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SACRAMENTO PLANT

REPORT NO. RP-DR-0005

TO

SPACE NUCLEAR PROPULSION OFFICE - CLEVELAND EXTENSION

PHOEBUS-2 SIX MONTHS MATERIALS DEVELOPMENT REPORT
FOR 15 AUGUST 1966 THROUGH 15 FEBRUARY 1967

(U)

MARCH 1967

CONTRACT SNPC-35

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Space Nuclear Propulsion Office
Cleveland Extension
National Aeronautics and Space Administration
21000 Brookpark Road
Cleveland, Ohio 44135

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Subject: Six Month Materials Report, RP-DR-0005,
Contract SNPC-35 (U)

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Very truly yours,

AEROJET-GENERAL CORPORATION

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Manager, Administration
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REPORT NO. RP-DR-0005

PHOEBUS-2 SIX MONTHS MATERIALS DEVELOPMENT REPORT
FOR 15 AUGUST 1966 THROUGH 15 FEBRUARY 1967

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PHOEBUS-2 SIX MONTHS MATERIALS DEVELOPMENT REPORT
FOR
15 AUGUST 1966 THROUGH 15 FEBRUARY 1967

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FOREWORD

This report (for the period 15 August 1966 through 15 February 1967) 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, development, studies and other efforts of the contractor relating to materials and their processing for use in fulfilling the technical requirements of this contract"

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I. INTRODUCTION AND SUMMARY

A. INTRODUCTION

This report documents and summarizes the results of final materials development activities, as well as the results of special testing and activities in support of nozzle fabrication.

The completion of low-cycle fatigue testing (formerly called biaxial testing) concludes the materials properties program. All brazing development and evaluation also is completed. Future welding effort encompasses only nozzle fabrication support. Procurement of the development material, Allvac 625, concludes this activity.

B. SUMMARY

The design-properties testing program was completed and test results reported for the grooved-tensile and low-cycle fatigue specimens. The grooved-tensile tests revealed that the braze joint carries its share of the hoop-tension loads. Low-cycle fatigue tests indicated a satisfactory thermal-strain cycle life for Hastelloy X in the Phoebus brazed condition.

The engineering and design effects of nickel plating on the braze cycle have been evaluated with satisfactory results. The shear strength of the joint was shown to be unaffected by the thickness of the plating. The extra braze-cycle resulted in only a small decrease in the tensile elongation of the jacket material.

Welding of the last nozzle-jacket assembly was accomplished successfully in September 1966. In addition, several procedures were evolved for successful repair of tubes and an improperly drilled coolant hole. Welding of the tori to the nozzle jacket is proceeding satisfactorily.

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All material procurement has been completed. INOR 8 and Allvac 625 stock are being held for future SNPO-C disposition.

All full-scale nozzle forgings have been procured and welded. The final and current test results from the individual annealed forgings are summarized by part number and heat number.

The activities of the Phoebus Materials Department are essentially terminated with this report. The remaining effort will consist of: (1) leak repairing of Nozzle S/N 880604; (2) assembly, brazing, and leak repairing of Nozzle S/N 880605; and (3) monitoring of tori welding on Nozzles S/N 880604 and 880605.

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II. DETAILED DISCUSSION

A. PROPERTIES PROGRAM

The mechanical and physical properties of Hastelloy X were reported in the preceding Six Months Material Development Report, RP-DR-0004. All data have been compiled and a rough draft of a summary report is being prepared. Report subjects will include physical metallurgy, melting, primary fabrication, secondary fabrication, physical properties, and mechanical properties. Results of special testing reported below will also be included.

1. Special Testing--Grooved Tensile

Special testing has been performed to evaluate the stress-concentration and thermal-stress conditions expected in the nozzle jacket. The tests included grooved-tensile and low-cycle fatigue specimens.

Tensile tests were conducted on grooved specimens of the kind and dimension shown in Figure 1. The groove was appropriately filled with metal strips to simulate the jacket joint. The specimen was then furnace-brazed prior to testing. However, test results were not fully conclusive because of failures in the specimen loading pin-hole and grip areas. Test results with the grooved and parent-metal (ungrooved) specimens (Heat No. 260-5-2813) exposed to the Phoebus braze cycle were:

Room Temperature Tensile Tests of Grooved
and Ungrooved Hastelloy X Jacket Material

<u>Specimen Type</u>	<u>UTS* (ksi)</u>	<u>Stress* at Grip Failure (ksi)</u>	<u>0.2% YS* (ksi)</u>	<u>Elongation (%)</u>
Parent Metal (ungrooved)	114.8	--	50.3	31
Grooved	--	97.6	--	13
Grooved	--	101.0	--	11.5
Grooved	--	100.1	--	10.5
Grooved	--	97.0	56.2	10

*Computed on basis of load and total or gross area of gage (0.9 by 1.0 inch)

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Figure 1

Grooved Tensile Specimen

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All grooved specimens exhibited some small "tear-type" cracks emanating from the bottom of the grooves. The stress at the grip-failure load, based upon the area under the groove (0.8 by 1.0 inch), varied from 110.1 to 113.9 ksi, or extremely close to the parent-metal tensile strength. The results of notched tensile testing (see NRO publication RP-DR-0004) would have indicated failure at approximately 111.0 ksi, based upon an overall notched-to-unnotched tensile ratio of 0.97. In an effort to verify grooved-specimen strength, two specimens were re-machined to a narrower gage-width (0.75 as compared to 0.9 inch) to prevent failure in the grip ends. The specimens were tested at -320°F to accentuate notch sensitivity because of reduced material ductility. The test results were:

-320°F Tensile Tests of Grooved Hastelloy X Jacket Material

<u>UTS*</u> (ksi)	<u>0.2% YS*</u> (ksi)	<u>Elongation</u> (%)	<u>Failure Location</u>
139.6	80.9	12	Parent-metal between grooves
125.0	81.0	9	Through groove

*Based upon load and total or gross area of gage (0.75 by 0.9 inch)

Tensile strengths based upon the area under the groove (0.75 by 0.8 inch), rather than the gross cross-sectional area, are 160.0 and 145.0 ksi, respectively. The following data correspond to the parent-metal (ungrooved properties) at -320°F:

-320°F Tensile Tests of Ungrooved Hastelloy X Jacket Material

<u>UTS</u> (ksi)	<u>0.2% YS</u> (ksi)	<u>Elongation</u> (%)	<u>Comments</u>
139.0	80.9	17	Standard braze-cycle
138.0	84.2	13	Standard braze-cycle
130.0	81.7	11	Standard plus extra braze-cycle
131.0	81.9	11	Standard plus extra braze-cycle

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The data reveal that the braze joint carries a satisfactory share of the loads involved.

