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SACRAMENTO PLANT

REPORT NO. RP-TR-0010
TO
SPACE NUCLEAR PROPULSION OFFICE - CLEVELAND EXTENSION

DEVELOPMENT OF IMPROVED STRENGTH
LARGE HASTELLOY X FORGINGS

MASTER

AUGUST 1966

CONTRACT SNPC-35

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ROCKET ENGINE OPERATIONS - NUCLEAR

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CLASSIFICATION CATEGORY

UNCLASSIFIED

CLASSIFYING OFFICER

DATE

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5

FOREWORD

This document, published in response to and in partial fulfillment of Task K, Article I, Statement of Work of Contract SNPC-35, covers nozzle forging investigations conducted in order to define those conditions which will produce forgings of optimum grain size and properties. The forging effort was conducted by Wyman-Gordon who also performed testing in the annealed condition. (Copies of the report by Wyman-Gordon are being submitted to SNPO-C only, and will appear as Appendix I). Aerojet conducted tests at -320°F and room temperature and prepared this final report.

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TABLE OF CONTENTS

	<u>Page</u>
I. SUMMARY	1
II. CONCLUSIONS	1
III. RECOMMENDATIONS	2
IV. DEVELOPMENT PROGRAM	2
A. Introduction	2
B. Test Results and Discussion	5
APPENDIX I - REPORT NO. RD 66-125, M.D. & E. #204	17
APPENDIX I TRANSMITTED TO SNPO-C ONLY	18

LIST OF TABLES

I	TENSILE PROPERTIES OF HASTELLOY X PANCAKE FORGINGS MADE FROM CAST INGOT (HEAT 260-5-2863) (ALL SPECIMENS EXPOSED TO PHOEBUS-2 BRAZE CYCLE)	10
II	TENSILE PROPERTIES OF HASTELLOY X PANCAKE FORGINGS MADE FROM CAST INGOT (HEAT 260-5-2863) (ALL SPECIMENS ANNEALED AT 2000°F)	11

LIST OF FIGURES

1	COMPARISON OF TENSILE PROPERTIES OF HASTELLOY X NOZZLE JACKET FORGINGS IN THE SOLUTION ANNEALED CONDITION	12
2	INGOT UPSET SPECIMENS - FORGE CYCLE ANALYSIS	13
3	FORGED RING SECTION - FORGE CYCLE ANALYSIS	14
4	UPSET INGOT SPECIMENS + FORGED RING SPECIMENS RELATIONSHIP OF 0.2% YIELD STRENGTH AND ELONGATION TO ASTM GRAIN SIZE	15
5	INTER-RELATIONSHIP OF FINISH FORGING TEMPERATURE, REDUCTION AND PFHT	16

I. SUMMARY

The 7000- to 8000-lb size of the Phoebus-2 nozzle, Hastelloy X, closed die forgings basically exceeded the current state-of-the-art for the process and the starting material. Nevertheless, 24-in. diameter, consumable-electrode, vacuum-melted ingots were produced and were forged directly into the desired closed die configurations. The forgings were produced to meet the minimum room temperature tensile requirements of 85.0 KSI tensile strength, 40.0 KSI 0.2% offset yield strength, and 40% elongation.

With product improvement as a goal, a development program was undertaken to demonstrate the practicability of improving the strength of these large forgings and to assess the effects on low temperature (-320°F) ductility. The program demonstrated that, remaining within the practical shop limitations for forgings of this size, the grain size could be controlled and enhanced strength attained. Some decrease in the room temperature minimum tensile ductility occurred in the annealed condition and exposure to the nozzle brazing cycle produced only a minor change. Testing at -320°F revealed a considerable improvement in tensile ductility of the brazed material.

II. CONCLUSIONS

Remaining within the practical shop limitations for producing the large Phoebus-2 nozzle forgings of Hastelloy X, it was demonstrated that improved tensile strength properties were attained. It is estimated that the 0.2% offset yield strength could be increased from a minimum of 40.0 KSI to 46.0 KSI and the minimum tensile elongation could be changed from 40% to 28%.

