3	4	3	3	3	3
TOTAL WELDMENTS - 19						

Table 4
Weld Test Program - Subtask D-2

	PARENT METAL				
	HASTELLOY X		HASTELLOY N		INCONEL 625
	<u>FORGED RINGS</u> <u>(3 HEATS)</u>	<u>PLATE</u> <u>(3 HEATS)</u>	<u>FORGED RINGS</u> <u>(3 HEATS)</u>	<u>PLATE</u> <u>(3 HEATS)</u>	<u>PLATE</u> <u>(3 HEATS)</u>
WELD QUALIFICATION TESTS PER WELDMENT					
WELD CHEMISTRY	1	1	1	1	1
TENSILE SPECIMENS					
(AXIAL)	3		3		3
(TRANSVERSE)	3		3		3
METALLOGRAPHIC	4	4	4	4	4
HARDNESS TRAVERSE	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>
 SUBTOTAL TEST SAMPLES (3 HEATS)	 36	 18	 36	 18	 36

TOTAL TEST SAMPLES - 144

Table 5
Weld Test Program - Subtask D-2

preparation shown in Figure 13. The rings were tack welded together in eight places on the OD, each tack being approximately 2-in. long. The assembled rings were mounted on a weld positioner. Dimensional measurements were recorded to evaluate distortion.

Six type 347 stainless steel plates were also machined with the double U groove joint preparation. These 1.5-in. x 6-in. x 24-in. plates were used to provide the initial welding parameters for the forged ring. Weld parameters for semi-automatic TIG welding were established on these plates using Hastelloy X filler wire as follows:

<u>Amperage</u>	<u>Voltage</u>	<u>Wire Diameter</u>	<u>Wire Feed</u>	<u>Travel</u>
180	10	0.045-in.	58 IPM	6 IPM

Using these parameters, a root pass was successfully deposited on the ID of the ring assembly. The weld was made using 18,000 joules-per-inch of travel with a ratio of filler wire deposition rate of linear travel rate of 6.1. Grinding was then performed on the OD to remove the tack-welds and lack of complete penetration.

Results of the weld penetration achieved on the first root pass in the ring weldment indicate that it may be desirable to reduce the joint land thickness of the jacket from the present 0.125 dimension to 0.075 so full penetration of the joint may be achieved on the initial ID root pass. Full penetration of the joint during the first root pass presents several advantages, foremost of which would be the elimination of the back-machining operation on the OD which is required if full penetration is not obtained eliminating an in-process jacket machining and handling problem.

Two additional root passes were deposited on the joint OD. Inert gas back-up was employed for all root passes. These root passes were inspected by radiographic and penetrant techniques and were found to be free of defects.

The Hastelloy X ring forging set on the rotating positioner for welding is shown in Figure 14. The weld preparation on the ID is identical with that of the OD but is somewhat obscured by the penetrant developer.

The tungsten inert gas automatic head is shown repositioned on the ID joint for illustration purposes. The torch, electrical and inert gas supply lines, push-type wire feed unit and cable, and torch extension arm are shown here. The

Figure 13
Weld Joint, Ring Forging

Figure 14
Automatic Welding of Hastelloy X Ring Forgings

torch extension arm and wire feed cable will have to be extended an additional 36-in. to accomplish the ID throat weld of the jacket. Procurement of these items has been initiated.

Fourteen additional passes were made alternately on the ID and OD of the ring forging in an effort to assess the problems that may arise in joining the more massive rings.

The restricted access to the ID of the rings requires a change in the method of filler wire deposition from the conventional "push" type of feed to a "pull" type mechanism. This change was incorporated.

Experience gained on this first ring weldment also indicates the desirability to have access to both ends of the nozzle during welding. The limited space on the small ID of the nozzle after the welding head and wire feed unit are in place makes it extremely difficult for a welding operator to observe the weld melt if access to the opposite end of the nozzle is denied because of fixturing.

Radiographic inspection of the Hastelloy X ring weldment after the 14th pass revealed scatter defects, i.e., gas holes, porosity, and inclusions, which were the direct result of mechanical malfunctions incurred by the wire feed system. These defects were removed by machining to remove the weld deposit to a minimum root thickness of 0.250-in. Radiographic inspection was employed after machining to assure that the defects had been removed.

The wire feed difficulty was resolved by installing an Airco HMF "pull" wire feed system which had been modified to adapt the wire feed to the ID joint configuration. Installation of a motor control unit for this wire feed apparatus, which will assure constant RPM of the wire feed motor, was completed.

Welding was resumed using the following welding conditions: shielding gas 75% He/25%A, Amperage 180, voltage 10, (0.045 dia.) wire feed 55 in./min., and a rotation travel speed of 6 in./min. The weldment was allowed to air cool after each pass with the inter-pass temperature being held below 180°F.

Several longitudinal cracks, the longest being approximately 3-in. long, were detected in the fifth weld pass. The cracks were removed by grinding; dye penetrant inspection was used to assure removal, and it was found that the defects were relatively shallow and did not extend below the root of the deposited bead.