2. Special Testing--Low-Cycle Fatigue

The initial low-cycle fatigue (thermal-stress simulation) testing was unsatisfactory because of excessive yielding (elongation and area reduction) of the test specimen* when loaded under a constant axial-tensile load and simultaneously subjected to reversed bending stresses imposed by a constant lateral-deflection (see Figure 2). The specimens also work-hardened rapidly within the first few cycles to the degree that lateral deflection was reduced to a minor level to maintain surface-strain requirements. This low-level deflection was indicative of low dynamic-stress in the specimen.

A review of the results and the plastic-strain mechanisms involved was made and the testing method revised. The new test eliminated axial-tensile loading to minimize specimen elongation, area reduction, and work-hardening. The specimen was tested as a simple free-ended beam in reversed two-point bending (the same as shown in Figure 2 but without tensile loading) and instrumented with post-yield strain gages. The strain level was established at 8000 microinches (240 ksi equivalent elastic-stress), or a 16,000 microinches range.

The first specimen (Heat No. 261-5-4001) was bent with controlled deflections in tension and compression to provide the required surface strain of 8000 microinches at room temperature. The test fixture is shown in Figure 3. The specimen failed at 950 cycles; however, the surface-strain range varied from 16,000 to 11,000 microinches as a result of work-hardening and test method inexperience. Work-hardening caused the bend radius to change: thus, the surface strain varied although the specimen was under constant deflection (constant load). In the latter 400 cycles of this test, the lateral deflection was increased to maintain the 16,000 microinches of

*All specimens were exposed to a simulated Phoebus braze-cycle

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Figure 2
Low-Cycle Fatigue Specimen (Lower) and
Original Test Method (Upper)

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Figure 3
Low-Cycle Fatigue Test Assembly

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surface-strain range. The second specimen (Heat No. 260-5-4000) failed after 545 cycles at room temperature. All but a few of the initial cycles were at the specified total strain-level. The second specimen, therefore, represented a more accurate life at the required strain-level than did the first specimen.

The third low-cycle fatigue specimen (Heat 261-5-4001) was placed in the testing fixture with post-yield strain gages on both sides. The specimen was tested and strained in the same manner as the room-temperature tests except that the temperature was reduced to -320°F. The full-cycle strain range was 16,000 microinches in reversed bending. Failure occurred at 934 cycles. Figure 4 depicts the typical load-deflection curve instrumentally plotted during testing and illustrates the load changes during the last four cycles as the sample failed.

The increased number of cycles-to-failure (at -320°F rather than room temperature) was as anticipated since the fatigue strength of a material increases as the tensile strength increases with decreasing temperature. This is illustrated by the average tensile properties of brazed Hastelloy X at room temperature and -320°F:

Average Tensile Properties of Brazed Hastelloy X

<u>Temp (°F)</u>	<u>UTS (ksi)</u>	<u>YS (ksi)</u>	<u>Elastic Modulus (10^6 psi)</u>
Room	93.3	45.0	30.0
-320	117.0	71.6	32.0

Utilizing the stress-strain curves at the respective temperatures (see Figure 5), the 240-ksi equivalent thermal-stress, based upon 8,000 microinches of strain (0.8%), produces the following actual stresses (based on first-cycle loading):

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Figure 4

Typical Load-Deflection Curve

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Figure 5

Hastelloy X Stress-Strain Curve

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Total and Plastic Strain for Hastelloy X

<u>Temp (°F)</u>	<u>Total Strain (%)</u>	<u>Plastic Strain (%)</u>	<u>Actual Stress (ksi) at 0.8% Strain</u>
Room	0.8	0.63	52.0
-320	0.8	0.55	80.5

Plotting the actual stress at each temperature versus the cycles-to-failure with ultimate tensile stress (at one cycle for failure) provides a preliminary S-N curve, on log-log coordinate paper, as shown in Figure 6. The curve utilizes one cycle as the ultimate tensile-stress failure point; however, in a reversed bending analogy, a tensile test technically represents failure in 1/4 cycle. This produces a small variance in the slope of each curve.

Although admittedly preliminary, the data indicate an adequate life-margin at an equivalent thermal-stress of 240 ksi. Thermal-stress equivalencies greater than 700 ksi are required to induce failure in less than 10 cycles.

3. Microstructure and Fracture Morphology

The increase in hardness (strength) of the test specimens was consistent with the progressive change in the load-versus-deflection curves: i.e., the hardness increased in proportion to the bending strain, and the neutral axis, which was subjected to the least strain, resulted in a "slight-to-no-change" in hardness. The microhardness surveys are shown in Figures 7 and 8. The scatter of values was partly attributable to the hard primary-phase concentrations in the microstructure and to the grain orientation of the large-grain forgings. Also there appears to be bands of hardness (illustrated by the dashed lines in Figure 8) for the specimen from Heat 260-5-4000.

It should be noted that all three specimens showed an increase in hardness at 0.004 to 0.010 inch below the surface when compared

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Figure 6

Preliminary S-N Curve

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Figure 7

Microhardness Profile, Room Temperature and
Liquid Nitrogen Tests, Heat 260-5-4001

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Figure 8

Microhardness Profile, Room Temperature Test,
Heat 260-5-4000

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to a below-surface depth of 0.002 inch. This effect may be due to: (1) the free surface on the strain distribution, so maximum plastic-deformation was affected at 0.008 to 0.010 inch below the surface; or (2) the induced surface compression-strain (on the reverse cycle) which produced a counteracting effect and caused maximum tensile-strain below the surface of the specimen.

The difference in maximum microhardness between the room temperature and the -320°F specimens of Heat 260-5-4001 (shown in Figure 7) may have been expected from the greater plastic-deformation exhibited by the failed specimens as shown in Figures 9 and 10, respectively. There was some difference in the history of testing of these two specimens. The room-temperature specimen was originally tested at constant deflection which resulted in a lower true surface-strain at the center as the material strain hardened. After about 280 cycles, the amplitude was increased to provide the originally desired surface-strain level of 8,000 microinches in both directions (16,000 microinch total range). The specimen tested at -320°F was maintained at a constant deflection throughout the test, hence less strain hardening occurred. It is also significant that the yield strength at -320°F is considerably higher than at room temperature; thus, for a given plastic deflection, a larger component is contributed by the elastic portion of the stress-strain curve than that which occurs at room temperature.

