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REPORT NO. RP-DR-0004

TO

SPACE NUCLEAR PROPULSION OFFICE - CLEVELAND EXTENSION

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

SEPTEMBER 1966

CONTRACT SNPC-35

44-611

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REPORT NO. RP-DR-0004

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

ROCKET ENGINE OPERATIONS - NUCLEAR

SEPTEMBER 1966

CONTRACT SNPC-35

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FOREWORD

This materials report (for the period 15 February 1966 through 15 August 1966) 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 "

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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 1966 through 15 August 1966. It documents and summarizes the results of the materials activities performed under the development phases of the contract in the specific areas of properties determinations, welding, brazing, special testing and material and component procurement support.

With the exception of a few remaining special properties tests (biaxial specimens), the properties program is completed, and a summary design data compilation is being prepared. Likewise, with the completion of testing of the remaining braze sector, the brazing development program is completed. The welding development activity has also been completed, and the effort presented hereafter will only describe the full-scale nozzle welding support. The procurement of full-scale ingot and forging components is completed also; however, the completion of the procurement of the development materials is pending receipt of the final Allvac 625 strip and wire.

The presentation in the Detailed Discussion is divided into the following six sections: Properties Program, Welding Development, Brazing Development, Heat Transfer Tubes - Thermal Cycling Tests, Coolant Tube Metallurgical Examination, and Material Procurement and Nozzle Procurement Support.

B. SUMMARY

All procurement of Hastelloy X for the Materials Development Programs was completed, as reported in the previous Materials Review Report No. RP-DR-0003. Subsequently, the INOR-8 procurement was completed with the cancellation of the strip and wire products by mutual agreement between AGC and SNPO-C. The Allvac 625 procurement will be completed by the middle of October 1966. Delays were incurred by the supplier caused by mill difficulties, annealing studies, and the weldability testing (now completed).

The design properties testing effort has been completed. Statistical analysis was applied to the tensile properties - notched and unnotched - for the forgings, weldments and strip stock of Hastelloy X. Additional mechanical properties test data obtained included shear strength, compressive strength and bearing strength. Physical properties measurements include tensile and shear module, coefficient of expansion and thermal conductivity. The data were conducted on three heats of material that were processed through a braze cycle simulating that of the actual nozzle.

The welding development activities were completed during this report period, including weld qualification testing of the third heat (260-5-2813) of forging material and testing and evaluation of the dissimilar metal (torus) weld joining. In addition, welding was completed on convectively cooled nozzle S/N's 880600, 880601, 880603, 880604 and 880605 as well as pressure test nozzle 880602. The weldability testing of Allvac 625 (in accordance with AGC-90059) was completed.

The braze-joint strength testing was completed, comparing parent metal values with the butt joint, simulated seal joint and surface diffusion effects of Nicro, Palnicro-7, Nicro 80 and copper braze alloys. In addition, a full-scale, contoured sector was brazed, revealing wetting and flow difficulties on the final cycle as a result of the aluminum content of the Hastelloy X. Following investigations with modified braze alloys, nickel plating and the introduction of a new

brazing alloy (Palcusil 25), a second sector was brazed demonstrating the benefits of nickel plating to evade the formation of aluminum oxide on the tube surfaces. Brazing of the second sector resulted in considerably fewer leaks than the first sector. During hydrotest of the first unit a burst occurred at 3700 psi because of inadequate braze joint assembly. The second unit was tested to 4300 psi without failure. Testing was discontinued on the second unit because of excessive leakage between the end tubes and the supporting side rails.

Thermal cycling tests were successfully completed on two tubes of Hastelloy X, demonstrating the material's ability to withstand twenty simulated coolant tube heating and cooling conditions.

A metallurgical examination of a coolant tube (produced by Straza Industries) was conducted revealing the fine grain structure of the tubes as well as: the stress equalizing and annealing effects of the braze cycle compared with a 1925°F annealing treatment, the slight increase in hardness resulting from the braze cycle or annealing cycle, the microstructural changes with thermal treatments, and the lack of a need to utilize a separate annealing cycle.

The procurement of all nozzle forgings was completed and the properties of the different forgings were compared and analyzed. The results of a successful effort to improve the strength of Hastelloy X forgings are presented, revealing a gain of 15% in yield strength at room temperature and 100% improvement in tensile ductility at -320°F.

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II. DETAILED DISCUSSION

A. PROPERTIES PROGRAM

The tensile tests of Phoebus Hastelloy X were performed in accordance with the test plan which was reported in the preceding Six Months Report, RP-DR-0003. The properties program was designed to obtain data representative of the Phoebus-2 Hastelloy X, vacuum melted nozzle material made to AGC-90056B (forging) and AGC-90057 (strip). All materials were given a simulated braze cycle treatment prior to testing.

The data obtained included unnotched and notched tensile properties of welded and unwelded ring forgings at temperatures from -423° to 1200°F , unnotched and notched tensile properties of channel strip (0.012-in.-thick) from -423° to 1800°F , shear ultimate from -320° to 400°F , compressive yield at room temperature, and bearing loads from -320° to 800°F . The physical properties determined included thermal conductivity (-320° to 930°F), mean coefficient of expansion (-400° to 1800°F) and tensile/shear moduli (-320° to 930°F).

1. Forging Test Results

The results of the tensile testing of the ring forgings from Heats 261-5-4000 and 261-5-4001 are presented in Tables 1 and 2, respectively. Examination of the results indicated that the ultimate tensile strength and ductility values in the tangential direction were consistently better than those from the axial direction. Direction did not significantly alter the yield strength values. The data from the Heat No. 261-5-4000 ring forging (Table 1) also showed a trend of higher strength and elongation values near the OD surface, and lower values near the ID of the ring. The properties of the ring from Heat No. 261-5-4001, (Table 2), were more uniform with respect to specimen location.

Additional data obtained from the tensile tests, which are not shown in Tables 1 and 2, include uniform elongation, modulus of elasticity, and stress-strain curves.

TABLE 1

TENSILE TEST DATA (UNNOTCHED) FROM HASTELLOY X FORGING,
HEAT NO. 261-5-4000 (COMPLETE RESULTS)

SPECIMEN ORIENTATION	SIZE	TEST TEMP. °F	UTS ksi	ID OF RING			%RA	UTS ksi	CENTER			%RA	UTS ksi	OD OF RING			%RA
				YS ksi	YS ksi	%EL			YS ksi	YS ksi	%EL			YS ksi	YS ksi	%EL	
Axial	R3	-423	115.0	85.0	3.5	4.2		114.0	87.2	5.0	5.2		125.8	85.2	8.0	10.0	
Tangential	R3	-423	130.8	91.4	9.0	11.2		136.0	87.6	12.0	11.2		132.0	87.8	12.0	11.3	
Axial	R3	-320	102	75.4	6.5	5.5		107.6	76.0	9.5	7.1		122.4	76.2	15	14.5	
								111.7	71.0	11	9		118.2	68.0	13	14.0	
Axial	R1	-320	97.7	69.6	5.5	5.0		108	69.5	9	7		110.3	69.0	9.5	8.9	
Tangential	R1	-320	118.5	69.5	14.0	12.0		119.3	69.1	14	12		120.5	68.7	14	12	
Tangential	R3	-320	123.0	76.4	15.0	14.7		132	86	16	12		133	71	18	15	
								123	76	17	13		131	78	17	15	
Short Transverse	R3	-320						106.7	67.2	7	8		117.6	69.9	11.5	11	
Short Transverse	R3	70						86.8	42.9	16.5	12.2		99.5	44	20	21	
Axial	R1	70						89.0	41.8	15	13.0						
Axial	R3	70						87.2	42.0	18	16						
								86.4	48.9	17.5	12.3						
Tangential	R1	70						99.1	42.7	26	22						
Tangential	R3	70						97.0	47.5	26.5	25.2						
Axial	R1	400						84.8	41.8	15	13						
Axial	R3	400						79.0	34.6	20	19						
Axial	R1	800						75.9	29.4	28	22		76.8	29.7	24	21	
Axial	R3	800	74.1	29.1	21	19		78.4	29.7	26	23		76.6	30.1	26	23	
Tangential	R1	800	82.2	30.2	41	31		81.0	29.6	32	29		86.8	30.0	35	30	
			82.4	29.4	40.5	31							81.6	28.7	40	30	
Tangential	R3	800	84.7	40.3	31.5	33.4		83.1	31	29	36						
Tangential	R3	800	83.2	32	32	35		83.3	-	37	35		84.9	38.4	33.5	30.7	
Axial	R1	1200	71.9	24.3	22	20		74.8	25.9	29	25		75.7	25.8	25	22	
Axial	R3	1200	69.5	29.4	20	20.4		-	-	-	-		77.8	32.8	32	26.7	
Tangential	R1	1200	76.6	26.2	33	28		77.7	26.6	37	32		80.5	27.2	37	35	

NOTE: 1. All specimens heat treated through three cycle simulated braze treatment.
2. Strain rate 0.005-in/in/min. to yield, then 0.05-in/in/min. to failure.
3. Material procured to AGC-90056, 23" O.D. x 17" I.D. x 6" long ring.

TABLE 2

TENSILE TEST DATA (UNNOTCHED) FROM HASTELLOY X FORGING,
HEAT NO. 261-5-4001 (COMPLETE RESULTS)

SPECIMEN ORIENTATION	SIZE	TEST TEMP. °F	UTS ksi	ID OF RING			UTS ksi	CENTER			UTS ksi	OD OF RING		
				YS ksi	%EL	%RA		YS ksi	%EL	%RA		YS ksi	%EL	%RA
Axial	R3	-423	111.4	88.4	5.0	8.0	117.1	87.4	7.0	10	111.5	83.5	5.0	6.5
Tangential	R3	-423	129.6	84.9	8.5	11.0	125.1	88.6	10.5	10	129.8	85.9	10.0	8.3
Axial	R1	-320	106.4	70.0	8.0	7.8	110.2	69.5	10.5	8.6	106.8	68.8	8.6	8.8
Axial	R3	-320	110.0	69.6	9.7	8.6	111.9	72.9	10.0	7.8	106.3	67.7	8.5	9.4
							120.0	74.5	16.4	12.4				
Tangential	R1	-320	117	69.0	13.6	12.3	121.8	71.8	16.7	12.7	124.8	67.0	19.0	16.0
Tangential	R3	-320	131.4	71.3	18.5	14.7	127.3	67.8	16.4	16.1	125.2	69.8	16.1	16.1
			125.9	72.4	16.8	12.6								
Axial	R3	70					84.4	46.5	15.0	11.0				
Tangential	R1	70					89.0	48.2	17.7	12.4				
Tangential	R3	70					97.5	41.9	30.2	25.3				
Tangential	R3	70					96.6	45.6	31.8	27.3				
Tangential	R3	70					95.7	43.0	38.4	26.7				
Tangential	R3	70					94.7	45.4	29.3	25.3	98.2	46.8	27.6	26.1
Axial	R1	400					76.2	32.3	18.0	16.5				
Axial	R3	400					76.8	34.2	24.9	16.8				
							77.6	32.6	20.8	16.1				
Tangential	R1	400	83.0	33.9	25.4	23	87.3	32.6	32.5	28.6				
Tangential	R3	400					86.4	33.8	30.0	31.6				
							84.5	36.2	29.1	27.5				
Axial	R1	800	72.8	28.8	21.2	24.2	79.1	29.0	32.0	25.2	79.9	29.2	35.3	30.3
Axial	R3	800	75.7	33.1	26.4	24.0	75.6	33.6	25.9	21.2	76.5	36.0	23.2	23.2
Tangential	R1	800	82.1	29.0	41.5	37.9	83.0	29.3	37.2	34.8	81.8	29.3	39.2	33.6
Tangential	R1	800	79.8	28.6	42.2	38.6	83.5	30.2	36.5	33.3	79.8	28.5	41.5	35.8
Tangential	R3	800	81.0	32.8	37.8	37.3	81	30.4	36.6	37.3	81.0	29.0	36.0	33.3
Tangential	R3	800	80.3	34.1	35.8	32.8	79	29	36.8	36.6	83.9	32.2	34.8	34.0
Tangential	R3	800					81	32	36.3	35.4				
Axial	R1	1200	67.3	24.2	21.2	21.4	72.8	25.1	29.5	26.7	75.0	24.9	36.8	27.0
	R3	1200	71.5	25.0	24.1	23.0	71.5	29.7	32.7	28.1	74.6	27.7	26.8	24.6
Tangential	R1	1200	75.5	25.1	37.4	34.6	76.6	25.0	39.4	35	77.5	26.0	39.8	34.6
	R3	1200					75.8	27.9	35.4	37.9	77.6	29.9	38.7	37.2

NOTE:
1. All specimens heat treated through three cycle simulated braze treatment.
2. Strain rate 0.005-in/in/min. to yield, then 0.05-in/in/min. to failure.
3. Material procured to AGC-90056, 23" O.D. x 17" I.D. x 6" long ring.

With the exception of the high temperature tests, the uniform elongation values were essentially the same as the total elongation. At 1200°F, the tensiles which exhibited greater than 33% total elongation did show some difference in the uniform elongation values; namely, the specimens which had 37 to 40% total elongation had a uniform elongation of 33 to 36%, while the specimens with 21 to 33% total elongation had equivalent 21 to 33% uniform elongation. The uniform elongation at 800°F was approximately the same; that is, 33 to 35% with the total elongation 2 to 3% higher. However, it was as much as 7% points higher in the R-1 specimens, showing 41 to 42% total elongation.

The modulus of elasticity data will be covered in the discussion of the physical properties. The stress-strain curves were obtained for all specimens; however, instrumentation limits made it necessary to limit the curves made at high strain magnification to a few percent strain to get good, reliable yield data. A representative group of specimens were tested at low strain magnification to obtain the complete tensile curves to fracture. Full length representative tensile curves at -320°F, room temperature, and 1200°F are shown in Figure 1. Included also is a tensile curve at 1600°F for the 0.012-in. thick strip to show the difference in uniform and total elongation at temperatures higher than that at which the forgings were tested.

Notched tensile specimens, with a stress concentration factor of $K_t = 6.3$, were also evaluated. The results demonstrated conclusively that a significant notch sensitivity problem does not exist with Hastelloy X after braze cycles treatment. The low temperature notch-to-unnotched ratios ranged from 0.98 to 1.10, while the ratios at 800° to 1200°F were between 0.90 and 1.0. The notched data are presented in statistical form in the section under "Statistical Evaluation of Forgings and Weldments".

2. Weldment Test Results

Rolled ring forgings of three heats of Hastelloy X were welded circumferentially by the semi-automatic TIG process. The unnotched tensile test results from the welded rings made from Heats 261-5-4000, 261-5-4001, and

Figure 1
Hastelloy X, Typical Stress-Strain Curves

Heat 260-5-2813 are shown in Tables 3, 4, and 5, respectively. It was observed that all but three of the tensile specimens fractured in the parent metal as a result of higher strength (tensile and yield) manifested by the weldments. Since the fractures occurred in the parent metal the test data did represent the true tensile properties of the weld deposit. The latter is presented in Tables 6 and 7, which list the all-weld-metal tensile data. The all-weld-metal specimens confirmed the fact that both tensile and yield strengths were greater for the weldments than for the forgings.

The notch tensile data for the welded forgings are presented in Tables 8 and 9. Since the notches were intentionally located in the weld zone (as illustrated in Figure 2), but the unnotched tensile specimens did not fracture in the weld, the notch-to-unnotched ratios are abnormally high. Use of the notched tensile data divided by the ultimate strength of the all-weld-metal data is considered a better evaluation.