To arrive at the optimum set of conditions which would act to lower the heat-input and increase the cooling rate, test welds were made on plate utilizing various welding conditions. The surface structure of these test welds was examined visually.

By experimentation it was found that a shielding gas mixture of 50% argon and 50% helium provided better atmosphere shielding of the molten puddle. Furthermore, since lower arc voltage at any given current level may be obtained using argon, a reduction in arc voltage from the previous 10 to an 8.5 setting was accomplished. The rotational travel speed was increased from 6 in./min. to 6.5 in./min. These changes in welding conditions caused a reduction in heat input from 18,000 joules/in. to 14,800 joules/in. and produced a somewhat narrower and more elongated weld bead which promoted an increase in the weld solidification cooling rate.

Welding was resumed on the rings using the techniques noted above. Radiographic inspection of the weld deposit was performed after completion of the 26th weld pass and no defects were detected. Welding continued through the 57th pass. The weld deposit thickness at completion of the 57th pass was 1-7/8-in. Radiographic inspection of the weld at this time revealed three tungsten inclusions of 0.040, 0.050, and 0.060-in. dia. scattered in a longitudinal direction through a four-in.-area of weld. No other defects were detected by the radiographic inspection.

Ultrasonic inspection of the weldment revealed several indications between 0.047 and 0.078 flat bottom hole equivalent size. The exact size could not be determined due to attenuation within the fusion zone. A one inch section transverse of the weld was removed from each of three indicated defect areas and from an area which contained no defect indications. The weld areas of the removed sections are currently being prepared for macro examination.

Shrinkage measurements taken periodically during welding are recorded in the shrinkage graph, Figure 15.

Certain remaining sections of the ring weldment were used in ultrasonic and radiographic inspection experiments to improve inspection techniques and defect evaluation methods.

Figure 15

Transverse Shrinkage Graph, Hastelloy X Ring Weldment (AGC S/N 002)

The root passes were deposited in the second Hastelloy X ring forgings, Heat No. X5-2718EE C.E.V.M., AGC serial No.'s 003 and 004. Erratic weldability characteristics, i.e., poor wetability and excessive foreign material in the molten puddle, observed during the deposit of the root welds led to an investigation of the weld wire which was used. N.D.T. tests and laboratory examination of the wire revealed spiral cracks, foreign inclusions, and holes. All weld wire (100 lbs) from this defective heat, X-4-2941 C.E.V.M., was subsequently rejected by AGC and a replacement order for new wire was issued.

Radiographic inspection of the root welds in this ring forging revealed porosity of maximum permissible size acceptable per AGC STD 4005, Cl. 1. The root passes were removed by machining and the original U joint configuration restored. The welding of Hastelloy X ring weldment composed of rings Serial No's 003 and 004 and two Hastelloy X plate weldments is pending delivery of replacement Hastelloy X CEVM filler wire.

4. Hastelloy X Plate Development

Test welds are being made on Hastelloy X plate, using the double U joint configuration, to determine if the grinding operation after each pass may be eliminated by using improved atmosphere shielding methods. These plates will also be used to further investigate welding parameters. Fifteen weld passes were accomplished on the plate weldment which was radiographically inspected. Each weld pass was cleaned by wire brushing; no grinding was employed.

Welding was completed on one Hastelloy X plate weldment, AGC SN-001, Heat No. X5-2752. Radiographic inspection revealed two separate lines of intermittent non-fusion the entire length of the weld. Ultrasonic inspection verified this discrepancy.

A transverse section of the weld was removed in a defect area and prepared and mounted for macro examination. Evaluation of the mounted specimen disclosed the non-fusion line was caused by positioning the torch at too great an angle during the deposit of a weld pass. This positioning error allowed a slight roll-over or cold lap at the edge of the weld bead. Deposit of the subsequent pass failed to penetrate to the bottom of the small crevice formed by the cold lap edge which resulted in the non-fusion line illustrated in Figure 16. The plate weld deposit was found to be otherwise sound. No micro cracks were found.

MAGNIFICATION 3X

ETCHANT $\text{HCl} + \text{H}_2\text{O}$

HASTELLOY X PLATE WELDMENT HEAT NO. X-5-2752 AGC SERIAL NO. 001

Photograph illustrating non-fusion lines indicated by
radiographic inspection at completion of 15th weld pass. Test
Referral No. 34466 - Log No. 51466

Figure 16
Non-Fusion Lines

5. Hastelloy N Development

The welding of three Hastelloy N Plate weldments, Heat No. 284-5-0100, AGC plate serial No's-001 through-006, was initiated. These plates were being welded under rigid restraint and like conditions, except amperage, voltage, travel speed, wire feed, and inter-pass temp., which was varied for each weldment.

Welding conditions for the three Hastelloy N plate weldments were as follows:

WIRE FEED

I.P.M.

