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DATE: September 3, 1956
SUBJECT: PROGRESS REPORT FOR WEEK
ENDING SEPTEMBER 1, 1956
TO: J. H. Frye, Jr.
FROM: Lt. T. Hikido

This document consists of // pages.

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INTRA-LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

To: J. H. Frye, Jr.
Building 2000

Date: September 3, 1956

Subject: PROGRESS REPORT FOR WEEK
ENDING SEPTEMBER 1, 1956

FABRICATION

Nickel-Molybdenum Alloys

Rod Extrusions.- Twelve rod extrusions were completed this week with moderate success to provide material for stress-rupture specimens and weld wire. Details are tabulated in Table I.

Cold swaging of weld wire from two extruded billets of alloy INOR-2 has been essentially completed. This material could be reduced 75% in area between anneals to a final size of 1/8-in. OD. Approximately 8-1/2 lbs of this wire will be turned over to the Welding and Brazing Group for evaluation.

TABLE I

<u>Nominal Composition</u>	<u>Extrusion Temp. Ratio OF</u>	<u>Results</u>	<u>Remarks</u>
Ni-16 Mo-6 Cr-1 Al-1 Cb- 0.08 C-0.5 Mn (INOR-7)	2150 12.7:1	Stalled-2 billets	Billets to be re-extruded.
	2150 4.3:1	Good-1 billet	Stress-rupture spec. to be fabricated.
Ni-16 Mo-5 Cr-0.5 Al- 0.06 C-0.5 Mn (INOR-2)	2150 12.7:1	Good-2 billets	Weld wire to be fabricated.
	2150 4.3:1	Good-1 billet	Stress-rupture spec. to be fabricated.
Ni-16 Mo-1.5 Ti-1 Al- 0.06 C-0.5 Mn (INOR-3)	2200 4.3:1	Good-1 billet	Stress-rupture spec. to be fabricated.
	2200 6.25:1	Good-1 billet	Weld wire to be fabricated.
	2300 12.7:1	Good-1 billet	Weld wire to be fabricated.

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TABLE I (Continued)

<u>Nominal Composition</u>	<u>Extrusion Temp. Ratio °F</u>	<u>Results</u>	<u>Remarks</u>
Ni-16 Mo-1.5 Ti-2 Al- 0.06 C-0.5 Mn (INOR-4)	2200 4.3:1	Good-1 billet	Stress-rupture spec. to be fabricated.
	2300 12.7:1	Good-2 billets	Weld wire to be fabricated.

Aging Studies - A study of the aging characteristics of numerous alloys is in progress. A second series of tensile tests at room temperature was completed for specimens aged for 119 hr at 1300°F. The results are shown in Table II.

TABLE II

Room Temperature Tensile Tests of
Ni-Mo Alloys Aged 119 Hr at 1300°F

<u>Alloy (Nickel Base)</u>	<u>Yield Point 0.2% Offset psi</u>	<u>Tensile Strength psi</u>	<u>Elongation % in 2 In. (aged)</u>
17 Mo-.5 Al	45,000	109,800	56.2
18 Mo-2 Nb-.25 Be	77,000	139,300	42.5
16 Mo-1.5 Ti-1 Al	48,800	119,600	51.8
15 Mo-1.5 Ti-1 Al	42,000	110,600	55.0
15 Mo-5 Cr-3 Nb-3 W	69,300	139,200	42.0
15 Mo-3 Nb-3 W-.25 C	63,500	131,600	34.7
15 Mo-3 Nb-3 W	59,200	132,300	46.7
15 Mo-6 Cr-4 Fe	46,600	113,800	46.7
16 Mo-1.5 Ti-2 Al	98,900	165,800	35.6
15 Mo-2 Nb-2 W	41,100	108,200	50.0
16 Mo-5 Cr-1 Al-1.5 Ti	80,000	146,700	41.7
15 Mo-2 Nb-2 V-.2 C	62,600	132,900	31.5
15 Mo-2 Nb-2 W-.2 C	57,300	126,600	36.0
15 Mo-2 V-2 W-.2 C	54,900	128,100	37.2
18 Mo-.5 Be	82,600	153,100	29.3

Several of the alloys show increases in hardness after aging with a corresponding decrease in the ductility. However, all the compositions show ductilities of the order of at least 30%.

Tubing Fabrication - Seventy-five ft of special alloy tubing, representing eight compositions, .500-in. OD x .035-in. wall was received from the Superior Tube Company. All the tubing was sound except the 17 Mo-4 Ti-Ni alloy which had numerous short longitudinal splits and the 4 Al-17 Mo-Ni alloy which split during intermediate reductions and yielded no tubing. The Welding and Brazing Group has been requested to fabricate thirteen thermal convection loops from this shipment.