3. Statistical Evaluation of Forgings and Weldments

The data obtained from the tensile tests of the ring forgings, both welded and unwelded, was statistically analyzed to develop mean values and sigma values for the ultimate tensile strength, yield strength and elongation. The statistical approach was also used to evaluate the significance of the variables, such as specimen size, directionality and the location of specimens within the forging. The values were compared for both heats combined, and for each heat separately. The results for the combined heats are presented in Tables 10, 11 and 12. The computed mean values and minus-3 sigma values are shown in Figure 3, which also includes various offset yield values. The statistical results for the welded forgings are summarized in Table 13.

The following conclusions were drawn from the statistical study:

a. The R-3 specimens (0.250-in.-dia) exhibited higher ultimate strengths compared with the R-1 (0.500-in.-dia) at -320°F, but not at room temperature.

TABLE 3

TENSILE PROPERTIES OF WELDED RING FORGING FROM HASTELLOY X HEAT 261-5-4000

WELDED WITH HEAT 260-5-2784 WIRE
 (SIMULATED BRAZE CYCLE HEAT TREATMENT) (a)
ALL SPECIMENS ORIENTED AXIALLY IN FORGING

TEST TEMPERATURE (°F)	SPECIMEN SIZE (b)	UTS (KSI)	YS (KSI)	ELONG (%)	RA (%)	NOTCHED TENSILE (KSI)
-423	R-3	133.7	97.2	7.5	10.5	175
	R-3	141.8	95.3	8.7	8.5	175
	R-3	134.3	95.9	8.0	8.2	176
-320°F	R-3	128.9	73.5	10	12	
	R-3	125.9	77.4	11	12	
70	R-1	99.0	48.0	21	20	
	R-1	99.7	48.6	20	20	
	R-1	99.4	48.4	20	19	
400	R-1	85.9	39.4	18 (c)	18 (c)	
	R-1	89.9	40.1	22	19	
	R-1	88.7	36.9	25	25	
800	R-1	84.9	34.6	32	29	82.6
	R-1	83.0	36.8	24	22	85.0
	R-1	84.3	36.4	26	26	82.9
800	R-3	86.4	36.9	23	32	
	R-3	86.8	36.6	23	28	
1200	R-1	78.3	30.1	30	32	
	R-1	79.8	31.7	30	32	
	R-1	80.3	31.9	27	26	

NOTE: (a) Annealed at 2150°F prior to braze cycle.

(b) R-1 Specimens 0.502 ± 0.001 -in.-dia. x 2.00 in. gage length contained weldments which amounted to 15 to 20% of the gage length.

R-3 specimens 0.250 ± 0.001 -in. dia. x 1.00-in. gage length contained $40 \pm 5\%$ weld deposits.

(c) Fracture occurred outside the gage length.

TABLE 4

TENSILE TEST DATA (UNNOTCHED) FROM HASTELLOY X FORGING
 WELDED WITH HASTELLOY X WELD WIRE
 (FORGING HEAT 261-5-4001, WIRE HEAT 260-5-2783)

<u>TEMP (°F)</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONG (%)</u>	<u>RA (%)</u>
-423 (R-3)	136.6	87.0	9.0	10
(R-3)	136.0	86.9	7.0	11
-320 (R-3)	123.2	74.5	(10.2)	(10.1)
(R-3)	124.0	71.5	11.8	11.7
-320	123.1	68.9	11.5	13.4
	118.8	70.9	11.0	12.6
	118.6	67.9	11.5	13.0
	118.3	71.1	10.0	11.9
RT	94.1	43.5	19.5	18.8
	94.6	42.7	22.0	18.0
	95.0	43.8	(19.5)	17.3
400	883.9	36.5	21.0	20.7
	86.7	31.6	25.5	26.8
	85.1	33.2	24.0	23.3
800	80.2	31.7	26.5	31.1
	79.9	30.4	31.0	25.8
	77.6	31.7	25.0	26.5
	81.1	30.6	(26.5)	27.7
1200	74.9	28.5	21.5	21.4
	75.3	27.8	27.0	31.4
	76.2	27.7	28.0	33.3
	75.9	28.6	29.0	32.5

- NOTE:
1. All specimens size R-1 except those noted R-3, and heat treated through three-cycle simulated braze treatment after annealing at 2150°F.
 2. Strain rate 0.005 in./in./min to yield, then 0.05 in./in./min to failure.
 3. Forging procured to AGC 90056, Size 20-in. OD x 17-in. ID x 6-in. Long.
 4. Wire procured to AGC 90058.
 5. Values in parentheses indicate fracture occurred outside gage length.

TABLE 5

TENSILE TEST DATA (UNNOTCHED) FROM HASTELLOY X FORGING
 WELDED WITH HASTELLOY X WIRE
 (FORGING HEAT 260-5-2813, WIRE HEAT 261-5-4003)

<u>TEMP (°F)</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONG (%)</u> 1 in. for R-3	<u>RA (%)</u>
-423 (R-3)	126.1	85.5	5.5	9.4
-423 (R-3)	124.1	96.2	5.0	6.3
-423 (R-3)	122.9	98.7	4.0	4.8
-320 (R-3)	120.3	75.0	(8.5)	(9.3)
-320 (R-3)	123.0	76.1	10.4	10.8
	124.7	75.2	10.8	8.9
RT (R-3)	88.2	48.7	12.3	15.2
RT (R-3)	96.8	46.3	18.5	15.0
RT (R-3)	91.5	45.4	17.2	16.8
(Elong in 2 in.)				
-320 (R-1)	108.4	74.3	6.0	7.8
	108.4	75.3	6.0	5.9
	107.4	72.5	6.7	6.7
RT (R-1)	92.2	45.8	13.8	13.4
	93.2	45.9	14.9	13.4
	91.9	46.6	14.2	14.4
400 (R-1)	83.5	36.4	16.8	16.2
	80.9	35.4	16.0	16.2
	82.7	36.5	16.5	15.8
800 (R-1)	82.7	28.9	24.4	22.5
	82.9	30.8	24.4	26.3
	83.1	31.4	26.2	25.6
1200 (R-1)	77.6	--	25.7	26.9
	71.8	23.6	20.0	21.5
	75.9	24.0	22.9	22.3

- NOTE: 1. All specimens heat treated through three braze cycles after annealing at 2150°F.
2. Values in parenthesis () indicated fracture outside gage length.
3. Strain rate 0.005 in./in./min to yield, then 0.05 in./in./min to failure.

TABLE 6

ALL-WELD METAL TENSILE PROPERTIES FORGING HEAT 261-5-4000
WELDED WITH HEAT 260-5-2784 WIRE
(SIMULATED BRAZE CYCLE HEAT TREATMENT)

<u>TEST TEMPERATURE</u>	<u>UTS</u>	<u>YS</u>	<u>%E</u>	<u>%RA</u>
-320	154	77.6	18	18
-320	155.5	82.5	16	18
R.T.	115.5	48.6	25	30
R.T.*	111.2	53.4	16*	24
800°	98.5	40.6	25	29
800°	97.1	42.8	21	28

- * NOTE:
1. One specimen showed a pin point defect when checked by die penetrant. The fracture occurred at the indicated defect with some apparent loss of elongation.
 2. All specimens R-3.

TABLE 7

ALL-WELD-METAL TENSILE DATA FOR HASTELLOY X
(FORGING HEAT 261-5-4001 WELDED WITH WIRE HEAT 260-5-2783)

UNNOTCHED SPECIMENS

<u>TEMP (°F)</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONG, 1-IN. (%)</u>	<u>RA (%)</u>
-320	159.1	75.0	29.5	24
-320	144.0	73.0	22.0	21
-320	152.0	79.0	18.5	16
800	97.0	38.0	30.0	35
800	99.0	38.0	32.0	37
800	99.0	38.0	30.0	28

- NOTE:
1. Strain rate 0.005-in./in./min to yield, then 0.05 in./in./min to failure.
 2. All specimens heat treated through three-cycle simulated braze treatment after annealing at 2150°F.
 3. All specimens R-3 size.

TABLE 8

NOTCHED TENSILE TEST DATA
FROM HASTELLOY X FORGINGS - WELDED WITH HASTELLOY X WELD WIRE

NOTCHED R-3 SPECIMENS ($K_t = 6.3$)

<u>TEMP (°F)</u>	<u>UTS (KSI)</u>
-423	161.7
-423	164.0
-423	162.5
-320	147.2
-320	144.0
-320	144.0

NOTCHED R-1 SPECIMENS ($K_t = 6.3$)

-320	135.3
-320	137.8
-320	136.2
RT	105.1
	100.4
	105.0
400	92.8
	91.4
	82.0
800	84.3
	85.8
	85.8
1200	77.4
	79.5
	78.6

NOTE: All specimens heat treated through three-cycle
simulated braze treatment after annealing at 2150°F.
Forging heat 261-5-4001, weld wire Heat 260-5-2783.

TABLE 9

TENSILE TEST DATA (NOTCHED) FROM HASTELLOY X FORGING
 WELDED WITH HASTELLOY X WELD WIRE
 (FORGING HEAT 260-5-2813, WIRE HEAT 261-5-4003)

<u>TEMP (°F)</u>	<u>UTS (KSI)</u>
R-3 Specimens	
-423	139.9 (heat affected zone) ⁽³⁾ 160.0 (weld metal) 160.5 (weld metal)
-320	149.7 150.6 143.5
RT	113.6 116.3 115.4
800	95.7 90.9 97.9
R-1 Specimens	
-320	138.7 138.5 134.0
RT	103.7 103.2 106.1
400	84.6 95.1 92.8
800	89.1 91.4 90.7
1200	86.1 85.1 86.2

- NOTE:
1. All specimens heat treated through three-cycle simulated braze treatment after annealing at 2150°F.
 2. $K_t = 6.3$
 3. All specimens were notched in the weld deposit except the first one listed where the notch was located in the heat affected zone .010 to .060 in. from the fusion zone.

Welded Forging Tensile Specimens
(R-3), Etched to show Weld
Zone prior to testing.

Notch R-3 Tensile Specimens
showing Location of Notch in
the Weldment.

Figure 2
Notch, Tensile Specimens, Showing Weld Zone

TABLE 10

HASTELLOY X (AGC 90056) FORGINGS
STATISTICAL TENSILE (UNNOTCHED) DATA VERSUS TEMPERATURE

ULTIMATE TENSILE STRENGTH

<u>TEMP (°F)</u>	<u>MEAN VALUE (KSI)</u>	<u>ONE SIGMA (KSI)</u>	<u>SAMPLE SIZE</u>
-423	123.2	--	12
-320	117.4	3.7	30
70/80	93.3	--	11
400	81.7	--	9
800	79.8	1.3	37
1200	74.8	--	19

0.2% OFFSET YIELD STRENGTH

-423	86.6	--	12
-320	71.7	2.2	30
70/80	45.0	--	11
400	34.7	--	9
800	30.8	1.6	37
1200	26.8	--	19

ELONGATION

-423	8.1	--	12
-320	13.1	1.9	30
70/80	24.4	--	11
400	24.0	--	9
800	32.3	3.5	37
1200	31.4	--	19

- NOTE:
1. Data obtained from two heats of material, 261-5-4000 and 261-5-4001; as forged rings with 23-in. OD x 17-in. ID x 6-in. long.
 2. All specimens heat treated through three-cycle simulated braze treatment.
 3. This analysis includes all specimen orientation and locations on forgings.

TABLE 11

MEAN TENSILE (UNNOTCHED) PROPERTIES AND TEST VARIABILITY VS TEMPERATURE
FOR AXIAL AND TANGENTIAL PROPERTIES OF HASTELLOY X FORGINGS
FROM HEAT NOS. 261-5-4000 AND 261-5-4001

AXIAL ORIENTATION

<u>TEMP (°F)</u>	<u>UTS (KSI)</u>		<u>YS (KSI)</u>		<u>ELONG (%)</u>	
	<u>MEAN</u>	<u>ONE SIGMA</u>	<u>MEAN</u>	<u>ONE SIGMA</u>	<u>MEAN</u>	<u>ONE SIGMA</u>
-423	115.8		86.1		5.6	
-320	110.0	3.0	71.2	1.4	10.1	1.3
70/80	86.8		45.5		16.6	
400	78.9		35.1		19.7	
800	76.2	1.7	30.3	5.7	25.7	5.7
1200	73.0		26.8		27.2	

TANGENTIAL ORIENTATION

-423	130.6		87.0		10.2	
-320	124.9	5.3	72.3	3.5	16.1	2.6
70/80	97.0		44.7		30.0	
400	85.3		34.1		29.3	
800	82.2	1.1	31.1	2.3	36.7	1.8
1200	77.2		26.7		37.2	

NOTE: Forging size; 23-in. OD x 17-in. ID x 6-in. long.

All specimens heat treated through three-cycle simulated braze treatment.

TABLE 12

UNNOTCHED AND NOTCHED TENSILE STRENGTH OF HASTELLOY X FORGINGS
MEAN VALUES AND VARIABILITY VERSUS
TEMPERATURE, (HEATS 261-5-4000 AND 261-5-4001)

<u>TEST TEMP, °F</u>	<u>UNNOTCHED SPECIMENS</u>		<u>NOTCHED- UNNOTCHED RATIO</u>	<u>NOTCHED SPECIMENS</u>	
	<u>MEAN</u>	<u>ONE SIGMA</u>		<u>MEAN</u>	<u>ONE SIGMA</u>
-423	123.2		1.06	130.3	
-320	117.4	3.7	0.97	114.7	2.9
70	93.3		0.97	90.5	
400	81.7		0.94	76.6	
800	79.8	1.3	0.96	76.9	1.5
1200	74.8		0.93	69.4	

<u>TEST TEMP, °F</u>	<u>UNNOTCHED SPECIMENS</u>				<u>NOTCHED SPECIMENS</u>			
	<u>AXIAL</u>		<u>TANGENTIAL</u>		<u>AXIAL</u>		<u>TANGENTIAL</u>	
	<u>MEAN</u>	<u>ONE SIGMA</u>	<u>MEAN</u>	<u>ONE SIGMA</u>	<u>MEAN</u>	<u>ONE SIGMA</u>	<u>MEAN</u>	<u>ONE SIGMA</u>
-423	115.8		130.6		124.5		132.2	
-320	110.0	3.0	124.9	5.3	112.2	3.1	117.1	3.1
70	86.8		97.0		84.9		93.8	
400	78.9		85.3		78.8		80.3	
800	76.1	1.7	82.1	1.1	73.4	1.2	80.4	1.0
1200	73.0		77.2		65.8		73.1	

NOTE: 1. Forging size, 23-in. OD x 17-in. ID x 6-in. long.
2. All specimens heat treated through three-cycle simulated braze treatments.

Figure 3
Hastelloy X Forgings (AGC 90056) Design Tensile Strengths
versus Temperature (4 April 1966, Revised 23 May 1966)

TABLE 13

TENSILE PROPERTY MEAN VALUES AND TEST VARIABILITY VERSUS
TEMPERATURE, UNNOTCHED AND NOTCHED WELDED SPECIMENS

UNNOTCHED SPECIMENS

TEMP, °F	<u>SIZE R1</u> UTS (KSI)		YS (KSI)		E(%)		UTS (KSI)		<u>SIZE R3</u> YS (KSI)		E(%)	
	$\bar{Y}$	σ	$\bar{Y}$	σ	$\bar{Y}$	σ	$\bar{Y}$	σ	$\bar{Y}$	σ	$\bar{Y}$	σ
-423	-	-	-	-	-	-	131.9	3.04	92.8	4.48	6.8	.88
-320	117.3	1.64	72.8	1.43	9.67	.54	124.3	1.92	74.7	1.79	10.4	1.09
RT	95.5	.51	45.9	.45	18.3	.95	92.17	4.33	46.8	1.71	16.0	3.26
400	85.3	1.63	36.2	1.77	20.8	1.83	-	-	-	-	-	-
800	82.0	1.11	32.2	1.04	26.6	2.85	-	-	-	-	-	-
1200	76.6	1.74	28.2	1.49	26.1	2.83	-	-	-	-	-	-

NOTCHED SPECIMENS

-423							163.8	6.83
0320	136.5	2.91					146.5	3.03
RT	103.9	2.19					115.1	1.37
400	89.8	5.70					-	-
800	864	1.13					94.8	3.58
1200	82.1	.86					-	-

NOTE: The mean values in this table represent summation over forging heats

b. The specimen size (diameter) did not influence the yield strength or the elongation.

c. Specimen orientation within the ring forgings was a significant factor in ultimate strength and in the elongation values.

d. The ultimate strength values were also affected by the specimen location within the ring forgings.

e. The yield strengths were not significantly affected by location in the forgings.