<u>No.</u>	<u>SN Plates</u>	<u>Amps</u>	<u>Volt</u>	<u>0.045 Dia. Wire</u>	<u>Travel I.P.M.</u>	<u>Shield Gas</u>	<u>Inter Pass Temp.</u>
1.	1 & 3	170	8.5	50	6.5	50% H 50% A	Below 100°F
2.	2 & 4	225	10	90	8	50% A	400°F Max
3.	5 & 6	250	10	100	10	50% A	400°F Max

No grinding of the weld passes was performed except if required to eliminate a cold lap or an out of contour condition, in which case a rotary file was used to blend the weld edge to the existing weld deposit contour. Inter-pass cleaning was accomplished by wire brushing and wiping with white cloths saturated with freon.

Weld passes were alternated from OD to ID every second or third pass. Each plate weldment was scheduled for three radiographic inspections;

At completion of root passes

At mid-point (15th to 20th pass)

Upon completion of weldment

Following final X-ray weldments were ultrasonically inspected.

Welding has been completed on plate weldment #1 which was in final X-ray; weldment #2 has had 20 passes completed; and weldment #3 has had the root passes deposited. Results of the X-rays which have been accomplished on the plate weldments to date, have shown no defects.

6. Nozzle Weldment Tooling Development

The design concept for the Phoebus nozzle weldment tooling was approved and preliminary drawings prepared. Basically the weldment tooling consists of

fixturing which will align, support, and position the nozzle for welding on a moveable cart. Rotation of the nozzle beneath a stationary TIG welding head will be provided by variable speed rolls. The cart will provide multiple service during the welding operation, in as much as it may be used to transport the nozzle to N.D.T. stations for radiographic or ultrasonic inspection if required. On site radiographic inspection of the nozzle welds is also being investigated.

7. Welding Facility

The Phoebus project was given assurance of first priority usage of the Aerojet Cyuaga boom and weld equipment in Building 2002; thus, the welding program will remain undisturbed throughout the development program and the first Phoebus nozzle welding.

8. Weld Development Schedule

Welding development schedules, which are primarily influenced by material deliveries, are depicted in Figure 17.

9. Subcontractor Weldability Test

A weldability test was witnessed at Union Carbide Corporation, Stellite Division on 15 through 20 July 1965, by representatives of Aerojet-General Corporation. The test performed represented Hastelloy X Heat No. 260-5-2793 intended for shipment to Wyman-Gordon and deliverable to Aerojet-General Corporation under Purchase Order 201560.

Stellite welding procedure was reviewed, revised, and approved to conform to the provision of AGC-90059B, Weldability Test for Nickel Molybdenum Chromium Alloy Plate, Forgings and Wire. The weldability test witnessed was successfully completed with the exception of the provision of a required check chemical analysis which had not been completed. The filler rod used in this weldability test was Hastelloy X Heat No. X4-2941.

C. BRAZING DEVELOPMENT

The objectives of the brazing program are to develop technology and establish brazing procedures for the solution strengthened nickel-base alloys for the Phoebus-2 nozzle. The experience developed in the fabrication of Nerva 347

*PREDICTED DEL.

Figure 17
Welding Development Schedule
(25 July)

stainless steel nozzles indicates that major assembling and brazing problems can be resolved by fabricating straight and contoured nozzle sectors.

The scope of the brazing program, as applied to the nickel-base alloys, will include development of applicable braze alloy systems and associated braze joint shear strength. Straight and contoured nozzle sectors will be fabricated to define the tooling, assembling, and brazing parameters, and to establish the effects of differential coefficients of expansion. Brazed sectors will be subjected to destructive testing to evaluate the joint design. Additional testing of sectors will include hydro-testing to proof pressures and metallographic joint coverage.

A concurrent NDT engineering program will be initiated to ensure braze joint quality and reliability. It is planned that the NDT test program be correlated with metallographic examination.

Additional samples of brazed sectors will be employed to develop non-destructive testing techniques and procedures which will establish quality brazed joints.

The Phoebus-2 brazing development program is shown in Table 6. The first task was to establish the brazing process parameters which can be formulated into a tentative brazing procedure for Hastelloy X, Hastelloy N, and Inconel 625. The initial braze alloy evaluations will be performed on 0.75-in. x 4-in. x 10-in. flat sectors containing 0.012-in. U-tubes. These sectors will be utilized for destructive tests to evaluate the brazed joint.

Shear specimens will be prepared and brazed to evaluate brazed joint shear strength. Braze alloy development work is currently planned for all three nickel-base alloy combinations. The program plan illustrates that 0.75-in. x 4-in. x 60-in. flat specimens having six 0.012-in. formed U-tubes will be prepared and fabricated to establish the assembling and brazing parameters.

Full length contoured sectors which simulate the nozzle configuration will establish the final brazing parameters to be applied to nozzle fabrication. Contoured sectors will establish braze alloy placement, show braze alloy coverage, eliminate excessive run off, and finalize the brazing specification.

Table 6
 Brazing Development Program Plan

The leak population of straight and contoured sectors will be measured under leak and proof pressures. Applicable repair techniques for joint leaks need to be developed.

In the case of Hastelloy X to Inconel 625 evaluation, it is necessary to determine the effects of the differential coefficients of expansion on straight sectors and establish the possibilities of overcoming this problem prior to brazing contoured sectors.