Following exposure to the Phoebus-2 brazing cycle, the 0.2% offset yield strength and tensile elongation at -320°F of the improved forgings increased about 10% (71.7 to 79.7 KSI) and 100% (13 to 26%), respectively, compared to the previous material. At room temperature the yield strength and elongation were only slightly changed by the brazing cycle; the average yield strength decreased from 51.8 KSI to 49.5 KSI and the elongation decreased from 31% to 29%.

III. RECOMMENDATIONS

The revised forging procedures and improved properties should be applied to any subsequent Hastelloy X nozzle component procurement.

A specification should be prepared incorporating the requirements for improved forgings. (A preliminary specification, identified as AGC-TAB01, has been prepared.)

IV. DEVELOPMENT PROGRAM

A. INTRODUCTION

The material requirements for the Phoebus-2 Nozzle Jacket exceeded the state-of-the-art for wrought Hastelloy X at the inception of the program. Specifically, the need was for large diameter forgings of heavy wall construction. The components indicated a starting multiple (billet or ingot) size of 22- to 24-in. diameter and 7000- to 8000-lb with the requirement for very large capacity forging equipment. The supplier was Wyman-Gordon Co. with their 50,000 ton capacity Air Force press.

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

<u>Component</u>	<u>Approximate Size of Forging</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 forgings of Hastelloy X made to date. They were produced direct from 24-in. diameter, consumable-electrode, vacuum-melted cast ingot, made by the Union Carbide Corporation, Stellite Division, to controlled chemistry. The following minimum tensile properties were required for the finished forgings 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>Nozzle Section</u>			
	<u>Convergent</u>		<u>Divergent</u>	
	<u>Room</u>	<u>1200°F</u>	<u>Room</u>	<u>1200°F</u>
UTS (KSI)	102.0	72.8	93.2	69.2
0.2% YS (KSI)	43.2	26.9	42.3	26.7
Elong. (%)	48	47	33	36

and the following tangential 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

* Size of components precluded adequate handling and facilities for water quenching.

The tensile values are more conveniently compared in bar chart form in Figure 1. With the exception of a few room temperature results from the divergent section, 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 KWI) and 1200°F (25.0 KSI), although the average values for the die forgings were a mere 5% 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 spread (below minimum) for the inlet rings was the result of a few low test results because the overall average is a ductile 48%. The low room temperature tensile strength and elongation for the divergent sections were the result of the forging practice employed and to localized concentrations of carbides in the area from which the test material was obtained. The divergent section was forged by upsetting, pancaking, piercing, and closed die extrusion, with the major metal movement toward the large diameter. The test material taken for specimen fabrication was removed from the less hot worked, smaller diameter areas and was machined transverse to the carbide stringers. By comparison, the convergent section was forged by upsetting, pancaking, and then rough-and-finish-form closed die forging to greater uniformity of hot work.

The results obtained were acceptable from both the engineering design and functional basis, especially for such large state-of-the-art pieces, but improvement and assurance that all forgings would exceed the minimum strength criteria was a product improvement goal. 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 Wyman-Gordon utilizing a 4-in. thick slice of a 22-in. diameter production ingot (Heat 260-5-2863) and a piece of a large ring of excess material machined from a partially processed Phoebus-2 forging (divergent section, S/N 880601, Heat 260-5-2850). The results are presented in Appendix I.

B. TEST RESULTS AND DISCUSSION

The Forging Improvement Program was directed toward providing a finer grain size, with the concomitant increased strength, through the control of 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-2 nozzle forging sizes and consistent with acceptable metallurgical practice for Hastelloy X. The condensed and simplified results are presented in Figures 2 and 3 for the ingot upset specimens and the forged ring section specimens, respectively. Using the current (recommended) initial forging temperature of 2150°F and a heavy finish forging reduction (50%) with a reduced PFHT (2000°F) a yield strength of 49.0 KSI was attained; likewise, decreasing the finish forging temperature (2000 or 2100°F) and limiting the final forge reduction to restrike level (5 to 10%) with a reduced PFHT (2000°F), consistent yield strengths of 48.0 to 50.0 KSI were attained. The forging grain sizes for these conditions ranged from ASTM 6 to 9. The effect of the improved grain size was also reflected at the 1200°F test temperature with the yield strength averaging 32.1 KSI.