4. Channel Strip Test Results

Tensile properties of the coolant channel strip (0.012-in.-thick) were determined for Heats 261-5-4000, -4001, and -4002.

The 0.012-in.-thick strips of all heats were run in an argon gas atmosphere to eliminate any effect of oxygen. The hardening effect of surface oxidation at 1600°F was previously observed to produce about a 10 to 12% increase in the ultimate tensile values, as well as a smaller increase in the yield strength which varied with time and, presumably, with specimen thickness.

The tensile test results are shown in Tables 14, 15, and 16, respectively. While the values of elongation measured at -320°F and higher were approximately 20% or greater, the elongation values observed at -423°F were generally below 10%. One experimental problem which appeared to contribute to the low elongation values was the tendency of the loading pins to bind up, which tended to induce tearing from one edge of the pin hole. Evidence of tearing was seen in some specimens which exhibited two or more fractures from one edge of the gage length. This problem was apparently manifested in the lower ultimate tensile strength values at -423°F as compared to the -320°F values. Correct values of ultimate tensile should be about 160 ksi, as was noted in Heat 261-5-4002 (see Table 16). The yield strength data are believed to be less affected by the

TABLE 14

TENSILE TEST DATA (UNNOTCHED) FOR 0.012-INCH THICK
HASTELLOY X STRIP
(HEAT 261-5-4000)

<u>TEST TEMPERATURE °F</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONGATION .2-IN. (%)</u>
-423	127.9	98.2	6.0
	140.3	94.4	7.0
	131.7	90.0	7.0
-320	140	73	22
	139	72	(20)
	141	71	(21)
RT	107	46	32
	103	47	(21.5)
	104	47	(20.5)
800	89	35	(32)
	88	35	37
	88	35	(32)
1200	80	31	37
	71	28	37
	77	31	35
1600	32	26	51
	34	28	43
	32	28	47
1800	18	16	41
	18	16	44
	17	15	45
	18	15	32

NOTE: 1. Strain rate 0.005 in./in./min to yield, then 0.05 in./in./min to failure.
 2. All specimens heat treated through three-cycle simulated braze treatment.
 3. Material procured to AGC 90057.
 4. Elongation values in () indicate failure outside gage marks.

TABLE 15

TENSILE TEST DATA (UNNOTCHED) FOR 0.012-INCH
HASTELLOY X STRIP
(HEAT 261-5-4001)

<u>TEST TEMPERATURE (°F)</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONGATION 2-IN. (%)</u>
-423	152.0	--	13.5
	143.9	93.0	(6.0)
	123.1	93.3	(4.5)
-320	144	80	19
	145	83	19
	148	78	24
RT	112	52	26
	109	47	26
	115	51	28
	111	53	23
800	92	37	29
	95	38	33
	92	42	25
1200	83	36	31
	86	--	24
	79	--	34
1300	75	33	34
1600	37	28	39
	40	27	40

- NOTE:
1. Strain rate 0.005 in./in./min to yield, then 0.05 in./in./min to failure.
 2. All specimens heat treated through three-cycle simulated braze treatments.
 3. Material procured to AGC 90057.
 4. Elongation values in () indicate failure outside gage marks.

TABLE 16

TENSILE TEST DATA (UNNOTCHED)
 FOR 0.012-IN.-THICK HASTELLOY X STRIP
(HEAT 261-5-4002)

<u>TEST TEMPERATURE (°F)</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONGATION 2-IN. (%)</u>
-423	131.4	93.6	(6.0)
	131.0	94.0	(5.5)
	162.0	--	--
-320	148	83	(21)
	148	80	(21)
	151	81	(23)
	149	78	(22)
RT	115	51	32
	115	49	30
	115	50	27
	112	50	(26)
800	96.2	40	(30)
	95	38	34
	94	42	33
	95	38	34
1200	87	35	38
	89	36	36
	79	33	34
	83	34	31
1600	34	23	40
	35	27	37
	36	26	32
	34	27	35

- NOTE:
1. Strain rate 0.005 in./in./min to yield, then 0.05 in./in./min to failure.
 2. All specimens heat treated through three-cycle simulated braze treatment.
 3. Material procured to AGC 90057.
 4. Elongation values in () indicate failure outside gage marks.

specimen test problem. The yield values (.2% offset) ranged from 90 to 98 ksi, with the majority of data at 93 to 94 ksi. The channel strip tensile values were consistently higher than the corresponding values for the forging material at low and elevated temperatures.

The notched strip tensile strengths for $K_t = 6.3$ are presented in Table 17. The notched strength values were substantially lower than the corresponding ultimate strength values from -423° to 1200°F ; however, at 1600° and 1800°F , the notched tensiles were higher than the ultimate tensile strength values.

The statistical mean values and the sigma values for the channel strip data were obtained by pooling the three heats. They are shown in Table 18. The data, excluding those values listed for -423°F are considered satisfactory. The -423°F data were adversely affected by the testing technique as mentioned above.

5. Shear, Compressive and Bearing Test Results

The design properties program included shear tests and bearing load tests on one heat of Hastelloy X and were designed to identify and verify the relationships between the tensile, shear, and bearing properties at several temperatures. The ultimate shear strength values for forging Heat 261-5-4000 were obtained at -320°F , room temperature, and 400°F , using the double-shear technique. The results are shown in Table 19. The values indicate that the ratio of ultimate shear to ultimate tensile strength increases to values of about 0.92 at -320°F . At 400°F the shear-to-tensile ratio was 0.81. Certain other nickel-base alloys, including Inconel 718, also show high shear strength at low temperature.

The bearing load tests were performed at temperatures from -320° to 800°F using specimens from Hastelloy X sheet (0.052 in.) and strip (0.012 in.) from Heat 261-5-4000. The results (Table 20) obtained were greater than the corresponding values previously obtained for austenitic stainless steels of comparable tensile strength (100 to 140 ksi for AISI 301 and 304 at room temperature).

TABLE 17

TENSILE TEST DATA (NOTCHED) FOR 0.012-IN.-THICK HASTELLOY X STRIP
NOTCH TENSILE STRENGTH (KSI)

<u>TEST TEMP.</u> <u>(°F)</u>	<u>HEAT NO.</u> <u>261-5-4000</u>	<u>HEAT NO.</u> <u>261-5-4001</u>	<u>HEAT NO.</u> <u>261-5-4002</u>
-423	115.3		117.1
	114.4		114.0
	112.7		119.0
-320	106.9		103.4
	104.4		104.0
	101.1		101.8
RT	79.6		78.4
	78.1		78.6
	78.5		81.5
800	62.3		67.1
	63.6		64.5
	62.6		65.3
1200	57.3		59.4
	56.7		61.2
	57.3		61.9
1600	42.0	41.6	34.3
	36.8	40.6	33.9
	40.4	40.2	43.2
1800	19.7		
	20.2		
	22.0		

- NOTE:
1. Strain rate 0.005 in./in./min to yield, then 0.05 in./in./min to failure.
 2. All specimens heat treated through three-cycle simulated braze treatment.
 3. Material procured to AGC-90057.
 4. $K_t = 6.3$

TABLE 18

TENSILE PROPERTY MEAN VALUES AND TEST
VARIABILITY VERSUS TEMPERATURE, UNNOTCHED
AND NOTCHED HASTELLOY X STRIP

UNNOTCHED STRIP

<u>TEST TEMP, °F</u>	<u>UTS (KSI)</u>		<u>YS (KSI)</u>		<u>E (%)</u>	
	<u>MEAN</u>	<u>STD. DEV.</u>	<u>MEAN</u>	<u>STD. DEV.</u>	<u>MEAN</u>	<u>STD. DEV.</u>
-423	135.2	10.25	93.8	2.91	6.98	3.01
-320	145.3	1.55	77.9	1.99	21.2	1.75
RT	110.7	2.07	49.4	1.71	26.5	3.82
800	92.4	1.11	38.0	1.58	31.9	3.92
1200	81.4	4.24	33.3	1.36	33.7	3.43
1600	34.8	1.95	26.4	1.78	39.5	3.30
1800	17.8	.33	15.5	.33	43.0	1.62

NOTCHED STRIP

<u>TEST TEMP, °F</u>	<u>UTS (KSI)</u>	
	<u>MEAN</u>	<u>STD. DEV.</u>
-423	115.4	2.01
-320	103.6	2.21
RT	9.1	1.35
800	64.2	1.06
1200	59.0	.94
1600	39.2	3.43
1800	20.6	1.21

NOTE: The mean values in this table represent summation over three forging heats.

TABLE 19

ULTIMATE SHEAR STRENGTH
OF HASTELLOY X FORGING
(HEAT 261-5-4000)

<u>TEST TEMP. (°F)</u>	<u>ORIENTATION</u>	<u>ULTIMATE SHEAR (KSI)</u>
-320	Axial	111.8
-320	Axial	104.0
-320	Axial	106.3
-320	Tangential	109.2
-320	Tangential	108.8
-320	Tangential	106.3
70/80	Axial	75.4
70/80	Axial	75.4
70/80	Axial	73.5
70/80	Tangential	74.9
70/80	Tangential	73.7
70/80	Tangential	71.4
400	Axial	65.6
400	Axial	64.0
400	Axial	64.0
400	Tangential	68.0
400	Tangential	68.9
400	Tangential	68.8

- NOTE: 1. All specimens treated through three-cycle simulated braze treatment.
2. Material procured to AGC 90056 as 23-in. OD x 17-in. ID x 6-in. long rings.
3. Specimens tested using double shear technique.

TABLE 20
HASTELLOY X BEARING LOAD TEST RESULTS

<u>Sheet Thickness, in.</u>	<u>Test Temperature, °F</u>	<u>Ratio: Edge Dist, Dia</u>	<u>Specimen Direction</u>	<u>Bearing Ulti- mate, F_{bru}, ksi</u>	<u>Nature of Fracture</u>
.012	-320	1.90	Longitudinal	252	Bearing
.012	-320	2.11	"	265	Bearing
.052	-320	2.04	Longitudinal	(∇ 323)	Tensile
.052	-320	2.01	"	(∇ 310)	"
.052	-320	2.07	"	(∇ 303)	"
.012	70	2.00	Longitudinal	205	Bearing
.012	70	2.06	"	229	"
.012	70	2.08	"	238	"
.012	70	2.03	Transverse	245	Bearing
.012	70	1.81	"	202	"
.012	70	2.11	"	244	"
.052	70	2.03	Longitudinal	233	Bearing First
.052	70	2.03	"	241	"
.012	400	2.08	Longitudinal	197	Bearing
.012	400	1.70	"	184	"
.012	400	2.06	"	201	"
.012	400	2.02	Transverse	207	Bearing
.012	400	1.72	"	171	"
.053	400	2.00	Longitudinal	207	Bearing
.053	400	2.00	"	203	"
.012	800	2.06	Longitudinal	181	Bearing
.012	800	2.07	"	172	"
.052	800	2.03	Longitudinal	173	Bearing
.052	800	2.04	"	167	"
.052	800	2.05	"	173	"

NOTE: 1. All specimens heat treated through three-cycle simulated braze treatment.
2. All material from Heat 261-5-4000.

The higher values for Hastelloy X illustrate the effect of the high shear strength (Table 19). The 0.052 sheet specimens (Figure 4) showed the unexpected tensile fracture at -320°F when the specimen withstood bearing loads in excess of 300 ksi. These values were calculated from the load divided by the pin diameter times the sheet thickness. The edge distance divided by the pin diameter was controlled at 2.0 in most cases (as shown on Table 20). The lower values, below 2.0, were found to produce smaller values of bearing ultimate (F_{bru}).

Figure 5 is a plot of the values for shear ultimate and the bearing ultimate strengths.

The results of the compressive strength tests are listed in Table 21. The compressive yield stresses were determined for both 0.2 and 0.4% offset, and appeared very consistent with the corresponding tensile yield stresses for the same forgings. The compression tests were continued to higher stress levels in order to evaluate the deformation obtained by excessive loads. After exceeding 10% compression, a non-uniform area increase (bulging) became the predominant deformation, without a corresponding area increase at the ends, and without any visible fracturing or defects. The compressive yield values of Heat 260-5-4000 were constantly higher than the corresponding values for Heat 260-5-2813 (which were in the same relation as the tensile values for the same heats).

The compression tests of Heat 261-5-4000 were performed in two directions (axial and short transverse) using square-end columns to determine if the plastic strain in compression was isotropic. The results, illustrated in Figure 6, indicated that there was no directionality difference. The corresponding directional variations in tension are believed to be the result of crack propagation characteristics after substantial strain, rather than plastic anisotropy of the Hastelloy X.

6. Physical Property Test Results

The physical properties of Hastelloy X relative to thermal stress use were determined. The tensile and shear moduli-of-elasticity were determined

Figure 4

Bearing-Load Test-Specimens from 0.052-in.-thick Hastelloy X Sheet
(Heat 261-5-4000) after Testing at Indicated Temperatures

Figure 5
Average Ultimate Tensile, Shear, and Bearing Strength
of Hastelloy X

TABLE 21

RESULTS OF COMPRESSION TESTS OF HASTELLOY X FORGINGS
ALL TESTS AT ROOM TEMPERATURE

Heat 261-5-4000

Axial Specimens

	Compressive Yield (KSI)	Final Stress (KSI)	Area (Max) ⁽³⁾ Increase (%)	Compression in Length (%)
<u>0.2%</u>	<u>0.4%</u>			
44.8	48.3	114.6	12.8	11.0
45.3	49.4	132.0	16.4	12.0
45.2	48.8	135.9	17.5	15.0

Short Transverse Specimens (Heat 261-5-4000)

44.3	48.1	142.5	22.9	16.0
45.2	49.2	140.0	22.0	15.2
45.0	48.7	140.0	22.9	15.4
46.0	49.6	140.6	22.4	15.2
45.9	49.4	139.6	22.2	15.0
45.1	48.5	139.4	22.1	15.2

Heat 260-5-2813

Axial Specimens

43.8	46.6	203.4	39.1	23.8
44.7	47.8	226.9	47.3	27.1
43.3	46.6	218.2	41.9	25.5
45.4	48.2	219.1	42.2	25.7
43.1	46.4	213.3	41.5	25.4
44.9	48.6	217.6	45.2	26.4

- NOTES:
1. Strain rate 0.005-in./in./min to yield, then 0.05 in./in./min to final load.
 2. All specimens heat treated through three-cycle simulated braze treatment.
 3. The maximum area represents area of the specimen at the maximum bulge after testing, transverse to the axis of compression.

Front view of short transverse compression block after 16% reduction in height. Note surface "orange peel" appearance.

Side view of compression block above.

Short Transverse (vertical) compression block before testing showing scribed gage lines and gold plated stripe for incremental compressive strain measurement.

Front view of axial compression blocks
after 11% (A-1) and 12% (A-2) reduction
in height.