Schematic views of present assembly techniques applied in the fabrication of Nerva nozzles and which are currently planned for Phoebus-2 nozzles are shown in Figure 18. View 1 illustrates the preplacement of 0.001-in., preformed, Nicro brazing foil in the jacket groove, which is positioned by a dielectric spacer for tack-welding.

View 2 shows the installation of 0.020-in. diameter Niro braze alloy wires along the entire joint length. The dielectric spacer provides the required spacing for channel installation during the probe tacking operation. View 3 illustrates probe tack welding of the "U" channel to the jacket wall. A thinner spacer which is employed to maintain the joint spacing and to hold the channel against the groove wall. The probe electrode is inserted into the jacket scallop and a spot weld is performed joining the channel to the jacket groove wall for brazing fixturing purposes only. View 4 illustrates the insertion of the adjacent channel. A similar technique is applied to the opposite channel leg. This operation is repeated until all "U" channels are assembled. View 5 illustrates insertion of 0.001-in. thick Nicro preformed brazing foil and a 0.005-in. full hard (Rc 35+) parent metal shim with the aid of a specially designed driving tool. The shim and braze alloy is driven completely to the base of the joint interface to ensure tight joint clearances. The assembly is furnace brazed in dry hydrogen at 1850°F with a controlled heating and cooling cycle. Views 6 and 7 illustrate the technique of sealing the shim butt ends with the application of a duplex alloy composed of 50% Palnicro 7 and 50% Nicro braze alloy powders. A second furnace brazing operation in hydrogen is then performed at 1800°F. As a final operation, precise amounts of Nicro 80 are then applied to all faying channel surfaces, and a final furnace brazing operation in hydrogen is performed at 1750°F.

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Figure 18
Braze Joint Design Comparison

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The full length contoured nozzle sector design, which is currently planned in the brazing program, is shown in Figure 19. Straight and contoured sectors provide a method of establishing process control of assembly and furnace brazing operations. This schematic view depicts the location of the braze joint shims and the method of sealing the shim butt ends. This brazing approach is currently being employed in the fabrication of NERVA nozzles.

A straight, U-tube, Hastelloy X brazed sector which is approximately 60-in. long is shown in Figure 20. This sector was prepared for the initial proposal effort. The sector is composed of a grooved Hastelloy X base and six 0.023-in. thick Hastelloy X formed U channels. The sector was brazed using the braze alloys and procedures previously described and which are identical to those presently being employed on NERVA nozzles. This sector was hydrotested to 3100 psi, which is in excess of the design requirements. Deformation and fracture of the channel side wall support precluded higher pressure hydrotests.

Successful experience by Aerojet-General in brazing U-tube nozzles for the NERVA program has lead to the continuation of the same or equivalent processes for the nickel base alloys of the Phoebus-2 system. The validity of this has been successfully demonstrated on a preliminary basis on the 60-in. sector, as previously shown. The selection of solution hardened Ni base alloys has circumvented potential problems of required surface preparation which are characteristic to the precipitation hardening alloys systems.

Further work to be accomplished includes the scaling up of experimental samples to full size contoured hardware, evaluating the braze alloy diffusion problem, and overcoming differential expansion mismatch in the event that channel material must be of a different alloy than that employed for the Phoebus-2 jacket.

1. Brazing Application

Brazing specimens of flat stock 347 stainless steel material utilizing the E.D.M. sections discussed in a following paragraph were assembled and brazed at the Pyromet Company. These specimens display good braze coverage.

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Figure 19
Full Length Contoured Sectors

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Figure 20
60 In. Hastelloy X-To- Hastelloy X, Straight U-Tube Sector

The results obtained on the diffusion studies of Nicoro-80 braze alloy on Hastelloy X were received. The ultimate tensile strength data obtained are useful; however, the tests to obtain yield strengths and percent elongations used a standard two-in. gage length and localized yielding could occur in the non-uniform thickness of the deposited braze alloy. Interpretation of these data with respect to embrittlement should be qualified with micro-examination. A Knoop hardness traverse across the base material, interface region, and braze alloy deposit did not reveal evidence of embrittlement since the hardness of the diffusion zone lay between the base material and the soft "as deposited" braze alloy. The yield and elongation data, using a linear gage length across a nonuniform structure, should be used as comparison tests only.

The following test programs were recommended:

Tensile specimens should be cycled for the alloys and temperatures listed in Figure 21. The surface of the specimens will be ground to a uniform thickness; however, the specimen structure in the gage lengths will consist of base material and a dendritic type nonuniform diffusion zone.

The specimen design for micro-examination incorporates a predrilled hole of known diameter in either plate or bar stock and investigations should be accomplished for the alloys and temperatures listed in Figure 22. The extent of diffusion after brazing can be quantitatively measured with an optical micrometer and microhardness traverses should be made on all specimens with a Knoop hardness tester.

Simulated seal joint specimens should be cycled for the alloys and temperatures listed in Figure 23. These specimens will be tested in tension at room temperature at 1200°F.