Tensile elongation at room temperature was decreased with values ranging from a low of 23% to a high of 35% (average 31%) on the ingot starting material and a range of 34 to 42% (average 36%) for the forged ring starting material.

Figure 4 presents all the test data for the yield strength and the tensile elongation from Appendix I 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 46 KSI combined with a useful 28% elongation.

The significance and the inter-relationship of the finish forging temperatures, reductions, and PFHT are more graphically illustrated in Figure 5.

The yield strength of the forgings was improved by:

1. Decreasing the final forging temperature in order to maintain the benefits of hot strain by minimizing the annealing and grain growth on the forge,
2. Decreasing PFHT in order to minimize the removal of the benefits of hot strain from annealing and grain growth, and
3. Increasing the final reduction in order to introduce more hot strain for recrystallization and grain refinement.

From a thorough review of the data and results, the following conditions were selected for testing at Aerojet, utilizing the braze cycle treatment required on the Phoebus-2 nozzle jacket:

<u>Forge Temp. (°F)</u>	<u>Finish Forge Temp. (°F)</u>	<u>Final Red. (%)</u>	<u>PFHT (°F)</u>	<u>S/N Upset Ingot/Pancake</u>	<u>Comments</u>
2150	2150	50	2000	26/10	This was a minimum deviation from the Phoebus-2 practice, but because of heavy final reduction presupposes no restriking.
2150	2100	10	2000	25/11	Restrike condition of high probability.
2150	2100	5	2000	--/14	This condition was not tested, but it represented a real restrike reduction condition.
2150	2000	10	2000	27/13	Same as above; however, restrike temperature represented probably lower limit with existing part sizes and press capacity.
2150	2000	5	2000	20/12	

Sample pancake forgings (approximately 1-in. thick by 10-in. dia., identified by S/N 10 through 14) were provided to Aerojet for exposure to the braze cycle and tensile testing (in duplicate) 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 because of the reduction in elongation as the grain size decreases as a result of the new forging practices. For comparison, the tensile properties of Hastelloy X obtained from cross-forged, rolled rings* (20-in. OD x 17-in. ID x 16-in. long) were as follows after being processed through the brazing cycle:

<u>UTS (KSI)</u>			<u>YS (KSI)</u>		<u>ELONG. (%)</u>	
Temp. (°F)	<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
1200	74.8	70.2	26.8	22.6	31.4	18.0

The average tensile properties of the sample pancake forgings (Numbers 10 through 14) produced by Wyman-Gordon under the improvement program were as follows after being processed through the brazing cycle:

<u>UTS (KSI)</u>			<u>YS (KSI)</u>		<u>ELONG. (%)</u>	
Temp. (°F)	<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	49.4	46.7	29	26

The detailed test results are itemized in Table I and the significant differences between the improved forgings and previous forging are obvious. The yield strength and tensile strength are distinctly improved, but an outstanding feature is the tensile elongation. The tensile ductility for the improved forgings

* Total reduction from ingot to forging was approximately the same as the convergent and divergent nozzle section reductions. ASTM Grain Size was 1 to 4.

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 significance of the improved yield strength is obviously important to the design, while the improved ductility is less obvious but nevertheless highly important in the cyclic thermal performance of a nozzle. Improved ductility provides an increased margin in the nozzle cyclic thermal exposure life.

Table II itemizes the as-annealed room temperature tensile properties from the forgings (S/N's 10 through 14) supplied to Aerojet for testing. Included also are the test results from the equivalent forgings (S/N's 20, 25, 26 and 27) uniformity of the test results from two series of separately forged pieces.