Side view of same block on left.

at IASL and submitted in accordance with IASL Memo SN-A:HH(1196) dated 2 May 1966 (see Figure 7). The static values of the tensile modulus of elasticity are generally lower, as normally observed, and with a larger spread in the data. The static modulus is taken from the tensile stress-strain curves and is affected by any variations in the curves caused by strain rate, the extensometer response (lag), and the minor bending suffered at low stress levels by non-perfect specimens.

The thermal conductivity data computed from the thermal diffusivity data are shown in Figure 8. The thermal diffusivity data were obtained by measurements made at Battelle Northwest, in accordance with a letter of 20 May 1966, (R. E. Nightengale to H. Hessing, SXPO, Los Alamos). The specimens were from Hastelloy X, Heat 261-5-4000 forging, which was heat treated through the simulated braze cycles. The data compare very well with the reported values from Haynes Stellite (Hastelloy X) data sheets of 1959 and October 1964).

The coefficient of thermal expansion values are shown in Figure 9. The curve was obtained from an average of three tests, extending from room temperature to 1800°F, which was then correlated with cryogenic contraction data from room temperature to -400°F. The low temperature data were lower than the literature data, but were verified by re-runs and by three separate specimens in the "braze condition".

Figure 7

Hastelloy X Forgings (AGC 90056) Design Tensile and Shear Moduli
 versus Temperature (Source: LASL, Memo SN-A:HH(1196), dated 2 May 1966)

Figure 8
Thermal Conductivity of Hastelloy X (Calculated from Thermal Diffusivity
Determined by R. E. Nightingale at Battelle-Northwest)

Figure 9
Mean Coefficient-of-Expansion for Hastelloy X Forging,
Heat No. 261-5-4000 (Axial Orientation)

B. WELDING DEVELOPMENT

1. Development Qualification Testing

With the exception of material Heat 260-5-2813, the weld qualification results of the welded rings were reported in the previous Six Months Materials Report (Report No. RP-DR-0003). The rings from this heat were welded on the Reed-Avery boom welder and, thus, they served as the certification of this welding equipment as a back-up machine to the Cayuga Boom Welder for nozzle jacket welding.

The qualification tensile test results for Heat 260-5-2813, welded with wire from Heat 261-5-4003, were as follows, for the annealed (2150°F) condition:

<u>Temp</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONG %</u>	
			<u>1.0-in.</u>	<u>2.0-in.</u>
Room	95.5	46.9	60	50
Room	95.7	46.2	64	54
1200°	64.6	29.1	70	58

All failures occurred in the parent metal with double necking (on either side of the weld). The fractures exhibited greater ductile characteristics than the test specimens from the previous heats (261-5-4000 and 261-5-4001) of ring forgings. This was manifested primarily by the higher elongation values. The following table compares the average 1.0-in. and 2.0-in. gage length ductilities from each of the welded ring forgings at room temperature:

<u>Ring Forging Heat No.</u>	<u>ELONG (%)</u>	
	<u>1.0-in.</u>	<u>2.0-in.</u>
261-5-4000	48	43
261-5-4001	47	43
260-5-2813	62	52

Figure 10 illustrates a typical macrostructure of the weld from the heat 260-5-2813 ring forging.

Figure 10

Typical Macrostructure of Ring Forging from Heat 260-5-2813
Welded with Wire from Heat 261-5-4003

A hardness survey of heat 260-5-2813 is shown in Figure 11. Lower hardness values were obtained from this single data point survey compared with the duplicate data point surveys conducted on each of the rings from the other material heats. The averaged comparison of data for all three heats is as follows:

<u>Ring</u>	<u>Heat No.</u>		<u>Hardness, Rockwell B</u>	
	<u>Weld Wire</u>	<u>Parent Metal</u>	<u>Weld Metal</u>	
260-5-2813	261-5-4003	89	86	
261-5-4000	260-5-2784	93	92	
261-5-4001	260-5-2783	95	95	

2. Dissimilar Metals Welding

The task of evaluating dissimilar metals welding was completed in July 1966. The objective was to provide data for use in selecting a filler wire and to develop a procedure for welding the torus inlet and adapter of the Phoebus nozzle which are composed of, respectively, Hastelloy X and AISI 347 stainless steel. The filler wires selected for evaluation are those most commonly employed for dissimilar metal welding, namely: Inconel A and Hastelloy W. The evaluation was accomplished by welding a total of six test pressure vessels. The design of these vessels is shown in Figure 12. Three of the six vessels were girth-welded using Inconel A as a filler wire, while the remaining three were joined with Hastelloy W wire. All welds were made by the tungsten inert-arc (TIG) process and, in each instance, one of the three pressure vessels was welded manually while the remaining two were welded automatically. The joint preparation consisted of a single "V" with a 90° included angle and a .060-in. root land. Mechanical testing was performed by submitting four of the six vessels to proof pressure and one to burst pressure, as well as by sectioning the remaining two into tensile and metallographic specimens. Transverse weld tensile test results were as follows:

INCONEL A FILLER WIRE

<u>Specimen No.</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>Elong % 2-in.</u>	<u>RA</u>	<u>Location of Failure</u>
1	90.2	51.1	38.5	64.7	347 P.M.
2	91.3	51.1	40.0	64.4	347 P.M.
3	91.8	50.0	40.5	63.4	347 P.M.

RING WELDMENT S/N 012, HEAT NO. 260-5-2813, WELDED WITH HEAT NO. 261-5-4003

Figure 11
Parent Metal-to-Weld-Metal Hardness Traverse

Figure 12
Test Pressure Vessel Design

HASTELLOY W FILLER WIRE

<u>Specimen No.</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>Elong % 2-in.</u>	<u>RA</u>	<u>Location of Failure</u>
1	89.7	48.2	38.5	66.0	347 P.M.
2	89.9	51.7	40.0	66.5	347 P.M.
3	89.4	50.6	-	63.3	347 P.M.

The typical chemical analyses of the Inconel A and Hastelloy W weld wires are as follows:

INCONEL A

<u>C</u>	<u>MN</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Cu</u>	<u>Cr</u>	<u>Ni</u>	<u>Ti</u>	<u>Fe</u>
.10	2.0/2.75	--	.015	.35	.50	14.0/17.0	67.0	2.5/3.5	10.0
Max			Max	Max			Min		Max

HASTELLOY W

.12	1.00	.040	.030	--	--	4.0/6.0	Bal	--	4.0/7.0
Max	Max	Max	Max						

One of the four vessels was tested to burst pressure. This vessel (No. 2) was semi-automatic TIG welded with Hastelloy W filler wire and failed at an internal pressure of 5800 psi. Failure initiated in a fissure or scratch (caused by grinding during removal of excess weld crown on the ID of the girth weld) which propagated to a small crack resulting in failure at that point. Without the failure-initiating grinding fissure the vessel might have sustained a slightly higher pressure. The three remaining vessels (Nos. 3, 4, and 5) satisfactorily passed proof testing at an internal pressure of 1450 psi.

Samples from each of the two welded joints (Inconel A and Hastelloy W) were examined metallographically. The photomicrographs in Figure 13 compare the cast-weld structure of Inconel A with that of Hastelloy W, and indicate that both structures are similar: each displays coring or segregation common to nickel base alloys. Hardness surveys, representative of both the Inconel A and Hastelloy W weld deposits are shown in Figures 14 and 15, respectively.

Figure 13

Microstructure (100X) of Weld Metal, Inconel A Filler Wire (Upper)
and Hastelloy W Wire (Lower) Etchant: Oxalic Acid

Figure 14
Hardness Survey Across Inconel-A Weld Deposit

Figure 15
Hastelloy Survey Across Hastelloy-W Weld Deposit

Even though evaluation of the results of the dissimilar metals project demonstrated that either Inconel A or Hastelloy W could be used to join Type 347 material to Hastelloy X with satisfactory results, the better fluidity and more favorable microstructure of the Hastelloy W weld deposit made it the preferred filler wire for the Phoebus inlet and adapter weld.

3. Nozzle Jacket Welding

As of the end of this report period (August 15) five convectively cooled nozzle jacket assemblies and a pressure-test nozzle have been welded. The nozzle serial numbers, the material heat number of each of the three forgings which comprise the nozzle jacket assembly, and the material heat numbers of the weld filler material used on each nozzle as well as the date of the final radiographic acceptance of each jacket are itemized in Table 22.

As the nozzle welding program progressed, refinements in the welding conditions evolved as problem areas were identified. Changes were made to prevent, or minimize, the recurrence of specific types of weld defects. With the exception of nozzle serial number 880601 no major problems were encountered and the quality and efficiency of the semi-automatic nozzle welding has steadily improved.

The problems encountered with nozzle serial number 880601 were precipitated by attempts to make manual weld repairs of defects in the weld of the aft (or convergent-to-divergent) joint which were detected during final radiographic inspection. Deep grind-outs (in excess of 5/16-in.) were required to remove defects. Attempts to make manual weld repairs resulted in multiple re-welds of local areas because of repeated cracking of the repair welds. In some areas the problem was further compounded by the necessity of grinding even deeper than the original defect depth in order to remove defects incurred by the manual repair attempts. The concentrated heat input and accompanying thermal stresses imposed locally by manual welding provided all of the conditions which induce cracking in Hastelloy X welds. In addition, manual welds introduce another variable - one which cannot be

TABLE 22

PHOEBUS NOZZLE JACKET WELDMENT DATA

Nozzle Jacket Serial No.	Nozzle Forging Identification and Material Heat No.			Filler Wire Material Heat No.	Weldment Acceptance Date	Comments
	Section	Dash No.	Heat No.			
880600	Ring	-13	260-5-2793	260-5-2783	2-23-66	Ring and Convergent section welded first. Divergent section delivery delayed - minor porosity in weld.
	Divergent	-11	260-5-2850			
	Convergent	- 7	260-5-2793			
880602 Pressure Test Nozzle	Ring	-13	260-5-2793	260-5-2784	3-6-66	Excessive porosity in weld attributed to defective length of weld wire.
	Divergent					
	Convergent	-15	260-5-2820			
880601	Ring	-13	260-5-2793	260-5-2784	5-6-66	Weld Repair of aft joint defects compounded by manual repair difficulties
	Divergent	-11	260-5-2863			
	Convergent	- 7	260-5-2850			
880603	Ring	-13	260-5-2901	260-5-4003	6-17-66	First nozzle welded in new weld station Bldg. 2004. Minor weld repair root passes defects incurred by malfunction of Reed-Avery weld unit.
	Divergent	11	260-5-2887			
	Convergent	7	260-5-2880			
880604	Ring	13	260-5-2901	260-5-4003	7-22-66	First nozzle welded under plastic canopy - Minor weld defects root pass only.
	Divergent	11	260-5-2887			
	Convergent	7	260-5-2880			
880605	Ring	13	260-5-2901	260-5-4003	8-16-66	Last deliverable nozzle. Minor weld repair aft joint root pass.
	Divergent	11	260-5-2920			
	Convergent	7	260-5-2900			

efficiently controlled - that of individual welder technique. Efforts to develop manual weld procedures and techniques which would result in successful manual repairs have been only partially successful. The difficulties inherent in making manual repairs resulted in the perfection of semi-automatic weld repair procedures, which have been used successfully when the depth of the grind-out does not exceed 3/16-in.

Porosity is perhaps the most commonly encountered weld defect, and the possible causes are many, including: excessive weld travel speed, excessive heat input and contaminants from sources such as joint surface, weld wire or shielding gas. Some of the first porosity encountered in the development welding was caused by contaminated weld wire. This condition was rectified and brought under control by improving the weld wire procurement specification (AGC-90058). Strict adherence to inter-pass cleaning procedures has contributed to the minimized incidence of gas porosity in the nozzle jacket welds.

A plastic canopy, Figure 16, which was installed in the new Phoebus weld facility in Building 2004, provides protection from air borne dust and dirt particles and shields the nozzle from drafts which disturb the inert gas atmosphere shielding during welding.

Figure 16

Plastic Canopy Installed Over Phoebus Nozzle Jacket During Welding

C. BRAZING DEVELOPMENT

1. Braze Joint Strengths

All mechanical properties specimens to evaluate the braze joint strengths were fabricated and tested.

The tensile test results for the 1/4-in.-round (Figure 17) specimens are presented in Table 23 for parent metal (exposed to Nicro braze cycle) and Nicro brazed, copper brazed, Palniro-7 brazed and Nicoro-80 brazed, respectively. The range of test results are listed below for each of the conditions tested. All failures occurred in the braze joint indicating the Nicro braze alloy is lower in strength than the Hastelloy X; however, with the exception of the copper braze, the tensile strengths all approximated the parent metal at room temperature. At 1200°F the braze joint strength ranged from 70% to 34% of the parent metal reflecting the more pronounced effect of temperature on the braze alloys (with lower melting ranges) than the high temperature (and higher melting range) Hastelloy X parent metal.

RANGE OF TENSILE STRENGTH VALUES FOR BRAZED 1/4-IN.-ROUND SPECIMENS

<u>Temp (°F)</u>	<u>Parent Metal</u>	<u>Nicro Braze</u>	<u>Copper Braze</u>	<u>Palniro-7 Braze</u>	<u>Nicoro-80 Braze</u>
Room	104.0 - 106.0	82.1-101.0	56.5-68.6	91.0-101.0*	107.0-108.0
1200	74.5-77.1	35.9-50.9	30.3-33.4	37.6-53.0	26.0-30.5
-320	112.6-127.6**	116.0-141.0	-	-	-

* One specimen with incomplete braze coverage resulted in failure at 49.9 ksi

** Statistical range from design properties program forging heats 261-5-4000 and 261-5-4001

The test data at minus 320°F were restricted to the Nicro alloy based on its contact with the low temperature environment of the nozzle. The tests were conducted primarily for engineering information, and they reveal strength

Figure 17
Round Tensile Specimen

TABLE 23

TENSILE TEST RESULTS FOR 0.250-INCH ROUND
HASTELLOY X BAR STOCK SPECIMENS (DWG. 1116011)

PARENT METAL - CYCLED 3 HOURS AT 1850°F, COOLED AT RATE EQUIVALENT TO
SLOW AIR COOL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>1-in. Elong. (%)</u>
N1 (BM)	RT	104,000	44,900	38.0
N2 (BM)	RT	106,000	47,000	38.0
N3 (BM)	RT	106,000	45,300	39.0
N4 (BM)	RT	105,000	44,200	38.0
N5 (BM)	RT	104,000	44,400	38.0
N6 (BM)	1200	74,500	29,200	64.5
N7 (BM)	1200	75,900	29,900	64.0
N8 (BM)	1200	77,100	30,000	59.0
N9 (BM)	1200	75,400	28,700	63.0
N10 (BM)	1200	77,100	28,400	60.0

NIOBO BRAZE - CYCLED 3 HOURS AT 1850°F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
1	-320	131,000
2	-320	121,000
3	-320	141,000
4	-320	116,000
5	-320	128,000
6	-320	131,000
N1	RT	94,600
N2	RT	100,000
N3	RT	101,000
N4	RT	82,100
N5	RT	96,700
N6	1200	50,900
N7	1200	35,900
N8	1200	48,100
N9	1200	42,700
N10	1200	46,600

TABLE 23 (cont.)

COPPER BRAZE - CYCLED 3 HOURS AT 2025°F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
C1	RT	63,500
C2	RT	68,600
C3	RT	56,500
C4	RT	66,300
C5	RT	65,900
C6	1200	32,200
C7	1200	32,100
C8	1200	31,600
C9	1200	33,400
C10	1200	30,300

PALNIRO-7 BRAZE - CYCLED 3 HOURS AT 1975°F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
P1	RT	100,000
P2	RT	49,900
P3	RT	101,000
P4	RT	98,900
P5	RT	91,900
P6	1200°F	53,000
P7	1200°F	51,800
P8	1200°F	37,600
P9	1200°F	52,700
P10	1200°F	50,300

TABLE 23 (cont.)