The general microstructures were normal for Hastelloy X forgings, with large grains and the primary phase in stringers (see Figure 11). The outer surfaces of the failed specimens exhibited transgranular cracking (as shown in Figures 12 and 13) generated at a distance from, and parallel to, the actual fracture surfaces. Also noted in the microstructures was an irregular displacement of the grain boundaries in the heavily strained surface areas (see Figures 12, 13, and 14). At the center of the specimen thickness, the fracture surface does not exhibit this grain-boundary shifting or displacement (see Figure 15).

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Figure 9

Failed Specimen from Heat 260-5-4001 (Unetched and slightly enlarged. Tested at room temperature. Initial fracture was from the bottom surface. Note localized reduction of area, present on all four surfaces but greatest on edges.)

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Figure 10

Failed Specimen from Heat 260-5-4001 (Unetched and slightly enlarged. Tested at -320°F . Fracture location is about 0.2-inch away from loading contact area. Strain gage shows as dark area at left. No significant area reduction observed. Initial fracture was on top surface)

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Figure 11

General Microstructure from Heat 260-5-4001 (Tested at room temperature. Etchant: Schartz. Enlarged 100 times. Longitudinal view left to right: tangential to original ring forging. Stringer particles are primary phase.)

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Figure 12

Longitudinal Section Near Surface in Compression in Final Bend of Specimen from Heat 260-5-4000 (Tested at room temperature. Two transgranular cracks shown in a plane parallel to fracture surface. Arrow points to grain boundaries which were irregularly shifted. Etchant: Schartz. Enlarged 250 times)

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Figure 13

Tension Surface of Specimen from Heat 260-5-4001 (Tested at room temperature. Shows transgranular cracks parallel to fracture surface in tension during last bend. Grain boundary shifting is shown by arrows. Etchant: Schartz. Enlarged 250 times.)

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Figure 14

Room Temperature Specimen from Heat 260-5-4000 (Transverse section near surface under compression in last cycle. Plastic strain has resulted in grain boundaries displacement or shifting. Etchant: Scharitz. Enlarged 250 times.)

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Figure 15

Fracture Surface, Near Origin, of Specimen from Heat 260-5-4000
(Tested at room temperature. Etchant: Schartz. Enlarged 100 times)

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The fracture surfaces of the room-temperature specimens exhibited a two-mode failure, originating as typical transgranular fatigue (as shown in Figures 12 and 13) and progressing subsequently to mixed trans- and intergranular-fracture (as depicted in Figures 16 and 17). The transition from the transgranular- to the intergranular-fracture mode is not readily evident from the fracture surfaces directly; however, while close examination of Figures 18 and 19 reveals a slight difference in appearance no such transition is discernible in Figure 20.

Figure 21 illustrates a failed specimen reflecting the typical failure emanating from the tensile surface and the resulting permanent set. While the original specimens tested with the axial-tensile load resulted in about 0.5 inch elongation of the entire ten-inch length of the specimens, and approximately 5% reduction in area at failure, the current specimens exhibited only 0.008 to 0.017-inch extension and approximately 1% reduction in area.

B. WELDING

1. Nozzle Jacket

Welding was completed 12 September on nozzle-forging assembly S/N 880606. The high-level of weld quality attained on this nozzle, the final nozzle weldment of the Phoebus-2 program, reflected the refinements and improvements in the weld procedure which evolved during the nozzle jacket-forging weld program. No weld defects were found in the forward or aft joint at any of the three nondestructive testing (NDT) inspection levels: root, mid-point, and final. Final inspection of the welds included visual, penetrant, radiographic, and ultrasonic methods.

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Figure 16

Specimen Section from Heat 260-5-4001 (Tested at -320°F . Fracture surface at thickness center shows both transgranular and intergranular modes. Etchant: Schartz. Enlarged 100 times.)

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Figure 17

Specimen from Heat 260-5-4001 (Tested at room temperature. Etchant: Schartz. Enlarged 100 times. Intergranular terminal fracture mode is shown. Cracks on bottom surface were closed except for very small ones near center.)

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Figure 18

Fracture Surface of Specimen from Heat 260-5-4001 (Tested at room temperature. A thin band shows - from center to right - where fracture was mainly transgranular. Unetched. Enlarged eight times.)

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Figure 19

Fracture Surface of Specimen from Heat 260-5-4000 (Tested at room temperature. A transgranular band - near center - shows where initial failure occurred. Unetched. Enlarged eight times.)

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Figure 20

Fracture Surface of Specimen from Heat 260-5-4001 (Tested at -320°F . No discernible difference is shown in the mode of fracture. Unetched. Enlarged eight times.)

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Figure 21

Fracture Specimen from Heat 260-5-4001 (Tested at -320°F . Reduction of area not exhibited at fracture. Distinct lines due to machining were not present on bending surface which had a roughness of eight microinches. Unetched. Slightly reduced.)

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2. Torus

Welding, NDT inspection, and hydrotesting of the fuel and bolt-coolant tori-to-nozzle jacket welds were successfully completed on Nozzles S/N 880600 and 880603. Torus welding on Nozzle S/N 880601 has been completed and accepted by NDT inspection. This nozzle is presently being machined prior to hydrotesting. Figure 22 shows Nozzle S/N 880600 mounted on the Aronson positioner with the welding boom in place to make a weld pass on the fuel torus.

A weld-cracking problem was encountered in making the adapter-to-torus segment weld. This weld serves the dual function of joining the adapter to the torus segment and of providing a reinforcement "collar" between the torus and adapter. To accomplish the latter objective, multiple weld-passes are required to build up the "collar" (see Figure 23). The weld passes are manually deposited with a GTAW torch. The massive build-up of weld metal and accompanying thermal forces cause shrinkage cracks in the weld. The vendor experienced difficulty in making sound, manual repair-welds. The weld procedure was reviewed at the vendor's plant by an AGC weld-engineer, and controls established to: (1) minimize the welding heat-input; and (2) improve the interpass cleaning procedures. Manual weld-repair procedures (established by AGC Specification 90067) which were utilized in making weld repairs on the nozzle forging weldments, were instituted at the vendor facility and have been successful in easing repair difficulties.