Micro-examination revealed a significant difference in the distribution of the aging precipitates (carbides) in the micro-structures of the forgings as a result of the post-forge annealing temperatures (PFHT) and the heat treatment through a simulated brazing cycle followed by slow cooling (see Figures 12 through 27 in Appendix I).

The complete solution anneal treatment at 2150°F produced a large grain structure with heavy, continuous grain boundaries and no precipitate within the grains (matrix), except for some primary carbide more-or-less evident in all the microstructures. After exposure to the brazing cycle treatment the grain boundaries contained more precipitate (continuous) and carbides were evident in the matrix.

When annealed at low temperatures (1950° to 2100°F), the forging microstructures revealed smaller worked grains, less grain boundary precipitate, discontinuous grain boundaries and no precipitates in the matrix. After the

brazing cycle considerable precipitation was noted in the matrix with slight-to-no change in the grain boundary precipitates. The discontinuity of the grain boundary precipitation appeared to increase with decreasing annealing temperature.

The observed phenomena supported the mechanical properties manifestation of the microstructures. The greater dispersion of the precipitates in a finer grained structure increased strength with some loss in ductility. The finer grain size increased the amount of grain interface, distributing the grain boundary precipitates over greater length thereby producing a discontinuous network of the low ductility constituent. This, in turn, permitted the ductile matrix to exert a greater influence on tensile ductility.

TABLE I

TENSILE PROPERTIES OF HASTELLOY X
PANCAKE FORGINGS MADE
FROM CAST INGOT (HEAT 260-5-2863)

(All Specimens Exposed to Phoebus-2 Braze Cycle)

<u>Serial No. and Specimen Ident.</u>	<u>Temp (°F)</u>	<u>UTS (ksi)</u>	<u>YS (ksi)</u>	<u>Elong (% in 1-in.)</u>	<u>RA (%)</u>
10C	-320	142.9	80.0	16	15
10D	"	146.7	77.0	21	19
11C	"	155.2	80.9	25	22
11D	"	157.5	80.6	29	27
12C	"	153.8	-	29	23
12D	"	147.6	84.3	19	15
13C	"	156.0	77.3	30	25
13D	"	154.4	80.1	28	23
14C	"	156.6	77.5	28	20
14D	"	154.1	-	31	25
Average	"	152.5	79.7	26	21
10E	Room	112.0	50.9	31	29
10F	"	108.8	46.8	26	25
11E	"	112.8	48.9	29	28
11F	"	113.3	46.7	32	34
12E	"	109.3	49.3	32	29
12F	"	111.9	54.1	26	25
13E	"	113.1	49.7	29	28
14E	"	111.5	50.3	31	30
14F	"	112.4	48.1	31	30
Average	"	111.9	49.5	29	27

TABLE II

TENSILE PROPERTIES OF HASTELLOY X PANCAKE FORGINGS
MADE FROM CAST INGOT (HEAT 260-5-2863)
(All Specimens Annealed at 2000°F)

Serial No. and Specimen Ident.	Final Forging Temp (°F)/ Reduction (%)	Test Temp (°F)	UTS (ksi)	YS (ksi)	Elong % in 1-in.	RA (%)
10A (26)	2150/50	Room	106.9	55.5	31	27
10B "	"	"	105.9	49.4	25	24
11A (25)	2100/10	"	110.0	45.8	32	33
11B "	"	"	111.0	49.4	32	34
12A (20)	2000/5	"	109.2	--	30	26
12B "	"	"	108.7	56.9	25	25
13A (27)	2000/10	"	111.4	50.4	35	35
13B "	"	"	113.0	52.7	34	32
14A	2100/5	"	115.0	52.2	27	22
14B	"	"	115.1	53.5	35	35
Average	-	Room	110.6	51.8	31	29
26 (10)	2150/50	Room	106.4	49.0	32	31
" "	"	"	107.6	49.0	32	32
" "	"	"	104.2	48.8	28	28
" "	"	"	107.0	49.0	30	30
25 (11)	2100/10	"	108.0	49.0	33	31
" "	"	"	108.0	48.4	33	33
" "	"	"	108.0	49.0	30	29
" "	"	"	107.4	50.0	30	23
20 (12)	2000/5	"	108.0	48.0	30	30
" "	"	"	109.0	48.0	34	36
" "	"	"	108.0	47.6	35	38
" "	"	"	109.0	48.0	31	29
27 (13)	2000/10	"	111.0	50.0	32	30
" "	"	"	111.0	49.0	31	31
" "	"	"	109.6	49.4	30	27
" "	"	"	108.4	51.0	23	25
-	2100/10	-	NO TESTS CONDUCTED			
Average	-	Room	108.2	49.0	31	31