NICORO-80 BRAZE - CYCLED 3 HOURS AT 1750°F, COOLED AT RATE EQUIVALENT TO SLOW
AIR COOL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
8 N3	RT	108,000
8 N4	RT	107,000
8 N5	RT	107,000
8 N6	1200	26,000
8 N7	1200	30,500
8 N8	1200	27,000
8 N9	1200	27,600
8 N10	1200	28,800

equivalent to the parent metal. Data at -320°F were not available on the actual parent metal stock utilized in this investigation but, based on the room temperature tensile strength of 104.0 to 106.0 ksi and data from the design properties program, it is expected the actual value at -320°F would have been about 137.0 ksi.

The flat tensile specimen (Figure 18) data (0.012-in.-thick strip) are presented in Table 24 and compare the parent metal strength and ductility with the effects of surface diffusion by each braze alloy. The results revealed the brazed specimens possessed the following: tensile strength equivalent to the parent metal at room temperature and at 1200°F, slightly higher yield strengths than the parent metal at room temp and at 1200°F, lower elongation than the parent metal (except for Nicoro-80 at room temp) at room temp and 1200°F. A summary comparison of the test data is itemized below:

RANGE OF TENSILE PROPERTIES FOR SURFACE BRAZED FLAT TENSILE SPECIMENS

Temp (°F)	UTS (KSI)	YS (KSI)	Elong %	Alloy	UTS (KSI)	YS (KSI)	Elong (%)
Room	111.0-117.0	57.4-58.1	14-17.5	Nicro	109.0-114.0	63.9-68.4	10.5-13.5
Room	112.0-116.0	52.5-53.3	23.5-32.	Copper	103.0-117.0	57.6-67.6	18.5-26
Room	110.0-115.0	52.2-52.4	19.5-27.5	Palniro-7	95.3-112.0	60.3-69.6	5-21
Room	113.0-115.0	51.7-53.0	23-27	Nicoro-80	115.0-117.0	61.1-63.2	27-29
1200	83.7-93.5	40.1-41.5	17.5-32	Nicro	86.3-90.0	43.8-46.2	8.5-20
1200	80.1-84.7	34.9-35.1	29.5-43	Copper	74.5-83.4	41.7-44.5	17.5-27
1200	86.3-89.6	36.6-37.9	32-37	Palniro-7	77.2-88.9	41.6-43.7	13.5-29.5
1200	85.0-89.6	38.3-40.9	36.5-37.5	Nicoro-80	83.9-87.2	40.7-42.4	28.5-30

Results of the simulated seal joint tests (Figure 19) are presented in Table 25 for two complete three-cycle braze treatments. The average test data are as follows (all failures occurred in parent metal):

Temp (°F)	Braze Cycle	UTS (KSI)	PARENT METAL Elong (%)	BRAZED UTS (KSI)
Room	1850°F/1825°F/1750°F	114.7	28	96.7
1200	1850°F/1825°F/1750°F	87.1	39	72.6
Room	1975°F/1950°F/1825°F	104.0	12	92.6
1200	1975°F/1950°F/1825°F	88.4	32	84.2

Figure 18
Flat Tensile Specimen

TABLE 24

TENSILE TEST RESULTS FOR 0.012-INCH THICK
HASTELLOY X STRIP BRAZE ALLOY DIFFUSION
SPECIMENS (DWG. 1116012)

BRAZE CYCLE: 3 HOURS AT 1850°F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

BRAZE ALLOY: NIORO

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1D1BMA	RT	111,000	58,100	14.0
1D1BMB	RT	116,000	58,500	15.5
1D1BMC	RT	117,000	57,400	17.5
1D2BMA	1200°F	83,700	40,400	17.5
1D2BMB	1200°F	93,500	40,100	32.0
1D2BMC	1200°F	90,700	41,500	28.0

NIORO SURFACE BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1D1A	RT	113,000	63,900	13.5
1D1B	RT	109,000	64,800	10.5
1D1C	RT	113,000	68,400	10.5
1D1D	RT	112,000	64,400	11.5
1D1E	RT	114,000	64,200	11.5
1D2A	1200	88,600	45,100	18.0
1D2B	1200	76,300	45,100	8.5
1D2C	1200	86,300	45,700	17.5
1D2D	1200	89,900	45,300	18.5
1D2E	1200	88,500	43,800	18.5
1D2F	1200	90,000	46,200	20.0

TABLE 24 (cont.)

BRAZE CYCLE: 3 HOURS AT 2025 °F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

BRAZE ALLOY: COPPER

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1B1BMA	RT	116,000	52,500	32.0
1B1BMB	RT	112,000	53,300	31.5
1B1BMC	RT	114,000	53,200	23.5
1B2BMA	1200	80,100	35,100	29.5
1B2BMB	1200	84,500	34,900	43.0
1B2BMC	1200	84,700	35,500	42.5

COPPER SURFACE BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1B1A	RT	108,000	62,700	19.5
1B1B	RT	103,000	57,600	18.5
1B1C	RT	117,000	67,600	26.5
1B1F	RT	112,000	59,500	26.0
1B1E	RT	113,000	65,600	26.0
1B2A	1200	80,100	44,500	21.0
1B2B	1200	83,400	43,100	26.0
1B2C	1200	82,600	44,500	27.0
1B2D	1200	79,600	44,500	25.5
1B2E	1200	74,500	41,700	17.5

TABLE 24 (cont.)

BRAZE CYCLE: 3 HOURS AT 1975°F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

BRAZE ALLOY: PALNIRO-7

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1A1BMA	RT	110,000	52,200	19.5
1A1BMB	RT	116,000	52,400	27.5
1A1BMC	RT	115,000	52,400	26.5
1A2BMA	1200	86,300	36,600	37.0
1A2BMB	1200	89,600	37,000	34.5
1A2BMC	1200	87,000	37,900	32.0

PALNIRO-7 SURFACE BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1A1A	RT	110,000	65,300	13.5
1A1P	RT	95,300	69,600	5.0
1A1L	RT	103,000	60,500	11.5
1A1L	RT	112,000	60,300	21.0
1A1L	RT	97,700	61,200	9.0
1A2A	1200	78,600	43,700	16.5
1A2B	1200	80,300	42,800	16.0
1A2	1200	77,200	41,600	13.5
1A2L	1200	88,900	43,200	28.5
1A2E	1200	87,600	41,000	29.5

TABLE 24 (cont.)

BRAZE CYCLE: 3 HOURS AT 1750°F, COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

BRAZE ALLOY: NICORO-80

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1C1BMA	RT	113,000	51,900	23.0
1C1BMB	RT	113,000	51,700	27.0
1C1BMC	RT	115,000	53,000	23.0
1C2BMA	1200	89,600	38,300	36.5
1C2BMB	1200	88,000	40,900	36.5
1C2BMC	1200	85,000	39,400	37.5

NICORO-80 SURFACE BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1C1A	RT	117,000	63,200	27.5
1C1B	RT	117,000	62,400	28.0
1C1C	RT	115,000	61,100	27.0
1C1D	RT	116,000	61,700	28.0
1C1E	RT	117,000	62,600	29.0
1C2A	1200	86,700	41,100	28.5
1C2B	1200	85,600	42,400	30.0
1C2C	1200	83,900	40,700	30.5
1C2D	1200	87,200	41,700	30.5
1C2E	1200	86,300	41,900	29.5

Figure 19
Simulated Seal Joint Tests

TABLE 25

TENSILE TEST RESULTS FOR HASTELLOY X SIMULATED
SEAL JOINT SPECIMENS (DWG 1116013) EXPOSED
TO MULTIPLE BRAZE CYCLES

CYCLES: 3 HOURS AT 1850°F (NIORO) + 3 HOURS AT 1825°F (NIORO + PALNIRO-7)
+ 3 HOURS AT 1750°F (NICORO-80), COOLED AT RATE EQUIVALENT TO SLOW
AIR COOL AFTER EACH CYCLE

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
1CABM	RT	114,000	55,500	24.5
1CBBM	RT	114,000	52,700	30.0
1CCBM	RT	116,000	52,600	30.0
1DABM	1200	86,500	37,600	38.0
1DBBM	1200	87,800	37,600	39.0
1DCBM	1200	86,900	36,700	41.0

BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
1AA	RT	110,000
1AB	RT	79,700
1AC	RT	97,900
1AD	RT	96,600
1AE	RT	99,200
1BA	1200	83,600
1BB	1200	78,200
1BC	1200	75,700
1BD	1200	73,300
1BE	1200	82,100

TABLE 25 (cont.)

CYCLES: 3 HOURS AT 1975°F (PALNIRO-7) + 3 HOURS AT 1950°F (PALNIRO-1 AND PALNIRO-7) + 3 HOURS AT 1825°F (NIORO), COOLED AT EQUIVALENT TO SLOW AIR COOL AFTER EACH CYCLE.

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
2CABM	RT	107,000	50,500	12.5
2CBBM	RT	105,000	51,900	14.0
2CCBM	RT	100,000	51,700	10.5
2DABM	1200	85,800	37,400	25.5
2DBBM	1200	90,000	30,700	33.5
2DCBM	1200	89,400	36,000	37.0

BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
2AA	RT	89,300
2AB	RT	91,400
2AC	RT	91,300
2AD	RT	97,300
2AE	RT	93,500
2BA	1200	88,900
2BB	1200	73,800
2BC	1200	85,500
2BD	1200	93,100
2BE	1200	79,800

A slight loss in tensile strength occurred at room temperature for the parent metal and the brazed specimens during the 1975/1950/1825 cycle. At 1200°F the parent metal appears unaffected but the brazed specimens increased in strength. Since all specimens failed in the parent metal it can be assumed that both sets of data represent normal spread in the parent metal samples, in which case the room temperature strength is lowered by the high temperature braze cycle and an apparent slight increase occurs at 1200°F. The significant feature of these data is the effect on parent metal ductility, which is lowered a pronounced amount at room temperature by the high temperature braze cycle and, although considerable recovery occurs at 1200°F, the value has not returned to the level of the low temperature braze cycle.

The effects of the individual braze alloys, using simulated seal joint specimens, are shown in Table 26. The tensile strengths of the brazed specimens are comparable to the parent metal at room temperature and 1200°F, except for copper at 1200°F which averages about 10 ksi lower than the Nioro, Palniro-7 and Nicoro 80 specimens.

Ten each of the Miller-Peaslee shear-test specimens (Figure 20) brazed with Nioro, copper, Palniro-7 and Nicoro-80 were shipped to ORNL for testing. Testing of unirradiated specimens at room temperature and 750°F has been conducted at ORNL. The unirradiated tests at -320°F are yet to be performed. Irradiation testing is scheduled for September 1966. In addition five thermal-fatigue specimens, consisting of five tubes Nioro brazed to a 1-in.-thick plate, were sent to ORNL for testing.

2. Full Scale Sectors

The full scale Hastelloy X sectors were contour machined at Standard Tool and Die. Machining stresses aided by the interrupted cut resulted in considerable residual stresses requiring the welding of stiffeners to the sectors to minimize distortion. The two least distorted sectors were sent to the Tracer Corporation and seven grooves were electrically discharge machined (EDM) into each part.

TABLE 26

TENSILE TEST RESULTS FOR HASTELLOY X SIMULATED
SEAL JOINT SPECIMENS (DWG 1116013) EXPOSED TO
SINGE BRAZE CYCLES

CYCLE: 3 HOURS AT 1850°F (NIORO), COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
3CABM	RT	112,000	55,600	18
3CBBM	RT	99,400	53,600	13
3CCBM	RT	107,000	54,500	20
3DABM	1200	84,200	35,500	40
3DBBM	1200	82,700	36,500	33
3DCBM	1200	84,700	36,300	38.5

BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
3AA	RT	111,000
3AB	RT	107,000
3AC	RT	105,000
3AD	RT	102,000
3AE	RT	109,000
3BA	1200	83,300
3BB	1200	69,800
3BC	1200	76,800
3BD	1200	82,700
3BE	1200	79,500

TABLE 26 (cont.)

CYCLE: 3 HOURS AT 2025°F (COPPER), COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
5CABM	RT	101,000	52,400	10.5
5CBBM	RT	108,000	55,500	18.0
5CCBM	RT	109,000	53,900	11.0
5DABM	1200	86,800	36,100	34.0
5DBBM	1200	88,100	36,200	36.0
5DCBM	1200	80,600	35,400	22.5

BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
5AA	RT	96,800
5AB	RT	95,900
5AC	RT	93,600
5AD	RT	110,000
5AE	RT	106,000
5BA	1200	69,000
5BB	1200	72,500
5BC	1200	72,700
5BD	1200	69,000
5BE	1200	73,000

TABLE 26 (cont.)

CYCLE: 3 HOURS AT 1975 °F (PALNIRO-7), COOLED AT RATE EQUIVALENT TO SLOW AIR COOL

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
6CABM	RT	112,000	52,500	26.0
6CBBM	RT	112,000	54,200	25.0
6CCBM	RT	116,000	55,300	30.0
6DABM	1200	84,200	36,200	42.0
6DBBM	1200	85,400	37,300	41.5
6DCBM	1200	87,800	37,700	43.0

BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
6AA	RT	106,000
6AB	RT	104,000
6AC	RT	101,000
6AD	RT	94,200
6AE	RT	108,000
6BA	1200	85,500
6BB	1200	80,900
6BC	1200	81,600
6BD	1200	80,800
6BE	1200	81,400

TABLE 26 (cont.)

CYCLE: 3 HOURS AT 1750°F (NICORO-80), COOLED AT RATE EQUIVALENT TO SLOW
AIR COOL

PARENT METAL

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>	<u>0.2% YS (psi)</u>	<u>2-in. Elong. (%)</u>
4CABM	RT	115,000	52,900	27
4CBBM	RT	113,000	53,100	24
4CCBM	RT	109,000	52,700	18.5
4DABM	1200	80,500	38,300	23
4DBBM	1200	90,000	38,400	37.5
4DCBM	1200	86,800	37,200	37.5

BRAZED

<u>Specimen Designation</u>	<u>Test Temp (°F)</u>	<u>UTS (psi)</u>
4AA	RT	115,000
4AB	RT	117,000
4AC	RT	117,000
4AD	RT	111,000
4AE	RT	115,000
4BA	1200	85,400
4BB	1200	85,200
4BC	1200	81,100
4BD	1200	85,200
4BE	1200	84,100

M-P SPECIMEN BEFORE BRAZING

M-P SPECIMEN AFTER BRAZING

Figure 20
Miller-Peaslee Shear Test Specimen (Scale: 2 times)

The first EDM grooved contour sector was cleaned, assembled and brazed at Pyromet, Inc., San Carlos, California, in accordance with AGC 90006B. A dull, gray surface film was observed on all Hastelloy X surfaces after the brazing cycles. The current AGC Hastelloy X materials specification allow a combined aluminum plus titanium content of 0.5% maximum. A review of the certifications for the heats of material involved revealed aluminum contents of 0.13 to 0.40 and titanium from 0.001 to 0.03. Electron diffraction patterns from the brazed specimen surfaces have confirmed the presence of aluminum oxide. The Union Carbide Corporation - Stellite Division, supplier of the Hastelloy X material, reported that aluminum is intentionally added as a melt deoxidizer. The Phoebus-2 materials specifications (AGC 90056, 90057, 90058 and 90060) have been changed to limit aluminum, titanium and zirconium to 0.02% maximum for future procurements.

After brazing, the side supports or rails were installed on the sector and the hydrotests were accomplished. A total of 67 leaks were detected during the 90-pound leak test. The leaks were torch repaired manually using BT alloy (72 Ag-28Cu) and the sector was then proof-tested satisfactorily at 1465 psi. Next, the sector was hydrotested to failure, which occurred at 3700 psi in a braze joint at the cylindrical section.