Data obtained from the outlined test program will yield a quantitative measure of the amount of diffusion, identification of embrittling constituents found in the diffusion zone, and degree of ultimate tensile strength reduction for the simulated joint specimens for the braze alloys and braze temperatures investigated.

Standard Flat Tensile
Specimen per AGC 255324

Note: Surface grind specimens
to .011 inch (Tentative)
to remove braze alloy
mound in the gauge length
prior to testing.

Specimen Configuration

<u>Braze Alloy</u>	<u>Braze Temperature</u>	<u>Time at Temperature</u>
Palniro-7	1975°F	3 hours
Copper	2025°F	3 hours
Nicoro 80	1750°F	3 hours
Nioro	1850°F	3 hours

Braze Alloys to be Evaluated

Specimen Requirements: Five specimens for each condition to be tested
at room temperature and at 1200°F. Total specimen requirement - 40
specimens.

Figure 21
Brazed Tensile Specimens

SPECIMEN CONFIGURATION

Chemistry %						
<u>Alloy</u>	<u>Trade Name</u>	<u>Au</u>	<u>Ni</u>	<u>Pd</u>	<u>Cu</u>	<u>Liquidus</u>
A	Palniro	50	25	25		2050
B	Copper				100	1981
C	Palniro 7	70	22	8		1899
D	Nicoro	35	3		62	1886
E	40Au-60Cu	40			60	1832
F	Nicoro	82	18			1742
G	Nicoro 80	81.5	2.0	2.0	16.5	1597
						<u>Solidus</u>
						2016
						1981
						1841
						1832
						1796
						1742
						1670

ALLOYS TO BE INVESTIGATED

Temperature °F (All Times at
 Temp = 3 hrs)

Alloys to be Included

1700	G						
1750	G	F					
1800	G	F					
1850	G	F	E				
1900	G	F	E	D	C		
1950	G	F	E	D	C		
2000	G	F	E	D	C	B	
2050	G	F	E	D	C	B	A
2100	G	F	E	D	C	B	A
2150	G	F	E	D	C	B	A

Total Specimen Requirement = 10

Figure 22
 Braze Alloy Diffusion Studies

SPECIMEN CONFIGURATION

Condition 1 (NERVA Cycle)	(Nicro) + (Nicro + Palniro 7) + (Nicro 80) 1850-3 hrs 1825-3 hrs 1750-3 hrs
Condition 2 (Alternate cycle)	(Palniro 7) + (Palniro 1 + Palniro 7) + (Niro) 1975-3 hrs 1950-3 hrs 1825-3 hrs
Condition 3	(Nicro) 1850-3 hrs
Condition 4	(Nicro 80) 1750-3 hrs
Condition 5	(Copper) 2025-3 hrs
Condition 6	(Palniro 7) 1975-3 hrs

BRAZE ALLOYS TO BE EVALUATED

Specimens Requirements: Five specimens for each condition and three control specimens for each temperature to be tested at room temperature and at 1200°F. Total specimen requirement = 60 brazed specimens and 36 parent metal specimens.

Figure 23
 Simulated Brazed Joint Specimen

2. Nozzle Grooving Development

The nozzle jacket material requiring grooving will be Hastelloy X, which has a machineability rating of 30 as compared to a rating of 50 for 374 stainless steel, based on 100 for mild machining steel. It is apparent the 40% decrease of the Hastelloy X suggests that special attention is required to reduce this predicted increase in grooving time by conventional methods. However, the jacket grooving cannot realistically be considered conventional machining simply because standard tools are used. The arm length or reach needed to machine a groove places the slitting saw in a precarious position to hold close tolerances, notwithstanding that the saw has no side cutting teeth to recover alignment in the event of true position run out during the cut. Aerojet-General has considerable experience in grooving nozzle jackets of 347 Stainless Steel for the NERVA program. This background will be upgraded to the Phoebus-2 nozzles through a development program, to optimize machine feeds, speeds, alignment control, and slitting saw materials. An alternate parallel nozzle grooving development process is also currently in effect to use an electrical discharge machining process. An initial attempt to elox a full length groove all at once in a 60-in.-long plate was accomplished. Electrical discharge machining has the advantage over conventional machining leaving no burrs. Assembly and brazing requires scrutiny of the groove corner radii and the groove surface finish and contamination.

The cleaning requirements for an E.D.M. groove were evaluated to assure a non-contaminated joint for brazing. Specific recommendations were made for incorporation into an A.G.C. process specification.

3. Electrical Discharge Machining

A 60-in.-long 347 stainless steel segment was grooved by electrical discharge machining to evaluate the feasibility of the process in regards to E.D.M. machining of the Phoebus jacket. The completed part was sectioned. Sections were subjected to metallography and inspection as to surface hardness and contamination. Figures 24 and 25 show E.D. M. grooves. Irregularities were found that would be detrimental to brazing, therefore, sections were assembled and brazed similar to procedures planned for Phoebus-2 nozzles. The brazed sections displayed good alloy coverage in view of poor dimensional control of the groove. Further development must be done to accomplish grooving dimensional requirements.