NOTE: Number in () indicates related forging identification.

FIGURE 2

INGOT UPSET SPECIMENS - FORGE CYCLE ANALYSIS *

(Heat 260-5-2863)

<u>PREDOMINANT GRAIN SIZE</u>	<u>FINAL FORGE (°F)</u>	<u>PFHT (°F)</u>	<u>FINAL REDUCTION</u>	<u>ROOM TEMPERATURE**</u>	
				<u>YS (ksi)</u>	<u>EL (%)</u>
• 2½	2150	2150	50%	42.5	39½
3	2150	2150	10%	44.0	43
2½	2150	2150	5%	42.9	43
✦ 3	2100	2100	10%	44.9	37½
▲ 5	2000	2000	5%	47.9	32½
7	2000	2000	10%	49.9	29
▲ 7	2150	2000	50%	49.0	30½
3	2150	2000	10%	44.8	35
2½	2150	2000	5%	44.0	36
▼ 9	2100	2000	10%	49.0	31½
□ 7	2000	1950	5%	48.8	33
	2000	1950	10%	49.6	29½

*Wyman-Gordon Company Report No. RD 66-125, MD&E No. 204, May 1966

**Average of four specimens for each condition.

FIGURE 3

FORGED RING SECTION - FORGE CYCLE ANALYSIS *

(Heat 260-5-2850 P/N 11148 56-11)

<u>PREDOMINANT GRAIN SIZE</u>	<u>FINAL FORGE (°F)</u>	<u>PFHT (°F)</u>	<u>FINAL REDUCTION</u>	<u>ROOM TEMPERATURE**</u>	
				<u>YS (ksi)</u>	<u>EL (%)</u>
• 5	2150	2150	5%	47.8	39½
5	2150	2150	10%	43.8	44
4	2150	2150	40%	44.1	44½
◊ 5½	2000	2050	5%	46.1	37
6½	2000	2050	10%	47.9	35
4½	2000	2050	25%	46.5	38
▲ 6½	2150	2000	5%	49.1	36
6	2150	2000	10%	49.4	36½
8½	2150	2000	40%	51.6	35
▲ 7½	2000	2000	5%	49.5	36
7½	2000	2000	10%	50.0	35
8	2000	2000	25%	53.0	34½

* Wyman-Gordon Company Report No. RD 66-125, MD&E No. 204, May 1966

** Average of two specimens for each condition.

FIGURE 4

Upset Ingot Specimens + Forged Ring Specimens

Relationship of 0.2% Yield Strength and Elongation to ASTM Grain Size

FIGURE 5

INTER-RELATIONSHIP OF FINISH FORGING TEMPERATURE, REDUCTION AND PFHT

(Ingot Upset Specimens, Heat 260-5-2863)

Effect of decreasing final forging temperature and PFHT with increasing restrike reduction on 0.2% Yield Strength.

Refer to Figures 2 and 3 for symbol identification

Effect of constant final forging temperature and decreasing PFHT with increasing reduction on 0.2% Yield Strength

APPENDIX 1

Report No. RD 66-125, M. D. & E. #204
Forging Experiment Aimed at Increasing The
Tensile Properties of Hastelloy X
(Phase 1)

APPENDIX 1 TRANSMITTED TO SNPO-C ONLY