The spot-weld of the tube wall to the groove-faying surface (which is done to insure good tube-to-groove engagement and bottoming) was not present in the failed area. The scallop for the spot-welding tip was not machined into the sector because the stiffener side plates (used to minimize distortion) resulted in an interference. Metallographic examination revealed the failed area contained only about 35% joint coverage and the fillet in the opposite joint was deficient; thus, the joint that failed was locally carrying the load of two tubes.

The metallographic examination also revealed the reduced wetting and resultant "tunneling" of the Nicoro-80 alloy in the non-structural sealing layer. The lack of wetting is attributed to the aluminum oxide film on the tube surfaces.

Prior to assembly and brazing of the second, full length, contoured sector additional testing was conducted with 20-in.-long grooved specimens. The first 20-in. specimen was brazed with a slightly faster heating rate (100°F part temperature differential) than the standard (50°F differential), in an attempt to minimum the aluminum oxide formation. The remaining cycles conformed to AGC 90006B. Changes in the last, or sealing, cycle involved variations in the application of the Nicoro-80 braze alloy: one joint was loaded with all fine-mesh powder, one joint with coarse mesh, and two joints with 0.5% phosphorus Nicoro-80. Four different heats of Nicoro-80 were included. No significant differences were noted for the different heats or mesh-size variations for the Nicoro-80; however; the phosphorus addition greatly improved flow characteristics. Unfortunately, intermetallic phosphorus compounds were readily apparent during microexamination.

The significant changes to the second 20-in. specimen were: the use of nickel plating (Model Plating Co.) on one-half of the tubes with Nicoro-80, and no plating on the other one-half of the tubes with the braze alloy Palcusil-25. The Palcusil-25 had better wetting and flow characteristics but produced lower fillets than the Nicoro-80, for the last braze cycle on unplated tubes.

Preliminary evaluation of the final-braze cycle difficulties, caused by aluminum oxide formation, revealed that the best results (filletting and flow) were obtained with nickel plating. The high fillet produced was also desirable for hand torch braze repair.

The preceding data and test results were reviewed during a meeting (among representatives of Pyromet, SNPO-C and Aerojet) at Pyromet on 26 July 1966. The difficulties were reviewed, test results from ORNL and Pyromet were analyzed (including quality of the atmosphere), and the various solutions thoroughly discussed. The agreed upon effort was to proceed with the nickel plating, using the Palcusil alloy as a back-up, and to conduct a qualitative experiment on the benefits of a titanium "getter" addition to the 30-in.-diameter retort furnace at Pyromet. Specifically the following plan of action was devised:

a. The remaining full-scale sector would be processed with one-half of the tubes nickel plated, using Nicoro-80 for the final braze cycle, and the other half of the tubes processed with the Palcusil-25 alloy for the final cycle.

b. The third 20-in. specimen would be brazed in Pyromet's 30-in. retort furnace with suitably distributed containers of titanium chips as an atmosphere contamination "getter."

The surface appearance of the third 20-in. specimen, cycled once through the 30-in. brazing furnace, was then compared with the first and second 20-in. specimens. A slight improvement in surface "brightness" was apparent. Based on this minor improvement, titanium chips were used as a "gettering" agent during all cycles of brazing with the second full scale sector. No difference was detectable in surface appearance of the sector at the completion of all the brazing cycles. Visual examination of the sector did reveal some Nicoro-80 wetting on the crowns of the nickel plated tubes.

Leak testing at 90-pounds pressure revealed one small leak (weeper) in the nickel plated tube joints and 33 leaks in the Palcusil-25 tube joints. The Palcusil-25 joints were re-alloyed and the sector subjected to another braze cycle. The subsequent leak test revealed only two small leaks in the Palcusil joints. The leak in the nickel plated tube joint, and the two leaks in the Palcusil joints, were all hand-torch repaired with BT alloy and the sector subjected to proof (1465 psi) and burst testing. Two small leaks were noted in the Palcusil joints after proof testing. The leaks were not repaired prior to burst testing, which was discontinued at 4300 psi because of excessive leakage between the end tubes and the side rails. No additional leaks were noted in any of the tube joints.

Based on the foregoing investigations, it was decided to nickel plate the first set of Phoebus-2 tubes and spacers at Model Plating and to conduct the brazing in accordance with AGC 90006 (Nicoro-80 on the final cycle).

3. Conventional Groove Machining

Conventional machining methods for grooving Hastelloy X material have been established. Preliminary machining variables data were established on a 60-in.-long Hastelloy X plate, using high carbon steel wetters, at Oakland Machine Co. The rates of feeds and cutter speeds were less than the feeds and speeds used for grooving AISI 347 stainless steel NERVA nozzle jackets. The final selection of machining variables for use on the Phoebus nozzles was established by Standard Tool and Die using the full length contoured Hastelloy X sector.

D. HEAT TRANSFER TUBES - THERMAL CYCLING TESTS

Previous thermal cycling tests with Hastelloy X tubing in the heat transfer facility were less than optimum, as reported previously (Report No. RP-DR-0003), because of defective tubing, stress-cracking and over-heating. To clarify the test results, a careful selection of an additional limited amount of Hastelloy X tubing was made. The tube material was inspected and metallurgically examined prior to fabrication into specimens. The tube material quality was as follows:

Source:	Tube Methods Inc.
Type and Size:	Welded and drawn, 0.190 in. OD x 0.020 in. wall
Chemistry:	Boron analysis not available, otherwise meets requirements of AGC-90057
Structure:	Uniform grain size less than ASTM 8 with scattered, small primary phase; grain size slightly coarser on OD. No evidence of weld. Acceptable.
NDT:	No outer surface defects and ID defects are less than 0.001 in. deep. Acceptable.

The tube samples were fabricated by brazing copper electrodes onto 12.0-in. lengths of tubing using Niore alloy (shims and powder) at a furnace temperature of 1810-1825°F in a hydrogen atmosphere followed by a second cycle at 1775°F to 1800°F and a third cycle at 1750°F to 1775°F. Cooling following the initial braze cycle from the braze temperature was equivalent to an air cool. The cooling rate was 100°F-per-hour for the second and third cycles. An additional tube without copper electrodes was also processed with the thermal cycling specimens. Tensile test results of this specimen are compared with those of 0.012-in. strip stock (brazed) as follows:

	<u>Tube</u>	<u>0.012-in. Strip (Average)</u>
UTS (ksi)	118.3	108.0
YS (ksi)	55.2	50.0
Elong (%)	22.0	25.0

The results compare favorably with the strip stock tested during the design properties program. The slightly higher tube strength reflects the lower annealing temperature commonly used in processing strip and tubing rather than the full 2150°F anneal originally employed on the 0.012-in. strip. Figures 21 and 22 exhibit the microstructure of the tubing in the "as-received" and braze-cycled conditions, respectively. The structures are essentially the same with the exception of an increase in the visible carbide precipitation in the brazed sample.

The tube samples were instrumented with four thermocouples and voltage taps restricted to the last 3 in. of the heated length, leaving the first 4 in. clear of any foreign material. Analysis of the wall temperature profile for this test section predicts that when the thermocouples located at axial distances of 5 in. and 6 in. read 1200°F, an outer wall temperature of 1600°F should occur approximately 2.0 in. from the inlet electrode. The nominal heat flux will be 18 Btu/in.²-sec with a drop of approximately 400°F through the wall. Figure 23 illustrates the tube sample in the thermal cycling fixture.

The tube samples were examined visually and with dye penetrant after every four cycles. The significant events were as follows:

<u>Tube Sample No. 1</u>		<u>Tube Sample No. 2</u>	
<u>Cycles</u>	<u>Observation</u>	<u>Cycles</u>	<u>Observation</u>
4	Tube bent 1 in. above base electrode. Slight penetrant indication noted.	4	Tube bent near mid section
		16	Tube bent 1 in. above base electrode.
8	Two surface rough spots in bend area.	20	Test discontinued
12	Three surface rough spots and faint lineal penetrant indication		
20	Surface roughness more extensive with faint penetrant indications. Test discontinued.

Figure 21

Photomicrographs of Hastelloy X Tubing Cross-Section at 100X (Top), 250X (Bottom) Showing the Microstructure Typical of the "As-Received" Condition. ASTM Grain Size 8. Etchant: Inco

Figure 22

Photomicrograph of Hastelloy X Tube Cross-Section at 250X Depicting
Typical Microstructure Caused by Simulated Braze Cycle. ASTM Grain
Grain Size 8 to 9. Etchant: Oxalic

Figure 23
Hastelloy X Sample No. 1, in Thermal Cycling Fixture

Sample No. 1 experienced bending in the lower portion of the test area. The deformation occurring approximately 1 in. above the base electrode was caused by the restricted thermal expansion of the tube. Figure 24 shows the bend position and angle of deflection as measured on a 10-square-to-the-inch grid. The shape of Sample No. 1 was identical to that of Sample No. 2 after the final test cycle. The change in bend configuration of the No. 2 tube during test is explained as a matter of probability. Some deflection and straightening occurs with each thermal cycle because of the restraint imposed by the test system. The ability of a tube to return to a given configuration after each cycle is due only to the weakened condition of the tube in that plane as a result of prior straining and the temperature.

Figure 25 (top) is a close-up view of the defects noted on Sample No. 1. The left side of the photo is that portion of the tube nearest the base electrode. Note the shallow pit defects (center) and the more pronounced textured pit (left). The pit on the left was the most recent, occurring on the twelfth cycle. The general smoothing of the pit surfaces accounted for the less pronounced dye penetrant indications recorded on the final inspection than those noted on earlier inspections of the sample (bottom).

No surface defects were noted on Sample No. 2, but bending occurred in two locations at different times as compared to the one bend which was experienced in repeated cycling of Sample No. 1. Although test procedures were the same, it is apparent that the stress fields were not.

Sample No. 1 was sectioned for metallurgical analysis in various sectors of the defect area. The tube at the defect area had increased slightly in diameter from 0.190 in. to 0.197 in. and had experienced a reduction in wall thickness from 0.020 in. to 0.018 in. The dimensional change was not uniform over-all because of the presence of the surface pitting approximately .002 in. deep. The expansion of the diameter was possibly because of the combined result of the compressive and bending forces exerted on the tube in thermal expansion.

Figure 24

Photograph at Approximately 3/4 Size Noting Position of Bend
in Hastelloy X Test Sample No. 1, Upon Completion of 20 Cycles.

Figure 25

Photograph at Approximately Ten Magnifications Showing Appearance of Surface Defects Incurred on Hastelloy X Tubing During Thermal Cycling. Top Photograph Shows Visual Appearance of Surface Pits; Bottom Shows Defects As Detected by Dye Penetrant Inspection

Figure 26 shows the microstructure of the Hastelloy X Sample No. 1 as sectioned through a surface pit and the area 180° away at the same level. Note that the inner diameter of the tube is significantly finer grained than the outer diameter. The transition in grain size did not show up well in the micrographs because of the difference in etching rate of the two sectors of the tube cross-section. The finer structure is identical to that of the original Hastelloy X tube exposed to the simulated braze cycle and is approximately an ASTM 8-9 grain size as compared with an ASTM 7 for the outer regions. Numerous areas of porosity were noted across the section of the tube in the area of surface pits (Figure 26 top). The sub-surface pores were large and few in number near the pits. A band of scattered, more numerous, smaller diameter pores marked the boundary of the grain size transition zone. The sub-surface porosity distorted the tube surface, indicating an internal expansion force. The presence of the porosity could change the electrical resistance heating characteristics of the material in that region and cause a localized increase in temperature. Figure 27 presents photomicrographs of a defect in the etched and unetched condition. Note the presence of an unidentified constituent in the larger porosity. The photographs exhibit a pitted surface cross-section and reveal no evidence of intergranular corrosion.

A chemical analysis was not conducted on the unidentified constituent. It appears, because of its low melting point and distribution, that the unidentified substance is not typical of the Hastelloy X alloy and probably was introduced as contamination during such processes as welding, deoxidation or desulfurization.

Figure 28 compares the tube sample ID surface before and after testing, revealing the increase in size of some of the original fissures (0.002-in.-deep).

In summary, the two tube samples satisfactorily completed twenty thermal cycles without cracking although some internal contamination was evident in tube No. 1.

Figure 26

Photomicrograph (100X) of Hastelloy X Tubing, Sample No. 1, Showing Tube Cross-Section of Area of Surface Defects (Top) and Area Directly Opposite (Bottom). Note the Variation in Grain Size and the Internal Porosity Between Areas. ASTM Grain Size 7 to 8. Etchant: Oxalic

✓

Figure 27

Photomicrographs (250X) of an Edge of a Surface Defect on Hastelloy X Tubing, Sample No. 1, in the Etched and Unetched Condition. Note the Unidentified Second Phase Present at Sites of Porosity. Also Note the Extent of Grain Growth Between the ID and OD portions of the Tube Cross-Section. Etchant: Oxalic

Figure 28

Photomicrographs (500X) of the ID of the Hastelloy X Tubing in the
Braze Condition (Top) and after Thermal Cycling (Bottom)

E. COOLANT TUBE METALLURGICAL EXAMINATION

A metallurgical investigation was performed on a Hastelloy X coolant tube produced by Straza Industries. The investigation consisted of microstructural, dimensional and hardness studies on samples from eight areas of the tube and a study of the effects of thermal treatment (simulated braze and annealing cycles per AGC-90071) on the test samples. Microstructural study and hardness determinations were also performed on a Hastelloy X strip (AGC-90057) sample representing the coolant tube raw stock.

The Hastelloy X coolant tube was metallurgically examined in accordance with the testing plan outlined below:

Phase A - Metallurgical analysis of "as-formed" tube

- 1 Microstructure examination of six tube sections (see Figure 29) at 100 and 250 X in the transverse orientation.
- 2 Microhardness survey of the tube sections.
- 3 Thickness measurement of the tube sections.
- 4 Microstructure examination of two welded endcap sections (see Figure 29) at 100 and 250 X in the longitudinal orientation.

Phase B - Study of the effect of a simulated braze cycle on the "as-formed" tube

- 1 Microstructure examination of six tube section samples (see Figure 29) at 100 and 250 X in the transverse orientation.
- 2 Microhardness survey on above tube section samples.
- 3 Microstructure examination of two welded endcap tube section samples (see Figure 29) at 100 and 250 X in the longitudinal orientation.

Phase C - Study of the effect of an annealing cycle and annealing-plus-simulated braze cycle on the following changes in the "as-formed" tube

Figure 29
Specimen Test Location, Hastelloy X Coolant Tube

- 1 Dimensional change.
- 2 Microstructural change.
- 3 Hardness change.
- 4 Annealing per AGC-90071.

Cycle A 1925°F/5 minutes in H₂, fast cool

Cycle B 1925°F/15 minutes in H₂, fast cool

The simulated braze cycle employed consisted of the following:

Cycle 1 Heat-up to 1830°F $\pm$ 20°F in 3 to 4 hours

Hold at 1830°F $\pm$ 20°F for one hour

Cool 100°F/hour to 975°F

Cycle 2 Heat-up to 1800°F $\pm$ 20°F

Hold one hour at 1800°F $\pm$ 20°F

Cool 100°F/hour to 975°F

Cycle 3 Heat-up to 1775°F $\pm$ 20°F

Hold one hour at 1775°F

Cool 100°F/hour to room temperature

Additionally, a strip of Hastelloy X (Heat 260-5-2813) representing the raw stock used in the coolant tube was micro-examined for structure and was also microhardness tested. These results are included under Phase A.