3. Coolant Tube End Caps to Nozzle Welds

Extensive cracking occurred in the forward-end cap-to-jacket weld in Nozzle S/N 880600, the first nozzle to be processed through the furnace-braze cycle. This is a "V-joint" weld requiring a minimum of three passes to join the end caps to the nozzle jacket. The open joint-line of the adjacent end-caps intersects the circumferential weld, creating a notch

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Figure 22

Aronson Positioner with Nozzle S/N 880600

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Figure 23

Coolant Inlet Adapter Weldment

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condition conducive to weld cracking. Pyromet End Cap Weld Procedure No. 128 was revised to include a weld bead along the joint-line of the adjacent end-caps prior to the deposit of the initial weld-pass joining the end caps to the jacket (see Figure 24). This procedure sealed the "notch", and was effective in preventing weld cracking when used for the first time on Nozzle S/N 880601.

Excessive leakage between adjacent aft end-caps was encountered on Nozzle S/N 880603. This was caused by braze alloy run-off during the furnace-braze cycle. The leaks were successfully repaired at AGC (under the requirements of AGC Specification 90116), using a GTAW torch and Nicro braze-alloy. To prevent recurrence on future nozzles, a weld bead was placed between aft end-caps, providing a dam to stop braze alloy run-off. This procedure, incorporated in Pyromet End-Cap Weld Procedure No. 128, has been successfully used on Nozzles S/N 880601 and 880604. Figure 25 shows the completed aft end-cap welds.

4. Tube Repair

During the manual torch-braze repair of tube-fillet leaks, two tubes in Nozzle S/N 880600 developed transverse cracks. These were the direct result of the braze repair of the joint between tubes 33 and 34. The repair of the cracked tubes was accomplished at AGC, following a procedure evolved from extensive experimentation in making simulated repairs in full-scale nozzle sectors (Ref. Letter, 26 October 1966, J. J. Beereboom to K. E. Estes, SNPO-C, Subject: Phoebus Nozzle Tube Repair). The area of the repair, approximately two inches aft of the nozzle throat, limited accessibility and made the repair difficult to accomplish. Repair was made with a small TIG torch and Hastelloy X weld-wire. The ends of the repair weld were sealed at the tube braze-fillet using a Water Welder (see Figure 26) and BT braze alloy. A subsequent nitrogen leak check at 100 psi revealed a small leak at the weld-braze intersection between the tubes. The leak was repaired with the Water Welder torch and BT alloy. A nitrogen leak check at 100 psi and a water leak check at 1450 psig revealed no leakage in the repaired area.

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Figure 24

Forward End-Cap Welds Showing
Weld-Bead Along the Joint Line

Figure 25

Completed Aft End-Cap Welds

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Figure 26

Water Welder Oxy-Hydrogen Gas Generator

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(U) The Water Welder unit, which generates a hydrogen-oxygen flame, is equipped with a small hand-torch using specially prepared hypodermic needle tips with orifice sizes ranging in diameter from 0.003 to 0.042 inch. There is a minimum of heat dissipation to surrounding areas because of the small flame size and pin-point concentration of heat. This unit has been employed for all subsequent Phoebus nozzle braze-leak repairs.

(C) During hydroproof testing of Nozzle S/N 880603, the common leg of two coolant tubes partially lifted from the groove. A procedure was formulated to make a simulated repair on a full-scale brazed tube-sector. The procedure consisted of removal of the damaged coolant-tube section and regrooving by the electrical discharge machining (EDM) process. A two-tube coolant section, matching the contour of the damaged tube-area, was assembled and furnace brazed. EDM cut-outs of the damaged two-tube section were made as shown in Figure 27. Matching cut-outs were made in the brazed, replacement coolant-tube sections. The center-tube leg section (see Figure 28) was placed in the groove and welded in place with beads one-inch in length placed in a staggered sequence on either side of the leg (see Figure 29). The tube crown-caps were then fitted into place and tack-welded (see Figure 30). Upon completion of welding, the welds were blended and BT braze-alloy deposited in the tube-to-tube trough the full length of the joint (see Figure 31). The oxy-hydrogen flame generator torch was used for this operation following the requirements of AGC Specification 90116. The sector was leak checked with air at 100 psi using Leak Tec solution. No leakage was noted in the repaired section. Subsequently, the simulated repair sector successfully passed hydroproof testing at 1450 psig and was then subjected to a burst test which resulted in a leak at 3500 psi and discontinuance of the test. The repair of the damaged tube section in the nozzle followed the same procedure used on the full-scale sector. Upon completion of the repair, a proof test with nitrogen at 200 psi and a leak check at 100 psi with Leak Tec solution were successfully completed. Subsequent hydrotesting at 1450 psi was accomplished with no leakage detected.

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Figure 27

EDM Cut-Outs in Repair Specimen (U)

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Figure 28

Replacement Coolant-Tube and Center-Leg Sections (U)

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Figure 29

Center-Leg Section in Repair Specimen (U)

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Figure 30

Tube Crowns Tack Welded in Repair Specimen

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Figure 31
Repair Specimen Welding Completed,
Prior to Weld Blending

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In the process of investigating repair procedures for the damaged coolant tubes, a new welding machine process was evaluated. Figure 32 shows the unit that was used. The process offers exceptional promise as a method to weld and braze thin gauge materials because of the precise control obtainable and the small "flame".

5. Coolant Hole Repair

One of the 1/2-inch-diameter forward coolant exit-holes in Nozzle S/N 880605 was improperly drilled by the vendor. A repair procedure was devised to weld-fill the hole. One-half-inch diameter holes (with spacing and edge-distance dimensions matching the nozzle holes) were drilled in a Hastelloy X plate one-inch thick. The simulated hole was weld-filled with a GTAW torch and Hastelloy N weld wire. Radiography and penetrant inspection revealed the repair to be free of defects. The repair procedure was used successfully to weld-fill the improperly drilled hole in the nozzle.

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Figure 32

Plasma Needle Arc Welder

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C. BRAZING DEVELOPMENT

At the time of publication of the previous materials report it was assumed that the brazing development program was complete. However, subsequent results in the brazing of the first nozzle, S/N 880600, revealed some difficulties.