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Thermal Barrier for Pump Shield Assembly

Three stabilized ZrO_2 discs have been received from the Ceramics Section. These are approximately 7-in. dia. x 1 in. thick and have densities of about 3.25 g/cm^3 . Analysis of BMI data on 3.52 and 3.08 g/cc zirconia gives the following expression for thermal conductivity of stabilized ZrO_2 (of density from $3.0\text{-}3.5 \text{ g/cm}^3$):

$$k = 1.82 \times 10^{-3} \rho^{-7.5 \times 10^{-7} T - 3.9 \times 10^{-3}}$$

OK. where k = thermal conductivity, cal/cm-sec- $^{\circ}\text{C}$

ρ = density, g/cm^3

T = temperature, $^{\circ}\text{C}$

The conductivity of the above discs is then:

$$k = 2.0 \times 10^{-3} - 7.5 \times 10^{-3} T$$

Nb- UO_2

The Nb- UO_2 sintered compacts were hot rolled at 1100°C in SS frames to a total reduction of 76.6%. Examination indicated no cracking of the core. One specimen is being aged 500 hr at 1000°C in the capsule. The other specimen will be examined metallographically to check bonding of the Nb to the Nb- UO_2 cores.

The second Nb-U alloy melt was hot rolled at 1200°C , in a capsule, to a total reduction of 82.2%. The capsule cracked at about 23% total reduction. Metallographic examination revealed oxide contamination in several areas of the sample. These areas were removed from the sheet, and the sheet was annealed for 1-1/2 hr at 1100°C . Roll cladding Nb-U alloy with Nb and Thermanol in an evacuated capsule will be attempted.

CORROSION

Inconel-Graphite-Fused Salt Test

An Inconel-C18 graphite-Bath 30 system and an Inconel-Bath 30 system were tested for 100 hr in seesaw furnaces with hot and cold zones at 1500 and 1250°F , respectively. The purpose of these tests was to determine if the graphite would "tie up" the chromium in the Inconel as carbides and thus affect the depth of corrosion attack.

The test results indicated that the presence of the C18 graphite had no effect on corrosion depth of the Inconel. Also, no attack of the C18 graphite by the fused salt was detected.

For the tests, two identical capsules were made from 1-in. Schedule 40 Inconel pipe and loaded with equal quantities of Bath 30 (Volume at 1500°F equaled one-third that of capsule). One of these capsules was used as a control sample, and in the other a 1/4-in. dia. x 18-in. long C18 graphite rod was suspended.

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Metallographic examinations of the control and test capsules showed typical fused salt attack to an average depth of 2 mils in each hot zone and no attack in the cold zones. This similarity in attack was substantiated by the chemical analyses of the fused salts (see Table 1).

Table 1

CHEMICAL ANALYSES OF SAMPLES OF BATH 30
FROM THE INCONEL-GRAPHITE-BATH 30 TESTS

<u>Source of Bath 30 Analyzed</u>	<u>Percent</u>			
	<u>Ni</u>	<u>Cr</u>	<u>Fe</u>	<u>C</u>
As-received	0.002	<0.001	0.022	0.79
Control (without C18 Graphite Rod)	<0.001	0.008	0.008	0.63
Test (with C18 Graphite Rod)	<0.001	0.007	0.009	1.11

Etchants and hardness tests revealed no detectable difference between the control and test capsules. There was a slight increase in hardness in both capsules over that of untested hydrogen-fired Inconel, but the etchants did not show any definite signs of carburization.

From the examination of the C18 graphite rod it was found that it had:

- 1) Sagged approximately 1/8 in. from its longitudinal center line.
- 2) A 1.28% weight gain (assumed to be due to a thin fused salt vapor deposit toward the hot zone).
- 3) A 0.06% change in diameter (average of 16 values).
- 4) No fused salt in its pores (based on metallographic examinations).
- 5) Not altered in structure nor were there any signs of attack.

A similar Inconel-fused salt test with 0.25% of amorphous carbon added is planned. The purpose will be to determine if the form of the carbon affects the carburization of the Inconel.

INSPECTION

Weld Inspection

The following welds were fabricated and inspected by the ANP Inspection Group during this period:

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<u>Bldg.</u>	<u>Total No. Inspected</u>	<u>No. Rejected</u>	<u>Percent</u>
9201-1	147	9	6
9201-3	<u>196</u>	<u>22</u>	<u>11</u>
	343	31	9

Approximately 60% of the rejected welds required only repair, while the remainder were re-welded completely. Principal rejectable conditions noted were porosity, cracks, lack of fusion, and irregular penetration.