Figure 24
Surface Condition of Electrical Discharged Machined Grooves

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Figure 25
Bottom Contour of Electrical Discharged Machined Grooves

4. Channel Forming Methods

There were two methods originally under consideration to form channels for the Phoebus-2 nozzles. Most of Aerojet-General's experience in forming U-tube coolant channels involves forming strip stock around the punch by wiping the material across the face of a die in stages similar to deep draw forming. An alternate method to deep draw channel forming is the stretch-form process. All stretch-forming methods employ constant longitudinal tension on the strip stock to alleviate unwanted compressional forces that tend to wrinkle the material.

Both approaches could satisfy design requirements in view of the larger throat radii of Phoebus nozzles; however, less spring-back, and the longer leg length of these channels, indicated the stretch-form method to be the better choice. The forming wrinkle problem occurs both in the throat radii (which places the channel leg in compression) and in the cylindrical-to-convergent section radii (which places the crown in compression). Development in this area was started and Figure 26 shows initial Hastelloy X tubes formed by the stretch form method. These tubes have a designed crown thickness taper which was a requirement when this development phase was started. Future tubes will not have the thin crown requirement although some areas of crown and leg will thin within print tolerances. The illustrated tubes were not stress-relieved during or after the forming process. The NERVA tube was formed from hardenable 347 austenetic stainless steel channels with several interstage anneals between the forming or deep drawing operation, but final draw of 15% cold work was not annealed. Aerojet plans to optimize process annealing during the forming development program.

After deep draw forming, the channels have a trim edge that must be removed to match the tube leg contour to the contour of the bottom of the groove in the nozzle jacket. A close fit-up of these parts is essential to maintain print tolerances and braze joint efficiency. The trim edge is removed on NERVA channels by clamping the channel on its side after filling the interior with a plaster and milling the trim edges to the correct contour, utilizing a tracer attachment and contour template. Stretch forming will allow predetermined blanks which eliminate edge trimming if tolerances permit.

Figure 26
Stretch Formed Development U-Channels

The equipment required to form channels by the deep draw process will be a conventional die with a male punch and female wiping die. The forming operation necessitates over-riding the room temperature yield strength material properties; therefore, the die and press will need greater capacity than that required for NERVA nozzles. This requirement for greater capacity also applies to the stretch forming methods where only the male punch is used for final form after the strip has been formed in the cross sectional configuration. The longitudinal tension is usually adjustable on most stretch forming machines.

Aerojet explored the capabilities of several vendors that could supply channels to our requirements. The development program, in addition to evaluating vendor capabilities, included refinement of existing specifications, inspection and quality control criteria, and N.D.T. practices.

Four vendors that responded to an advance request for quotes regarding Phoebus "U" tube channel forming were invited to AGC for review of their facilities and fabrication processes prior to final requests for quotes. Recommendations for product improvement and cost savings were solicited from each vendor during these meetings. Material requirements, design changes, and fabrication processes were scrutinized for problem areas. Two vendors, the Marquardt Corporation and Precision Sheet Metal Company, advocated forming by the wipe die or deep draw process while Straza Industries and Jet-Air Corporation would form a channel by stretch form methods. Final requests for quotes established Straza Industries as Aerojet's choice to form channels for the Phoebus-2 nozzle.

The facilities and capabilities of Straza Industries were reviewed with regard to tube forming.

5. Forging Development

Four concepts of forging requirements were originally envisioned as usable to produce a Phoebus-2 nozzle jacket. A monolithic forging concept shown at the top of Figure 27, which is similar to those presently in use for NERVA nozzles, would permit avoidance of joining development. However, the concept of a one piece forging (or even a two-piece forging) had to be discarded because of existing melting and forging capacity limitations.

Figure 27
Forging Concepts

Two of the country's leading producers of forgings were requested to propose on the ingot size limitations of the Union Carbide (Haynes Stellite) Company the existing weldability and forgeability specifications. Stellite had melted two, 24-in.-dia, 10,000 pound, Hastelloy X Consumable Arc Vacuum melted ingots and expressed confidence they could melt 28 or 30-in.-dia Hastelloy X ingots up to 32,000 pounds. However, new larger copper molds would be required to make this melt. In addition, Stellite lacked equipment for handling and forging this large ingot, therefore, the responsibility for the billet would be shifted to the forger. The original request for proposals stipulated omission of weld joints forward of the throat.