1. Nozzle S/N 880600

Phoebus Nozzle S/N 880600 was assembled with nickel plated components and brazed in accordance with Specification AGC 90006 (the publication number was later changed to AGC 90169). At the completion of the brazing operation, leak testing with gaseous nitrogen (at 50 psig) and acetone was conducted. After checking 92 channels, a total of 203 leaks was recorded. A decision was then made to braze an extra cycle to reduce both the leakage and the number of manual braze-repairs that would be needed.

The nozzle was loaded again with braze alloy and brazed in the same manner as the previous cycle except that the temperature was maintained 25 to 50°F lower. Following brazing, the nozzle was leak checked with gaseous nitrogen and acetone which revealed 189 leaks. However, the leak testing was conducted without the benefit of the leveling fixture and this, coupled with the low surface-tension of the reagent grade acetone, resulted in an insensitivity to "tiny" leaks. During manual braze-repair the total number of leaks recorded was 552. The repair was conducted in the nozzle leveling fixture and water used as the leak detection fluid. Each joint was tested with water, leaks marked, then repaired, rechecked and repaired again as necessary. The use of reagent grade acetone for preliminary leak checking was based upon minimum contamination and ready volatility for subsequent braze-repair (i.e., either another furnace cycle or by manual means).

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(U) The number of leaks encountered after the extra braze-cycle, although greatly reduced, was considered excessive and exceeded the extrapolation made from full-length sector No. 2. The sector, which contained two test joints with nickel-plated components, had only one leak after the standard brazing cycle, compared with 63 leaks for five joints with unplated components in Sector No. 1.

(U) The decision to nickel-plate the nozzle components was based upon results with the two full-length sectors, the established presence of aluminum in the Hastelloy X, and the formation of aluminum oxide on critical sealing-surfaces. The nickel plating (conducted in accordance with Specification AGC 90112) consists of a nickel chloride-hydrochloric acid-strike followed by plating to the desired thickness in a Watts bath.

(C) The tube plating-thickness of sector No. 2 ranged from 0.00047 to 0.00075 inch, with an average of 0.00061 inch. Spacer plating thickness varied from 0.00027 to 0.00051 inch, averaging 0.00036 inch. Plating-thickness requirements for Nozzle S/N 880600 tubes and spacers were held to 0.0002 to 0.0004 inch and 0.0001 to 0.0004-inch, respectively, due to the accumulation of jacket-groove tolerances not permitting achievement of the same thickness as used on sector No. 2. Heavier plating would have required elimination of the inner braze-foil preform.

(U) The nature of the porosity, the appearance of the braze joints, and the tube surfaces all indicated that the plating was marginal in its basic function of providing a barrier to aluminum oxide formation along the seal-joint surfaces. Heavier plating, as obtained on sector No. 2, was necessary but tolerance accumulation precluded this.

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2. Test Program

(U) The test program evolved at AGC consisted of the following:

(C) (a) A full-scale test sector was to be brazed with nickel-plated components (the same as sector No. 2) and with the inner braze-foil preforms removed. The test sector was to be leak and hydrotested until it burst. The sector was to be brazed in the nozzle furnace with the following facility changes:

(U) (1) The pre-purge vacuum pump was to be installed on the furnace exhaust-line (the pump was originally on the inlet line).

(U) (2) The exhaust-stand pipe was to be modified to prevent possible back-streaming of water vapor formed by the burning of effluent gas.

(U) (3) The Alnor dew-point indicator was to be installed on the exhaust to monitor exit-gas quality.

(U) (b) The effect of the extra braze-cycle on the tensile properties of the jacket material was to be determined at room temperature and at -320°F.

(U) (c) The shear strength of the braze joint was to be determined at ORNL using Miller-Peaslee specimens nickel-plated to thicknesses of 0.0002, 0.0005 and 0.0008 inch. Four specimens at each thickness were to be tested at room temperature.

(U) (d) Five round (butt) tensiles were to be tested at -320°F and room temperature to evaluate the joint sandwich-strength with nickel-plating thicknesses of 0.0002, 0.0005 and 0.0008 inch. Parent-metal control specimens were also to be tested.

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(U) (e) A tubular thermal-cycle fatigue specimen was to be fabricated with simulated manual braze-repairs for testing at ORNL.

(U) (f) Three "Tee" specimens of Allvac 625 strip with 0.08 to 0.10% aluminum were to be brazed along with the full-scale test sector.

3. Plating

(U) Plating of components for Nozzle S/N 880600 and the requirement for heavier plating revealed a need for better measurement and understanding of the nickel-plating thicknesses. Extensive tests were conducted to correlate nondestructive plating measurements with metallographic and micrometer measurements. The Magnagage is normally acceptable for plating measurement (in this thickness range) on materials plated upon one surface only and on materials thicker than 0.060 inch.

(C) The Phoebus coolant-channels and spacers require nickel plating on Hastelloy X material of 0.012-inch and 0.008-inch thickness, respectively; therefore, acceptance data had to be established for the Phoebus program. The edges and corners of the channels and spacers also accumulated heavy plating build-up. This necessitated "robbers" to reduce this condition.

(C) The robber employed on the coolant channel, which is shown in Figure 33, resulted in elimination of plating on the inside of the channel. This allowed micrometer measurement to be used to determine plating thickness. Plating on the coolant channel interior-dimension was of no value in subsequent brazing operations. Several specimens had to be plated to determine the A and B dimensions of the robber along the longitudinal axis. The Magnagage method of plating-thickness measurement was selected and the established limits were:

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Figure 33

Robbers and Spacers used in Nozzle Component Plating (U)

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(C) The corners and edges of the spacers presented the same problem as the channels: i.e., they, too, were subject to heavy plating build-up. Holding racks, as shown in Figure 33, were devised which allowed 12 spacers to be plated simultaneously. However, test specimens indicated inconsistencies in the Magnagage plating-thickness measurements because the gage would partially read both plated sides. Specimens were then measured for thickness and plated on one side only and the Magnagage readings were checked against super-micrometer measurements. A formula was devised which compared Magnagage readings to those for micro-specimens, based on statistical averages and evaluated against simulated production procedures. The established limits were:

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4. Test Program Results

a. Full-Scale Test Sector

(C) A full-scale test sector (No. 3) was brazed with nickel-plated tubes and spacers to the thickness limits indicated above. To compensate for the added plating thickness, the inner braze-foil preforms were removed. The sector was brazed in the full-scale nozzle furnace with the prescribed changes: i.e., the prepurge vacuum-pump in the exhaust line, the modified exhaust-stand pipe, and the Alnor dew-point indicator in the exhaust gas line.