Phase A - Metallurgical analysis of "as-formed" tube and raw stock strip samples

The microstructures of the coolant tube sections and the raw stock strip samples are shown in Figures 30 and 31, respectively. The coolant tube samples had a predominant grain size of ASTM No. 7, with a range from ASTM No. 6 to 8. There were no significant differences in grain size among the samples taken from the six locations. There was evidence of considerable grain twinning (in all samples) because of cold work from tube forming and carry-over from the raw stock strip.

Tube Section 1-1

100X

Tube Section 1-1

250X

Tube Section 2-1

100X

Tube Section 2-1

250X

Figure 30 (Sheet 1 of 3)

Microstructure of Tube Sections in "As-Formed" Condition.

Etchant: 10% Oxalic, Electrolytic

Tube Section 3-1

100X

Tube Section 3-1

250X

Tube Section 4-7

100X

Tube Section 4-7

250X

Figure 30 (Sheet 2 of 3)

Microstructure of Tube Sections in "As-Formed" Condition.
Etchant: 10% Oxalic, Electrolytic

Tube Section 4A-7

100X

Tube Section 4A-7

250X

Tube Section 5A-0

100X

Tube Section 5A-0

250X

Figure 30 (Sheet 3 of 3)

Microstructure of Tube Sections in "As-Formed" Condition.

Etchant: 10% Oxalic, Electrolytic

TRANSVERSE ORIENTATION
MAGNIFICATION: 100X

TRANSVERSE ORIENTATION
MAGNIFICATION: 250X

LONGITUDINAL ORIENTATION
MAGNIFICATION: 100X

LONGITUDINAL ORIENTATION
MAGNIFICATION: 250X

Figure 31
Microstructure of Hastelloy X Rawstock Strip, Heat 260-5-2813

The raw stock strip sample (Figure 31), like the coolant tube samples described above, had a predominant grain size of ASTM No. 7, with a range of ASTM No. 6 to 8. The longitudinal section showed evidence of primary phase segregation in the matrix. This condition was not evident in the transverse section or in the transversely mounted coolant tube samples. Considerable grain twinning was also evident.

The grain size of the tube and strip samples was essentially equivalent. Also, the general morphology was similar; however, in several cases, the tube samples appeared to have areas of denser carbide precipitation than in the strip samples. Compare Figures 30 and 31. The grain boundaries of the tube samples, in several instances, are exaggerated due to over-etching with oxalic acid.

Hardnesses ranged from knoop hardness number (KHN) 204 to 325 (Rockwell B 90.5 to Rockwell C 32) on the coolant tube samples and were generally higher at the tube "crown" areas (KHN 265 average) than at the "leg" areas (KHN 230 average). Samples from the throat radius region had the highest hardness. Hardness of the legs were generally consistent in all samples.

Hardnesses ranged from KHN 230 to 241 (Rockwell B 96 to 98) for the raw stock strip-sample.

The wall thickness survey on the coolant tube is shown in Table 27. Thickness ranged from 0.0116 to 0.0124-in. Values were obtained using a measuring microscope.

The microstructure of a typical endcap weld is shown in Figure 32.

Phase B - Study of the effect of a simulated braze cycle on the "as-formed" tube

The microstructures of the coolant tube sections, after exposure to the braze cycle, are shown in Figure 33. The predominant grain size was ASTM No. 7. The grain size ranged from ASTM No. 6 to 8. Compared to the "as-formed" tube (Figure 30),

TABLE 27

WALL THICKNESS SURVEY: AS - FORMED TUBE CONDITION*

TUBE SPECIMEN	THICKNESS (INCH)		
	A	B	C
1-1	0.0120	0.0120	0.0120
2-1	0.0120	0.0120	0.0124
3-1	0.0120	0.0120	0.0120
4-1	0.0124	0.0122	0.0120
4A-7	0.0116	0.0122	0.0120
5A-6	0.0116	0.0116	0.0116

* Thickness measurements taken at areas shown in sketch below.

Figure 32

Microstructure of Welded End Cap Section 6-1 in "As-Formed" Condition.
 Etchant: 10% Oxalic, Electrolytic Magnification: 100X Upper, 250X Lower

Tube Section 1-2

100X

Tube Section 1-2

250X

Tube Section 2-2

100X

Tube Section 2-2

250X

Figure 33 (Sheet 1 of 3)

Microstructure of Tube Sections After Simulated Braze Cycle.
Etchant: 10% Oxalic, Electrolytic

Tube section 3-2

100X

Tube Section 3-2

250X

Tube Section 4-2

Tube Section 4-2

250X

Figure 33 (Sheet 2 of 3)

Microstructure of Tube Sections After Simulated Braze Cycle.
Etchant: 10% Oxalic, Electrolytic

Tube Section 4A-2

100X

Tube Section 4A-2

250X

Tube Section 5A-2

100X

Tube Section 5A-2

250X

Figure 33 (Sheet 3 of 3)

Microstructure of Tube Sections After Simulated Braze Cycle.

Etchant: 10% Oxalic, Electrolytic

the braze cycle exposed tube contained a greater amount of the carbide (M_6C) precipitate in the matrix and especially at the grain boundaries. The somewhat reduced amount of twinning in the grains indicates an annealing effect.

The microhardness survey indicated that the simulated braze cycle caused an increase in the hardness of the leg area (because of carbide precipitation) from KHN 228 to KHN 250, while a slight decrease, KHN 265 to KHN 242, occurred at the crown.

The microstructure of the welded endcap after the brazing cycle is shown in Figure 34.

Phase C - Study of the effect of an annealing cycle and annealing-plus-simulated braze cycle on the "as-formed" tube

The H and ID dimensions of the U-tube samples before and after the 1925° minute anneal is shown in Table 28. The annealing cycle caused no significant change in the tube dimensions. The only large difference was noted in one ID dimension (Specimen 3-4) wherein the maximum variation was about 0.011-in.

Typical microstructures of the coolant tube sections after annealing at 1925° and after annealing plus the braze cycle treatment are shown in Figure 35. The annealed and annealed-plus-braze-cycle-treated samples contained less precipitated carbide than the direct braze cycle treated samples (Figure 33). The annealed-plus-braze-cycle samples contained more precipitate within the grain boundary than the annealed only samples. There was no grain size difference compared to the "as-formed" samples (Figure 30). Note, however, the complete removal of grain twinning.

100X

250X

Figure 34
Microstructure of Welded End Cap Section 6-2 after Simulated
Braze Cycle. Etchant: 10% Oxalic, Electrolytic

TABLE 28

EFFECT OF ANNEALING CYCLE* ON TUBE CROSS SECTION DIMENSIONS AND LOCATION

<u>SPECIMEN</u>	<u>MEASUREMENT BEFORE</u>		<u>MEASUREMENT AFTER</u>	
	<u>H</u>	<u>ID</u>	<u>H</u>	<u>ID</u>
1-3	0.430	0.656	0.430	0.656
1-4	0.431	0.658	0.430	0.657
1-5	0.425	0.693	0.427	0.693
1-6	0.428	0.675	0.428	0.674
2-3	0.508	0.508	0.510	0.577
2-4	0.516	0.570	0.516	0.569
2-5	0.516	0.570	0.517	0.569
2-6	0.512	0.572	0.514	0.567
3-3	0.519	0.432	0.519	0.433
3-4	0.519	0.420	0.518	0.431
3-5	0.516	0.415	0.516	0.419
3-6	0.512	0.430	0.512	0.431
4-3	0.520	0.153	0.520	0.146
4-4	0.523	0.160	0.524	0.160
4-5	0.527	0.148	0.528	0.148
4-6	0.528	0.107	0.529	0.108
4A-3	0.525	0.150	0.526	0.148
4A-4	0.518	0.135	0.519	0.135
4A-5	0.523	0.149	0.524	0.150
4A-6	0.525	0.146	0.526	0.146
5A-3	0.600	0.210	0.600	0.208
5A-4	0.606	0.225	0.606	0.224
5A-5	0.608	0.225	0.610	0.225
5A-6	0.618	0.193	0.618	0.187

*5 minutes at 1925°F, aircool

Tube Section 4-3
Annealed 5 min @ 1925°F

100X

Tube Section 4-3

250X

Tube Section 4-4
Annealed 5 min @ 1925°F + Braze Cycle

100X

Tube Section 4-4

250X

Figure 35

Microstructure of Tube Sections 4-3 and 4-4 in the Annealed and
Annealed Plus Simulated Braze Conditions. Etchant: 10% Oxalic,
Electrolytic

The microhardness survey revealed small changes in hardness for the 1925°F annealing treatment with a slight decrease on the crown and a larger increase on the leg. A small additional increase in hardness occurred for both crown and leg when the braze cycle was imposed upon the annealed condition. Summarized, the average hardness values were as follows:

<u>Condition</u>	<u>Hardness (KHN)</u>	
	<u>Crown</u>	<u>Leg</u>
As-formed	265	228
5 min./1925°F	266	260
15 min./1925°F	262	261
5 min./1925°F Plus Braze Cycle	268	268
15 min/1925°F Plus Braze Cycle	273	267

The annealing treatment served to promote uniformity of hardness (equalization) by stress relief with the concomitant increase in hardness caused by a precipitation age hardening reaction.

The average KHN microhardness values for the various tube locations and for the leg and crown areas are summarized in Table 29. In addition to the changes in hardness with the thermal cycle and location in the tube, the variation in total spread of the hardness values is also apparent from these data. Notice that the great variation in the "as-formed" crown areas, 102 KHN, was reduced to a more consistent value after the thermal treatments. The narrow spread for the original stock is reflected also in the "as-formed" leg values, but following the thermal treatments the leg hardness variation increased to within the approximate spread limits as the heat treated crown values. The effects of the braze cycle and/or the 1925°F treatments produced a stress-relieved material that responded to precipitation quite uniformly.

TABLE 29
AVERAGE MICROHARDNESS (KNOOP) COMPARISONS ON
HASTELLOY X COOLANT TUBE

SAMPLE LOC.	AS FORMED		AS FORMED +		AS FORMED +		AS FORMED 5 MIN @ 1925°F +		AS FORMED 15 MIN @ 1925°F +		AS FORMED 15 MIN @ 1925°F +	
	LEG	CROWN	BRAZE LEG	CYCLE CROWN	5 MIN @ 1925°F LEG	CROWN	BRAZE LEG	CYCLE CROWN	15 MIN @ 1925°F LEG	CROWN	BRAZE LEG	CYCLE CROWN
1	230	251	250	239	255	260	270	266	245	251	257	266
2	231	272	285	261	276	266	285	269	278	254	284	272
3	229	204	237	236	252	260	264	264	260	266	263	269
4	233	274	245	246	259	285	264	273	270	274	268	294
4A	223	306	239	237	270	273	276	265	259	270	276	261
5	221	284	242	232	245	254	247	271	252	255	252	277
AVERAGE	228	265	250	242	260	266	268	268	261	262	267	273
SPREAD-TOTAL	12	102	48	25	31	31	38	9	33	23	32	35
PLUS	5	41	35	19	16	19	17	5	17	12	17	23
MINUS	7	61	13	10	15	12	21	4	16	11	15	12

ORIGINAL MATERIAL (AGC-90057): $236 \pm \frac{5}{6}$

This investigation has shown that a hardness increase occurred after the various thermal cycles. These results are compatible with the general property trends found by Comprelli and Wold (Reference (1)), who found substantial increases in the strength (or hardness) with exposures at 1050°F. The increase of hardness was accompanied by a decrease in ductility. The hardness increase has been observed also by Kaufmann (Reference (2)), and corroborated by Wagoner (Reference (3)). The increase in hardness is attributed to precipitation producing a strengthened matrix. Phase M_6C is believed to be the major precipitate conferring increased hardness; however, the existence of other phases, $M_{23}C_6$, Laves phase, and Mu phase cannot be discounted. In the case of annealing, further contribution to strengthening is believed to be caused by solutioning of grain boundary carbides and subsequent reprecipitation within the matrix.

In summary, this investigation revealed the following:

1. Both the "as-formed" coolant tube and the Hastelloy X strip-sample were fine grained with the predominant grain size being ASTM No. 7. Grain size range was generally ASTM No. 6 to 8.
2. The simulated brazing, annealing, and annealing-plus-brazing cycles did not promote grain growth in the "as-formed" coolant tube.
3. The simulated brazing and annealing-plus-brazing cycles promoted carbide precipitation and produced a hardness increase in the coolant tubes.

Reference (1) Metals Engineering Quarterly, November 1965, "Stability of High-Nickel Alloys in Superheated Steam," F A Comprelli and V E Wolff.

Reference (2) Thomson Engineering Laboratory Report R62SE17, November 1963, General Electric Company, Phase Studies - Miscellaneous Alloys, M Kaufmann.

Reference (3) E L Wagoner, Physical Metallurgy and Mechanical Properties of Hastelloy Alloy X, Haynes Stellite Publication, June 23, 1961.

4. The simulated annealing-plus-brazing cycle produced slightly higher hardnesses than either annealing or brazing alone.

5. The annealing treatment produced higher hardnesses than the simulated brazing cycle. The annealing treatment prior to the braze cycle reduced the amount of carbides precipitated at the grain boundaries.

6. A partial stress-relief or stress-equalizing of the cold worked Hastelloy X was achieved by the brazing cycle, annealing and annealing-plus-simulated-brazing cycles.

7. The "as-formed" tube samples from the throat radii region exhibited a hardness gradient from stretch forming; viz., cold-working. Maximum hardness was observed at the crown of the tubes.

8. The annealing cycles caused no significant changes in the H (height) and ID (inside diameter) dimensions of the "as-formed" tubes.

9. In view of the cost and risks, because of possible longitudinal distortion of the long non-rigid tube, the 1925°F annealing treatment is not considered to be advisable. Adequate stress-relief is accomplished during the brazing cycle.

F. MATERIAL PROCUREMENT AND NOZZLE PROCUREMENT SUPPORT

1. Development Materials Procurement

a. Hastelloy X

All Hastelloy X material required for the development programs has been received. These materials have been utilized in the design properties, brazing and welding development tasks.

b. INOR-8

INOR-8 development material is presently being stored at Aerojet in a bonded area, except for the .012-in.- and .008-in.-thick strip and the weld wire. These items were cancelled, with SNPO-C's approval, because of conversion difficulties at Allvac. No further reporting will be conducted on this material.

c. Allvac 625

Material delivery status of Allvac 625 is shown in Table 30. All items received to date have been placed in a bonded storage area. The outstanding items have delivery dates as noted and are based on Supplier's Information Requests (SIR) No. 10835 and 10549, approved by AGC. SIR 10835 allows an annealing temperature of 1825° plus/minus 25°F on the strip material. Tensile test results indicated that the 2150°F annealing temperature, in accordance with AGC-90057, resulted in material that failed to meet minimum yield strength requirements, while annealing at 1825°F adequately met the specification values. Supplier's Information Request No. 10549 allows Allvac to waive paragraph 4.3.2 (Weldability Tests) of AGC-90058B (Weld Wire) and proceed based on compliance to Weld Performance Testing (Reference paragraph 4.3.4 through 4.3.4.4 of AGC-90058B). Monitoring of the Allvac 625 procurement will continue until all deliveries are complete.