One company chose to stay with the present forging state-of-the-art and actually offered a feasible approach with the maximum possibility of making the part satisfactory at this time. Their facilities consist of a 23,000 ton press with 16 feet between platens, and a 200-in. stroke. Counter-blow hammers are available for the fabrication of 24-in. ingots. They had experience working Hastelloy X in 24-in. ingots and had completed 60-in.-dia rolled ring forgings. This large ingot size needs full work to effectively utilize the ring rolling process which removes centerline effects of the ingot. Extrusion would precede the ring rolling and align defects in the circumferential direction corresponding with the hoop stress. The proposal involved four separate forgings. Figure 27, lower view depicts the selected method of forging a rolled ring of the 20-in. billet of 3,500 pound capacity for the aft divergent section; a 20-in. billet of 2000 pound capacity for the forward divergent section; a proposed 24-in. ingot of 6000 pounds, contour extruded, rolled center, and closed-die forged for the convergent section; and a 16-in., 1500 pound, forged ring for the cylindrical section. Separation of the cylindrical section from the convergent section with the resultant unwanted weld joint at the time reduced the demand for the untried 30-in. ingot and made it possible to use a 10,000 pound ingot of 24-in.-dia.

The second company took no exceptions to Aerojet-General's request and initially-proposed a three forging concept utilizing the 30-in. untried ingot. Handling and processing an ingot of this size was outside the capabilities of this vendor, therefore, subcontracting was included in their proposal. A 28-in. billet with a wrought structure weight of 10,200 pounds was proposed to make the convergent and

cylindrical section in one piece. A 22-in.-dia, 4,200 pound, billet was proposed for the forward divergent section and a 9,900 pound billet would be required for the aft ring.

A design review indicated that a weld would be acceptable in the cylindrical section forward of the flange. Vendors were requested to resubmit a new proposal based on the new design weld. The new proposal from the second forger employed only three ingots of 24-in.-dia derived from one, 30,000 pound, air melt broken down into the cylindrical section, which is a rolled-ring weighing 2450 pounds; the convergent section and the divergent section each weighed 6700 pounds, as shown in Figure 27, center view. The convergent and divergent section ingots of 55-in. will be upset, drawn out, and re upset to put the work in prior to going into a blocker die. Final forge configuration is established in a closed-finish die. The forging is then solution-heat treated, tested, and rough machined. In the interest of expediency, the weld groove may be machined at this time while the forgings are chucked up in the machine.

Wyman Gordon Company was chosen as the forging subcontractor for Phoebus -2 nozzles based on final quotes.

6. Procurement

The procurement activities for the Phoebus Materials Evaluation Program have progressed to the point where the latest required amendments have been made to existing specifications and all Hastelloy X, N and 625 materials have been placed on purchase orders reflecting the latest specification revision and price quotes. Purchase orders for Hastelloy X were limited to Haynes Stellite because of patent restrictions. The Inconel 625 and Hastelloy N materials are on Purchase Orders to the Allvac Company. There have been numerous changes in the past six months in the material requirements from the commercial items Aerojet intended to use for initial development. Prior to the requirement to apply Oak Ridge specifications, some materials were ordered against A.M.S. specifications. Further ordering was curtailed until these specifications were approved by SNPO. This review resulted in the first specifications revision and material ordering was resumed. Procurement was again halted because of radiation effects and the previously discussed boron problems until changes could be agreed upon and

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incorporated in another specification revision. The latest changes to the specifications reflect agreement between SNPO, ORNL, and Aerojet-General.

These changes are as follows:

All Hastelloy X, Hastelloy N, and Inconel 625 Aerojet-General specifications limited boron content to ten parts-per-million.

Phosphorous was limited to 200 parts-per-million in Hastelloy X.

Plate, sheet, strip, and bar room temperature ultimate tensile strength changed to 95,000 psi and yield changed to 40,000 psi.

Cross head strain rate above yield was modified to 0.5 in.-per-minute and the die-penetrant requirements on the AGC weldability specification were changed to three applications only: one on the root pass, one on the midway cover passes, and one on the final cover passes (comparable to the previous requirement that each layer of passes be penetrant-inspected).

Molybdenum range for Allvac 625 was changed to allow 7.5 - 10.0.

Aerojet-General allowed exceptions to the specifications on their purchase orders for plate and forgings of Hastelloy X and waived the ten parts-per-million boron requirement in favor of 50 parts-per-million to expedite procurement for program needs.

A review of the materials procurement status is shown in Tables 7 through 9. All of the Hastelloy X material requirements (Table 7) are on purchase orders. The material forms requiring the A.M.S. specifications were ordered some time ago, all of these, except for the 0.045-in.-dia CEVM weld wire are presently at AGC. Three different heats of ring-forgings, plate, strip, and weld wire are ordered. The tentative quoted lead times required to get this material are listed in Table 7; however, delivery dates are contingent on weldability specification requirements.

Table 8 reveals Hastelloy N material requirements closely resembling those of Hastelloy X. The controlled chemistry items are those ordered early in the program. The AGC specification items are currently on purchase orders. The material forms being ordered in three heats are identical to those of Hastelloy X.

Table 9 shows the Inconel 625 requirements presently on purchase orders. Three heats will be ordered of the same material forms as Hastelloy X and N, with the exception of the ring forgings which will not be procured for this alloy.