(U) At the completion of the standard braze-cycle, the sector was leak tested both with nitrogen at 50 psig and with acetone and found to contain one tube-to-tube and three aft end-cap leaks. All leaks were repaired at AGC. The sector was then leak tested with nitrogen at 100 psig. No leakage was discovered and the sector was then pressurized to 5400 psig, at which time an end tube-wall burst in the transition radius between the inlet and the convergent section (see Figure 34).

b. Effect of Extra Brazing-Cycle

(U) The effect of the extra braze-cycle on the tensile properties of Hastelloy X were first analyzed empirically. The opinion obtained was that a very small effect would result but that elongation would probably present the most notable change (decrease). To quantitatively evaluate the effect on the jacket material, samples of Heat No. 260-5-2813 were exposed to the standard braze-cycle and to the standard cycle plus an extra braze-cycle. The samples were obtained from the previously brazed first and second sectors, respectively. The tensile test results, at room temperature and -320°F, are presented in Table 1, which also includes the solution annealed properties (not brazed) of this heat.

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Figure 34

Burst Tube in Full-Scale Test Sector

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TABLE 1

Effect of Brazing Cycles on Hastelloy X
Tensile Properties

(Heat 260-5-2813)

Annealed, AGC 90056

<u>Temp (°F)</u>	<u>UTS(ksi)</u>	<u>YS(ksi)</u>	<u>ELONG (%)</u>	
Room	104.7	45.0	56	
	105.0	45.5	58	
	(99.9)	(43.2)	(41)	(Annealed Forgings) Average
-320	-	-	-	
	(136.0)	(77.8)	(27)	(Annealed Forgings) Average

Standard Braze

<u>Temp (°F)</u>	<u>UTS(ksi)</u>	<u>YS(ksi)</u>	<u>ELONG (%)</u>	
Room	108.0	48.5	22	
Room	108.0	48.7	23	
Room	(93.3) ^{+ 11*}	(45.0) ^{+ 6.6*}	(24) ^{+ 9*}	(Design Average)
-320	139.0	80.9	17	
-320	138.0	84.2	13	
-320	(117.4) ^{+ 11*}	(71.7) ^{+ 6.6*}	(13) ^{+ 5.6}	(Design Average)

Standard Braze + Extra Braze Cycle

<u>Temp (°F)</u>	<u>UTS(ksi)</u>	<u>YS(ksi)</u>	<u>ELONG (%)</u>	
Room	106.0	47.6	21	
	107.0	47.2	21	
-320	130.0	81.7	11	
-320	131.0	81.9	11	

*3 Sigma

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Specifically, the results reveal a slight-to-negligible reduction in UTS and elongation but no change in YS at room temperature. However, a greater change is evident in UTS and elongation at -320°F , with but very slight difference in YS. The three-sigma variation in elongation at -320°F was found to be minus 5.6% in the design properties task. Application of the minus three-sigma value to the average of 11% elongation obtained at -320°F results in a design minimum of 5.4%, an acceptable value.

c. Shear Strength of Plated Braze Joint

Eighteen Miller-Peaslee braze-joint specimens (for shear testing at room temperature and -320°F), were Niore-brazed with faying surfaces nickel-plated to thicknesses of 0.0002, 0.0005, and 0.0008/0.0009 inch. The specimens were tested by ORNL and the reported results are shown in Table 2.

Summarized, the room-temperature results reveal almost identical average strengths regardless of thickness of the nickel plate, as well as very similar minimum/maximum values and spread. Combining room temperature data results in an average shear-strength of 60.5 ksi, with a standard deviation of 3.26 ksi. The minus-3 standard deviation shear-strength is 50.7 ksi at room temperature. The limited number of tests at -320°F is not capable of producing data of equal confidence with room temperature values; however, the average shear-strength at -320°F is 81.3 ksi, and the standard deviation is 2.06 ksi, providing a minus-3 standard deviation shear-strength value of 75.1 ksi.

The tensile properties also were reported by ORNL for several braze-alloys as follows:

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<u>Alloy</u>	<u>Test Temp. (°F)</u>	<u>UTS (ksi)</u>	<u>YS (ksi)</u>
Nicoro 80	68	56.5	44.8
Nioro	68	68.0	38.4
Palniro 7	68	62.8	39.7
Nicoro 80	750	34.9	32.5
Nioro	750	53.4	31.5
Palniro 7	750	53.8	31.4

(C) These data reveal a very high ratio of shear-strength to ultimate tensile-strength (about 0.89). The most recent hydrotest failure qualitatively confirms the reliability of the foregoing data. During proof stress of Nozzle S/N 880603, a tube-joint sheared from the groove at about 1050 psi. Examination of the tube leg revealed only a partial braze-joint at the center of the area. The area was estimated for the central one-inch length of the groove, the load per lineal inch was calculated, and the stress estimated to be 57.0 ksi.

d. Tensile Strength of Plated Braze Joint

(U) The tensile strength across the brazed joint was measured at room temperature and at -320°F. Standard R-3 specimens, with the braze-joint composite construction located in the center of the gage, were tested with nickel plating of 0.0002-, 0.0005- and 0.0008-inch thickness.

(U) The tensile test-results are presented in Table 3. Included also are the parent-metal results as control values. The results indicate: (1) some decrease in scatter as plating thickness increases; and (2) a decrease in the average strength. The 89.4 ksi, 97.6 ksi, and 77.6 ksi average tensile-strengths at room temperature for the 0.0002, 0.0005, and 0.0008 inch plating thicknesses compare with the 94.9 ksi average for previous tests without plating (see NRO publication RP-DR-0004).