TABLE 30

PHOEBUS-2 MATERIAL DEVELOPMENT PROCUREMENT OF ALLVAC 625

<u>ITEM</u>	<u>SIZE (IN.)</u>	<u>QUANTITY</u>	<u>HEAT NO.</u>	<u>P.O.</u>	<u>STATUS</u>
Weld Wire	0.062 dia.	75 lbs.	3 heats	201270	Delivery scheduled 10-15
Bar	0.750 dia.	150 ft.	5940	201270	Procurement complete
Plate	1-1/2 x 12 x 36	9	5940, 5941, 5942	201270	Procurement complete
Sheet	0.050 x 12 x 48	1	5940	201270	Procurement complete
Strip	0.012 x 8 x 30	3	3 heats 5940 received	201270	Delivery scheduled 8-29
Plate	0.187 x 2 x 60	1	5940	201878	Procurement complete
Strip	0.012 x 2 x 60	9	1 heat	201878	Delivery scheduled 8-29
Strip	0.008 x 3 x 60	1	1 heat	201878	Delivery scheduled 8-29

Successful weldability tests in accordance with the requirements of AGC-90059 were completed in March 1966, on the Allvac 625 material from Heats 5940, 5941, and 5942. The weldability tests were conducted by Metcut Research Associates, Inc., Cincinnati, Ohio, under the surveillance of a Phoebus Materials Representative. Inconel 625 air melt filler wire, Heat No. N-X-9648A, produced by Huntington Alloy Products Division, International Nickel Company, and supplied by the Oak Ridge National Laboratory, was used as the weld wire for the weldability tests. With the exception of Heat 5940, specimen -3, no defects were found in metallographic examination of the welded specimens. The -3 specimen of Heat 5940 exhibited an elongated void-type defect .050-in.-long and .015-in.-wide which extended from the weld deposit to the interface of the parent metal. This was considered a borderline condition of no major significance, possibly caused by impurities contained in the air melt filler wire, and the material heat was accepted as meeting the requirements of AGC-90059.

2. Nozzle Procurement Support

a. Hastelloy X Ingot and Forging

All forging of Phoebus-2 nozzle components has been completed. Machine components for all nozzles, S/N's 880600 through 880606, have been received. Welding has been completed on S/N's 880600 through 880605.

The identification of each nozzle by serial number, forging part number, and material heat number is presented in the following:

Nozzle Serial No.	<u>Forging Material Heat Number</u>			
	<u>P/N 1114856-13 (Inlet Ring)</u>	<u>P/N 1114856-7 (Convergent)</u>	<u>P/N 1114856-11 (Divergent)</u>	<u>P/N 1114856-15 (Pressure Test) Convergent</u>
880600	260-5-2793	260-5-2793	260-5-2850	
601	260-5-2793	260-5-2863	260-5-2850	
602	260-502793			260-5-2820
603	260-6-2901	260-5-2880	260-5-2887	
604	260-6-2901	260-5-2880	260-5-2887	
605	260-6-2901	260-6-2900	260-6-2920	
880606	260-6-2935	260-6-2898	260-6-2898	

Test samples were obtained from every forging for tensile evaluation in the annealed condition (2150°F for one hour and then fan cooled). The specimens were oriented tangentially in the forging. The average test results from the annealed specimens are shown in Table 31 by heat number. The uniformity of the 0.2% offset yield strength (YS) is readily apparent. The variation in the ultimate tensile strength (UTS) and elongation is greater than the yield strength, with the actual data variance as follows for all heats of material:

TABLE 31
AVERAGE TENSILE PROPERTIES OF ANNEALED* HASTELLOY X FORGINGS

<u>HEAT NUMBER</u>	<u>TEST TEMP (°F)</u>	<u>UTS (KSI)</u>	<u>YS (KSI)</u>	<u>ELONG. (%)</u>	<u>NO. OF TESTS</u>
260-5-2793	Room	95.8	41.5	49	19
260-5-2820	"	100.5	42.7	52	8
260-5-2850	"	86.3	40.3	28	8
260-5-2863	"	101.7	42.2	45	4
260-5-2880	"	104.5	42.6	52	12
260-5-2887	"	101.4	44.7	34	9
260-6-2898	"	98.5	43.8	38	11
260-6-2900	"	104.5	45.4	41	8
260-6-2901	"	109.3	45.7	39	20
260-6-2920	"	96.1	42.1	41	4
260-6-2935	"	103.7	45.0	32	4
Average	Room	100.2	43.3	41	
260-5-2793	1200	66.0	26.5	53	11
260-5-2820	"	75.4	26.2	51	2
260-5-2850	"	63.8	24.9	28	2
260-5-2863	"	73.5	27.8	45	1
260-5-2880	"	76.3	26.0	50	4
260-5-2887	"	75.9	28.5	42	2
260-6-2898	"	71.3	27.9	38	3
260-6-2900	"	75.3	29.4	37	2
260-6-2901	"	78.5	28.8	40	3
260-6-2920	"	72.3	26.4	47	1
260-6-2935		Not available			
Average	1200	72.8	27.2	43	

* 2150°F for 1 hour, then fan cooled to room temperature

Temp. (°F)	UTS (ksi)			YS (ksi)			ELONG (%)		
	<u>Average</u>	<u>Min.</u>	<u>Max.</u>	<u>Average</u>	<u>Min.</u>	<u>Max.</u>	<u>Average</u>	<u>Min.</u>	<u>Max.</u>
Room	100.2	80.3	112.8	43.3	39.2	52.0	41	22	58
1200	72.8	61.7	82.9	27.2	24.2	32.1	43	24	56

Specification AGC-90056

Room	85.0	40.0	40
1200	60.0	25.0	20

Several test results reveal requirements below the minimum of the forging, Specification AGC-90056. The location and causes of these discrepancies can be ascertained by analysis of the test results by forging component, as follows:

Part No. <u>Name</u>	No. of <u>Pieces</u>	No. of <u>Heats</u>	Test Temp (°F)	UTS (ksi)			YS (ksi)			ELONG. %		
				<u>Aver.</u>	<u>Min.</u>	<u>Max.</u>	<u>Aver.</u>	<u>Min.</u>	<u>Max.</u>	<u>Aver.</u>	<u>Min.</u>	<u>Max.</u>
111425- 13 Inlet Ring	7	3	Room	104.4	92.0	112.8	44.2	41.0	47.4	43	28*	55
			1200	70.5	61.7	82.9	26.3	24.5+	29.6	48	27	56
111425- 7 Converg.	7	6	Room	101.9	93.2	107.0	43.1	40.0	52.0	48	36**	58
			1200	72.8	64.3++	79.5	27.0	25.4	32.1	47	33	55
111435- 11 Diverg.	6	4	Room	93.2	80.2	104.6	42.3	39.2	46.0	33	22***	46
			1200	69.2	62.2	76.6	26.7	24.2	28.6	36	24	47

***Only 3 were 40% or greater

** 4 were below 40%

* 12 were below 40% with 7 values at 37% or higher

+ All from heat 260-5-2793

++ 2 below 65 ksi from heat
260-5-2793

The inlet rings, as a group, met specification requirements at room temperature except for some elongation test results confined to heats 260-6-2901 and 260-6-2935 which averaged considerably higher tensile strengths and, therefore, lower elongation than heat 260-5-2793. At 1200°F the inlet rings, collectively, did not meet the minimum UTS and YS, which were confined primarily to parts from heat 260-5-2793, consistently lower in strength than heats 260-6-2901 and 260-6-2935. The predominant stress direction for the inlet rings is compression.

The convergent section forgings met all requirements except for four tests which failed to comply with the room temperature minimum elongation requirement of 40%, and two tests for heat 260-5-2793 which were just below the minimum YS at 1200°F.

The divergent section forgings, collectively, varied from the requirements, with low UTS and YS at room temperature and 1200°F and low elongation at room temperature. These results occurred because of the location of the test material within the forging; that is, the test material was removed from the smaller diameter which had received less hot work during forging and contained un-solutioned carbide stringers oriented transverse to the specimen axis. The stress levels in the divergent section are low.

To evaluate the strength values of the foregoing annealed forgings, several samples were processed through the simulated brazing cycle for comparison with the data obtained in the design properties phase of the development program. The averaged data are presented in Table 32. The effect of the brazing cycle (to increase the UTS and YS and decrease the elongation) is evident. The results indicate a small reduction in the data spread for the brazed condition and good correlation with the design values of the nozzle forgings obtained in the design properties program.

b. Improved Hastelloy X Forgings

The three forging components employed to produce the nozzle jacket consist of the following:

TABLE 32

COMPARISON OF PROPERTIES OF ANNEALED AND ANNEALED-AND-BRAZED
HASTELLOY X FORGINGS

Heat Number	<u>Room Temperature</u>						<u>1200°F</u>					
	UTS (KSI)		YS (KSI)		ELONG (%)		UTS (KSI)		YS (KSI)		ELONG (%)	
	Ann'd	Brazed	Ann'd	Brazed	Ann'd	Brazed	Ann'd	Brazed	Ann'd	Brazed	Ann'd	Brazed
260-5-2793	95.8	103.3	41.5	44.1	49	25	66.0	79.7	26.5	27.9	53	35
260-5-2820	100.5	111.7	42.7	44.6	52	29	75.4	87.0	26.2	29.1	51	35
260-5-2863	101.7	111.3	42.2	46.5	45	30	73.5	86.1	27.8	29.5	45	34
260-6-2901	109.3	115.1	45.7	44.8	39	37	78.5	90.2	28.8	30.0	40	39
Average	101.8	110.4	43.0	45.0	46	30	73.4	85.8	27.3	29.1	47	36
Mean												
Design Values:												
Total		93.3		45.0		24		74.8		26.8		31
Tangential		97.0		44.7		30		77.2		26.7		37

<u>Component</u>	<u>Approximate Size</u>
Inlet Ring	Right cylinder with 65-in.-dia x 14-in. long x 3-in. thick
Convergent Section	Cone with 72-in. max. dia x 33-in. long x 2-1/2-in. min. thick
Divergent Section	Cone with 53-in. max dia x 46-in. long x 3-in. min. thick

The inlet rings were produced from 14-in. billet stock by ring-rolling. The convergent and divergent sections were the largest and heaviest closed-die forging of Hastelloy X made to date, and they were produced directly from cast ingot to controlled chemistry (AGC-90067) with the following minimum tensile properties required in the 2150°F solution-annealed and fan-cooled condition:

	<u>Room Temp.</u>	<u>1200°F</u>
UTS (ksi)	85.0	60.0
0.2% YS (ksi)	40.0	25.0
Elong. (%)	40	20

In actuality, the following average tangential properties were obtained for the annealed closed die forgings:

	<u>FORGING SECTION</u>			
	<u>CONVERGENT</u>		<u>DIVERGENT</u>	
	<u>Room</u>	<u>1200°F</u>	<u>Room</u>	<u>1200°F</u>
UTS (ksi)	101.9	72.8	93.2	69.2
0.2% YS (ksi)	43.1	27.0	42.3	26.7
Elong. (%)	48	47	33	36

The following values were obtained from the rolled inlet rings:

	<u>Room</u>	<u>1200°F</u>
UTS (ksi)	104.4	70.5
0.2 YS (ksi)	44.2	26.3
Elong. (%)	43	48

With the exception of a few results, collectively, the forgings readily met the tensile strength specification requirements at room temperature and 1200°F. The yield strength values, for practical purposes, all met the specification requirements at room temperature (40.0 ksi) and 1200°F (25.0 ksi), although the average values for the die forgings were a mere 8% above the minimums. The elongation values reflect greater variance and, although the 20% minimum was readily attained at 1200°F, there were a number of test results below the 40% minimum for room temperature.

The results obtained were acceptable from both the engineering design and functional basis, but improvement and assurance that all forgings would exceed the minimum strength criteria were product improvement goals. Analysis of the forging practice, the experience accumulated, and the observed large grain size (ASTM 1 to 4) resulted in an optimistic plan to improve the forging strength by revision to the forging schedule and procedures. The program was undertaken by Wymark-Randall utilizing a 4-in.-thick slice of a 24-in.-diameter production ingot (heat number 260-5-2863) and a piece of a large ring of excess material machined from a partially processed Phoebus-2 forging (P/N 1114856-11, heat number 260-5-2863).

The program was directed toward providing a finer grain size, with concomitant improved strength, through controlled final forging or restrike reductions, forging temperatures, and post-forge heat treatment (PFHT). The conditions of temperatures and reductions were selected based on actual forge practice capabilities for Phoebus nozzle forging sizes and were consistent with acceptable metallurgical practice for Hastelloy X. The condensed and simplified results are presented in Figure 36 for the annealed ingot upset specimens and the forged ring section specimens. Figure 36 presents all the test data for the yield strength and the tensile elongation plotted against grain size. The trends are obvious, and the advantages and disadvantages of the finer grain sizes are noted, respectively, by the increasing yield strength and decreasing elongation. An ASTM grain size of 6 appears as a desirable and practicable value indicating a probable minimum yield strength of 45 ksi combined with a useful 28% elongation. Specifically, the yield strength of the forgings can be improved by:

Figure 36

Relationship of 0.2% Yield Strength and Elongation to ASTM Grain Size
for Upset Ingot Specimens Plus Forged Ring Specimens

(1) Decreasing the final forging temperature to maintain the benefits of hot strain by minimizing the annealing and grain growth on the forge.

(2) Decreasing PFHT to minimize the removal of the benefits of hot strain due to annealing and grain growth.

(3) Increasing final reduction to introduce more hot strain for recrystallization and grain refinement.

The benefits of the improved forging are also retained at 1200°F. The following table illustrates the tensile properties range at 1200°F for samples with an ASTM grain size of No. 5 or finer:

<u>UTS (ksi)</u>		<u>YS (ksi)</u>		<u>ELONG. (%)</u>	
<u>Average</u>	<u>Minimum</u>	<u>Average</u>	<u>Minimum</u>	<u>Average</u>	<u>Minimum</u>
77.7	75.8	32.4	31.7	37	33

Sample pancake forgings (approximately 1-in.-thick by 10-in.-diameter) were provided to Aerojet for exposure to the braze cycle and tensile-testing at room temperature and -320°F. The brazing cycle employed was known to reduce the tensile elongation of Hastelloy X. This effect was considered especially significant at -320°F with the reduction in elongation as the grain size decreases.

The tensile properties of the sample pancake forgings produced by Wyman-Gordon under the improvement program and exposed to the brazing cycle treatment were as follows:

<u>Temp (°F)</u>	<u>UTS (ksi)</u>		<u>YS (ksi)</u>		<u>ELONGATION (%)</u>	
	<u>Average</u>	<u>Minimum</u>	<u>Average</u>	<u>Minimum</u>	<u>Average</u>	<u>Minimum</u>
-320	152.5	142.9	79.7	77.0	26	16
Room	111.9	108.8	49.5	46.7	29	26

The foregoing values are to be compared with the following Hastelloy X properties obtained from cross forged, rolled rings (20-in. OD x 17-in. ID x 6-in. long) processed through the brazing cycle:

<u>Temp (°F)</u>	<u>UTS (ksi)</u>		<u>YS (ksi)</u>		<u>ELONGATION (%)</u>	
	<u>Mean</u>	<u>Minus 3-Sigma</u>	<u>Mean</u>	<u>Minus 3-Sigma</u>	<u>Mean</u>	<u>Minus 3-Sigma</u>
-320	117.4	106.4	71.7	65.1	13.1	7.5
Room	93.3	85.0	45.0	38.4	24.4	18.8

The significant differences between the improved forgings and previous forging are manifest. The yield strength and tensile strength are distinctly improved, but the prominent feature is the tensile elongation. The tensile ductility for the improved forgings in the annealed condition averaged 31% for the upset ingot specimens (36% for forged-ring specimens) and only decreased to 29% in the braze cycle, heat treated condition at room temperature and 26% at -320°F. The original Hastelloy X forgings decreased from an annealed ductility of 41% at room temperature to 24% after brazing, which further decreased to 13% at -320°F.

The application of this technology for subsequent procurement of large forgings is recommended, with the following target requirements for the specification tensile properties

	<u>Room Temperature</u>	<u>1200°F</u>
UTS (ksi)	85.0	65.0
YS (ksi)	45.0	29.0
ELONG. (%)	28	30