<u>MATERIAL FORM</u>	<u>MELT</u>	<u>SIZE</u>	<u>QTY</u>	<u>NO. OF HEATS</u>	<u>SPECIFICATION</u>	<u>REQ.</u>	<u>REQUISITION RELEASE DATE</u>
WELD WIRE	AIR	.045 DIAMETER	50 LBS	1	AMS 5798	475409-1	4-1-65
WELD WIRE	CEVM	.045 DIAMETER	50 LBS	1	AMS 5798	475409-1	4-1-65
WELD WIRE	AIR	.062 DIAMETER	25 LBS	1	AGC 90058A	479487	4-6-65
WELD WIRE	CEVM	.062 DIAMETER	25 LBS	1	AGC 90058A	459487	4-6-65
WELD WIRE	CEVM	.062 DIAMETER	100 LBS	2	AGC 90058A	459495	4-10-65
FORGED RINGS	CEVM	23"ODx17"IDx6"	4	1	AMS 5754	478292-1	4-1-65
FORGED RINGS	CEVM	23"ODx17"IDx6"	6	2	AGC 90056A	468135	4-10-65
PLATE	CEVM	1.5"x4"x12"	6	1	AMS 5536	475410-1	4-1-65
PLATE	CEVM	1.5x6"x12"	4	2	AGC 90057A	468136	4-10-65
BAR	CEVM	.750 DIAMETER	150 FT	1	AGC 90060	459483	4-7-65
SHEET	CEVM	12"x48"x.050"	1	1	AGC 90057A	459493	4-7-65
STRIP	CEVM	8"x90"x.012 ^{+.001}	12	3	AGC 90057A	459493	4-7-65
				MATERIAL - STOCK			
				FORGING - 4-6 WEEKS			
				AIR MELT - (.045 - 2-3 WEEKS			
				(.062 - 8-10 WEEKS			
				CEVM (.045 - 8-10 WEEKS			
				(.002 - 8-10 WEEKS			
				PLATE - - - - - 8-10 WEEKS			
				BAR - - - - - 8-10 WEEKS			
				SHEET/STRIP - - - 10-12 WEEKS			

Table 7
Test Program Material - Hastelloy X

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							REQUISITION RELEASE
<u>MATERIAL FORM</u>	<u>MELT</u>	<u>SIZE</u>	<u>QTY</u>	<u>NO.OF HEATS</u>	<u>SPEC.</u>	<u>REQ.</u>	<u>DATE</u>
WELD WIRE	AIR	.045 DIAMETER	50 LBS	1	CONTR. CHEM.	475414-1	4-1-65
WELD WIRE	CEVM	.045 DIAMETER	50 LBS	1	CONTR. CHEM.	475414-1	4-1-65
WELD WIRE	CEVM	.062 DIAMETER	25 LBS	1	AGC 90058A	479722	4-6-65
WELD WIRE	CEVM	.062 DIAMETER	100 LBS	2	AGC 90058A	459496	4-10-65
FORGED RINGS	CEVM	23"ODx17"IDx6"	9	3	AGC 90056A	459480	4-6-65
PLATE	CEVM	1.5"x4"x12"	6	1	CONTR. CHEM.	475408-1	4-1-65
PLATE	CEVM	1.5"x6"x12"	4	2	AGC 90057A	479725	4-10-65
BAR	CEVM	.750 DIAMETER	150 FT	1	AGC 90060	479723	4-7-65
SHEET	CEVM	12"x48"x.050	1	1	AGC 90057A	459493	4-7-65
STRIP	CEVM	8"x90"x.012 ^{+ .001}	12	3	AGC 90057A	479717	4-7-65
		RING FORGINGS - -	MATERIAL -	8-10 WEEKS			
			FORGING -	4-6 WEEKS			
			AIR MELT {	.045 - 12 WEEKS			
		WELD WIRE - - - -		{ .062 - 11-12 WEEKS			
			CEVM {	.045 - 15 WEEKS			
				{ .062 - 15-16 WEEKS			
LEAD TIME		PLATE - - - - -	10 WEEKS				
		BAR - - - - -	12 WEEKS				
		SHEET - - - - -	20 WEEKS				
		STRIP - - - - -	20 WEEKS				

Table 8
 Test Program Material - Hastelloy N

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<u>MATERIAL FORM</u>	<u>MELT</u>	<u>SIZE</u>	<u>QTY</u>	<u>NO.OF HEATS</u>	<u>SPEC</u>	<u>REQ</u>	<u>REQUISITION RELEASE DATE</u>
WELD WIRE	CEVM	.062 DIAMETER	75 LBS	3	AGC 90058A	468157	4-10-65
PLATE	CEVM	1.5"x36"x12"	9	3	AGC 90057A	459492	4-7-65
BAR	CEVM	.750 DIAMETER	150 FT	1	AGC 90060	459490	4-7-65
SHEET	CEVM	12:x48"x.050"	1	1	AGC 90057A	459494	4-8-65
STRIP	CEFM	8"x90"x.012 ⁺ .001	12	3	AGC 90057A	459494	4-8-65

LEAD TIME	WELD WIRE	10-15 WEEKS
	PLATE	10-15 WEEKS
	BAR	10-15 WEEKS
	SHEET	10-15 WEEKS
	STRIP	10-15 WEEKS

Table 9
 Test Program Material - Inconel 625