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TABLE 2

Miller-Peaslee Shear Tests of
Nickel Plated and Braze Hastelloy X

<u>Test Temp. (°F)</u>	<u>Plating Thickness (in.)</u>	<u>Shear Strength (ksi)</u>	<u>Spec. No.</u>	<u>Comments</u>
-320	0.0002	78.9	04	-
-320	0.0005	82.8	08	BBM*
-320	0.0008/9	82.1	12	-
-320	Overall Average	81.3	-	-
Room	0.0002	60.2	01	BBM
Room	0.0002	60.1	02	
Room	0.0002	57.4	03	
Room	0.0002	59.5	13	
Room	0.0002	62.6	14	
Room	0.0002	60.0	Average	
Room	0.0005	52.3	05	
Room	0.0005	62.9	06	
Room	0.0005	62.7	07	
Room	0.0005	62.0	15	
Room	0.0005	63.8	16	
Room	0.0005	60.6	Average	
Room	0.0008 to 0.0009	63.0	09	
Room	0.0008 to 0.0009	66.1	10	BBM
Room	0.0008 to 0.0009	59.5	11	BBM
Room	0.0008 to 0.0009	57.1	17	
Room	0.0008 to 0.0009	59.1	18	
Room	0.0008 to 0.0009	60.9	Average	
Room	All	60.5	Overall Average	

*BBM = Broke in Base Metal

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TABLE 3

Tensile Test Results for 0.250-inch
Round Hastelloy X Bar Stock Specimens
With Various Nickel Plate Thicknesses

Exposure: Standard braze cycle

Specimen: Drawing 1118593

Material Heat No: 260-5-2783, 3/4-inch diameter bar

Test Temp(°F)	Spec. Ident.	Plating Thick(in)	UTS (ksi)	Spec. Ident.	Plating Thick(in)	UTS (ksi)	Spec. Ident.	Plating Thick(in)	UTS (ksi)
Room	1	0.0002	104.0	11	0.0005	101.0	21	0.0008	86.2
	2	0.0002	65.1	12	0.0005	83.2	22	0.0008	73.6
	3	0.0002	88.0	13	0.0005	99.7	23	0.0008	68.6
	4	0.0002	106.0	14	0.0005	102.0	24	0.0008	93.7
	5	0.0002	83.8	15	0.0005	102.0	25	0.0008	65.7
Average		0.0002	89.4			97.6			77.6
-320	6	0.0002	131.0	16	0.0005	130.0	26	0.0008	124.0
	7	0.0002	99.4	17	0.0005	136.0	27	0.0008	117.0
	8	0.0002	133.0	18	0.0005	100.0	28	0.0008	116.0
	9	0.0002	135.0	19	0.0005	134.0	29	0.0008	110.0
	10	0.0002	132.0	20	0.0005	137.0	30	0.0008	135.0
Average		0.0002	126.1		0.0005	127.4		0.0008	120.4

Parent Metal Control Specimens

Spec. Ident.	Test Temp(°F)	UTS (ksi)	0.2% YS (ksi)	Elong (%)
31	Room	106.0	44.1	38
32	Room	106.0	44.1	39
33	Room	106.0	44.4	37
Average	Room	106.0	44.2	38
34	-320	139.0	71.6	26
35	-320	140.0	75.4	20.5
36	-320	141.0	72.1	23.5
Average	-320	140.0	73.0	22

e. Tubular Thermal Fatigue Specimens

A single thermal-fatigue specimen (consisting of five tubes brazed to a flat plate) was sent to ORNL for testing. The specimen contained numerous simulated hand braze-joint repairs. The test results were to be compared with previous specimens tested at a maximum tube-crown temperature of 1800°F. The previous testing also included test results at 1600°F and 1900°F. A report on the results (Thermal Fatigue Analysis of a Cryogenically Cooled Rocket Nozzle by A. E. Carden, D. G. Harman and E. A. Franco-Ferreira) was presented at the Southwestern Symposium in Missiles and Aerospace Vehicles Sciences, American Astronautical Society, Huntsville, Alabama, December 5-7, 1966. The major test results are shown in Table 4.

The specimen containing the manual braze-joint repairs was tested by heating to 1800°F and holding at that temperature for five minutes with liquid nitrogen flowing through the tube at essentially zero pressure. One cycle consisted of a five-second heat-up, a five-minute hold (at 1800°F) and a five-second cool-down with flowing liquid nitrogen. From the limited test results, it appears that a weld repair has no worse effect than a tube dent and that braze repairs, per se, have no effect on the cyclic life. The results are shown in Table 5 with summarized results from Table 4 for smooth and dented tubes (but without braze repairs).

f. Allvac 625 "Tee" Specimens

Three "Tee" specimens of Allvac 625 strip, 0.012-inch in thickness, from Heat 5940 (containing 0.08 to 0.09% aluminum) were brazed with the full-scale test sector. The results were not discriminatory: that is, the braze-alloy flow and appearance of the specimens were similar to the unplated, back-up sector plate from the Hastelloy X Heat No. 260-5-2813 that contained 0.24% aluminum. This confirms the results of evaluating samples of Hastelloy X with a wide-range of aluminum contents. The sheet samples (provided by the UCC Stellite Division) were brazed through the Phoebus cycle. The results were:

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<u>Heat No.</u>	<u>Al (%)</u>	<u>Visual Appearance</u>
260-6-4568	0.42	Grey, non-bright
260-6-4589	0.27	Grey, non-bright
260-6-4765	0.06	Noticeably lighter grey, brighter

It is felt that the Allvac "Tee" specimens and the evaluation of the Hastelloy X sheet samples present satisfactory evidence that an aluminum content of 0.02% is a requirement to be reasonably certain of good braze-flow and wetting in a Phoebus-type braze cycle.

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TABLE 4

Thermal Tests* of Flat Tubular Hastelloy X** Specimens

Specimen No.	Temp. Max (°F)	Temp. Min (°F)	T Max Hold Time (min)	Tube Condition	Number of Thermal Cycles to Failure		
					Visible Wrinkling	First Visible Crack	Crack Through Wall
X-1	1800	-200	5	Smooth	14	41	50
X-2	1800	-200	15	Smooth	30	70	80
X-3	1900	-200	5	Smooth	11	32	42
				20-mil dent ^a		18	22
				20-mil dent ^a		21	26
X-4	1800	-200	5	Smooth	14	49	57
				Small dent ^b		25	33
				Med. dent ^b		39	49
				Large dent ^b		56	NF ^c
				20-mil dent ^a		19	28
				20-mil dent ^a		22	30
				20-mil dent ^a		25	33
				10-mil dent ^a		25	35
				5-mil dent ^a		NF ^c	NF ^c
X-5	1600	-200	5	Smooth	15	75	79
				20-mil dent ^a		23	35
				20-mil dent ^a		25	38

Notes: ^aDepth of controlled dent made in top of tube crown with a 1/8-inch R indenter.

^bDescription of dents in "as-received" specimen.

^cNo failure.

^dTube-crown heating time from -200°F to T max was approximately 5 sec/cycle.

*Tested at ORNL

**Tube-material Heat No. 260-5-2813

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TABLE 5

Thermal Fatigue Tests of Flat Tubular Specimens
of Hastelloy X (Heat No. 260-6-4002)

<u>Tube Condition</u>	<u>Cycles to Failure</u>	<u>Comments</u>
Braze repair	12	Failed at pin hole in tube wall between tubes, adjacent to braze.
Braze repair	25	Failed at weld repair of a pin hole similar to first specimen.
Braze repair	47	Smooth tube failure.
No repair	50 to 57	Smooth tube failure (see Table 4)
No repair but dented	28 to 49	Failure in dents (see Table 4)

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D. MATERIAL PROCUREMENT

All Hastelloy X and INOR-8 materials were procured and previously discussed in NRO publications RP-DR-0003 (March 1966) and RP-QR-0004 (June 1966). The completion of the outstanding Allvac 625 material is discussed below.

Because of repeated difficulties in producing defect-free weld wire, the vendor requested cancellation at no charge to AGC. Verbal concurrence was received from SNPO-C and the weld-wire requirement on Purchase Order 201270 was cancelled. Delivery of the remaining Allvac 625 material was completed in October 1966. Table 6 itemizes the sizes and amounts of material procured, which is now stored at AGC, with the exception of the following items transferred to the NERVA program, Contract SNP-1, at SNPO-C direction:

Table 6 <u>Item No.</u>	<u>Form</u>	<u>Quantity</u>	<u>Heat No.</u>
2.	Plate	1	5942
4.	Strip	180-in.	59

E. NOZZLE FORGING PROPERTIES

The tensile properties, measured on specimens from the various full-scale nozzle forgings, were reported previously in NRO publication RP-DR-0004 for the annealed condition and the annealed and brazed condition. Although the previous results were considered essentially complete, some additional annealed test-data have been obtained. Revised and current results are presented in Table 7, which lists the average tensile properties by forging number and by heat number. The heat-number tabulation also indicates the minimum and the maximum values recorded.

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TABLE 6

Phoebus-2 Material Development
Procurement of Allvac 625

<u>Item</u>	<u>Form</u>	<u>Size (in.)</u>	<u>Quantity</u>	<u>Heat No.</u>	<u>P.O.</u>
1	Bar	0.750 dia	150 ft.	5940	201270
2	Plate	1-1/2 x 12 x 36	9	5940, 5941, & 5942	201270
3	Sheet	0.050 x 12 x 48	1	5940	201270
4	Strip	0.012 x 8 x 360	3	5940, 5941, & 5942	201270
5	Plate	0.187 x 2 x 60	1	5940	201878
6	Strip	0.012 x 2 x 60	9	5941	201878
7	Strip	0.008 x 3 x 60	1	5942	201878

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TABLE 7

Summary of Average Tensile Properties of Annealed* Nozzle Forgings
of Hastelloy X (AGC 90056)
by Forging Number and Material Heat Number

<u>Forging, Number</u>							
<u>No.</u>	<u>Name</u>	<u>Temp(°F)</u>	<u>UTS(ksi)</u>	<u>0.2% YS(ksi)</u>	<u>EL(%)**</u>	<u>No. of Forgings</u>	<u>No. of Heats</u>
1114856-13	Inlet	Room	104.3	44.3	42	7	3
1114856-7***	Converg.	Room	102.0	43.1	48	7	6
1114856-11	Diverg.	Room	93.2	42.3	33	6	4
Average	All	Room	100.8	43.3	42		
1114856-13	Inlet	1200	70.5	26.3	48	7	3
1114856-7***	Converg.	1200	72.2	27.5	46	7	6
1114856-11	Diverg.	1200	69.2	26.7	36	6	4
Average	All	1200	71.2	27.0	45		

Material Heat Number

<u>Heat No.</u>	<u>Temp(°F)</u>	<u>UTS(ksi)</u>	<u>YS(ksi)</u>	<u>EL%</u>	<u>No. of Forgings</u>
260-5-2793	Room	95.8	41.5	49	4
260-5-2820	Room	100.5	42.7	52	1
260-5-2850	Room	86.3	40.3	28	2
260-5-2863	Room	101.7	42.2	45	1
260-6-2880	Room	104.5	42.6	52	2
260-6-2887	Room	101.4	44.7	34	2
260-6-2898	Room	98.5	43.8	38	2
260-6-2900	Room	104.5	45.4	41	1
260-6-2901	Room	109.3	45.7	39	3
260-6-2920	Room	96.1	42.1	41	1
260-6-2935	Room	103.7	45.0	32	1
Average	Room	100.8	43.3	42	
Minimum	Room	80.3	39.2	22	
Maximum	Room	112.8	52.0	58	

Notes: *One (1) hour at 2150°F and fan cooled
**All specimens R-3 with 1.0-inch gage length
***Includes P/N 1114856-15

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TABLE 7 (cont.)

<u>Heat No.</u>	<u>Temp(°F)</u>	<u>UTS(ksi)</u>	<u>YS(ksi)</u>	<u>EL%</u>	<u>No. of Forgings</u>
260-5-2793	1200	66.0	26.5	53	
260-5-2820	1200	75.4	26.2	51	
260-5-2850	1200	63.8	24.9	28	
260-5-2863	1200	73.5	27.8	45	
260-6-2880	1200	76.3	26.0	50	
260-6-2887	1200	75.9	28.5	42	
260-6-2898	1200	71.3	27.9	38	
260-6-2900	1200	75.3	29.4	37	
260-6-2901	1200	78.5	28.8	40	
260-6-2920	1200	72.3	26.4	47	
260-6-2935	1200	-	-	-	
Average	1200	71.2	27.0	45	
Minimum	1200	61.5	24.2	24	
Maximum	1200	82.9	33.8	56	

1. Strain rate 0.005-in/in/min to yield then 0.05-in/in/min to failure
2. Specimens oriented tangentially, unless otherwise stated, in rings sectioned from full scale forgings.