Inspection of Calrod Heaters

Radiographic inspection of Calrod heaters has been requested by the ARED Operations Group. The purpose of this examination is to check the orientation of the conduit throughout the entire length of each rod. Those rods found with improper positioning of conduit are subject to rejection. During this week 100 units, each thirty-six in. long were radiographed. All were found to be acceptable for use.

Black, Sivalls and Bryson Radiator

A 1/2 Mw HCF radiator was received from Black, Sivalls and Bryson for inspection. A report will be made on completion of inspection and review of vendor fabrication data.

Metallographic Examination

Metallography reports were received on the following items:

Inconel Plate, Shell No. 1 - Inspection was requested for evidence of intergranular corrosion. The examination revealed there was no appreciable evidence of corrosion; however, propagation of cracks from inclusions was noted. This condition apparently resulted from over-stressing and improper heat treatment during forming.

Valve Assembly, NaK Circuit, 321 SS - Inspection was requested to determine cause of failures occurring in bellows during initial flow at 1000°F. Examination indicated the failures were due to stress and corrosion.

Elongation of Heat Exchanger Tubes

X-ray exposures of 0.188-in. OD tubes elongated 0.9, 1.1, 1.4, and 3.5% were taken to determine changes in wall thickness. No changes in density other than those within normal tube tolerance were noted. Additional exposures will be made on as received tubes and stretched tubes in an attempt to measure changes in wall thickness.

Specification

Material Specification MMS-7 for forging application has been revised to conform to verbal agreements with the International Nickel Company. Revisions on Shell VII

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fabrication specification dealing with material, forging, heat treatment, and inspection requirements have been submitted to the Reactor Construction Group.

Material Inspection

<u>Item</u>	<u>Description</u>	<u>Quantity</u>	<u>Method of Inspection</u>	<u>Reject</u>
Tubing	Inconel, 1-in. OD x 0.065-in. W.T.	54 ft	Visual, Dye penetrant	100%

Comments: Numerous indications were noted on dye penetrant inspection which were found to be greater than 5% of the wall thickness after filing.

Inconel, CX-900
0.187-in. OD x 0.025-in. W.T.

Comments: No reports were issued on completed batches; however, the following inspections were performed:

Pentrex Fluorescent Penetrant	3150 ft
Radiography	1519 ft
Film Interpretation	1260 ft

Since more than one batch of tubing is represented in the above figures, rejection rates will be given on final inspection results only.

Pipe	Inconel, various sizes	351 ft	Visual, Dye penetrant Ultrasound	124 ft
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Comments: The rejected material was found to have a general pitted condition which was found to be greater than 5% of the wall thickness. It was rejected from use in the ART and ETU.

Plate	Inconel, various thicknesses	25 sq ft	Visual, Dye penetrant reflectoscope	None
	Hastelloy B	26 sq ft	Visual, Dye penetrant reflectoscope	None
Sheet	Inconel, 0.062 in.	14 sq ft	Visual, Dye penetrant reflectoscope	None
Bar	Inconel, various diameters	30 ft	Visual, Dye penetrant reflectoscope	None
	316 SS 8-in. dia.	1/2 in.	Visual, Dye penetrant reflectoscope	None
Fittings	Pipe cap 2 and 4-in. IPS 4 ea		Visual, Dye penetrant	None

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METALLOGRAPHY

Status of Examination of York No. 5 Radiator

The radiator designated York No. 5 failed in service and was received for examination for mass transfer, failure, and fin-to-tube joint efficiency.

The radiator has been sectioned and twenty-four samples have been removed. These samples have all been mounted in Hysol and are being polished. In addition, three samples were removed from the failed area. Visual examination shows only one failed tube. Macrophotographs have been made as the work called for them.

WELDING AND BRAZING

Stress Relieving of Inconel Reactor Components

A study is being conducted to determine the optimum stress relief temperatures for Inconel reactor components from the standpoints of minimization of surface oxidation films and subsequent higher temperature brazeability. Two compositions of as received Inconel were heated in a dry-hydrogen atmosphere for 1/2 hr at 1100, 1200, 1300, 1400, 1500, 1600, 1700, 1800, and 1900°F. A visual analysis of the surface films (if any) is being made and compared to those obtained on samples of nickel and type 310 stainless steel which were treated under the same conditions. Additional samples of pre-oxidized Inconel, nickel, and stainless steel were also heated at these temperatures to determine the bright-annealing tendencies at these temperatures.

Brazing studies will also be performed on these samples of material to determine the effects of oxide film formation. A report on this investigation will be written as soon as tests are complete.

Main Heat Exchanger Fabrication

A sample header, of a design similar to that proposed for the main heat exchangers, has been received from Y-12 for tube-to-header welding studies. This header, which contained holes for approximately ten tubes, has been bright-annealed to remove oxides near the weld zone. Manual welding studies will be initiated this week.

Welding of Nickel-Molybdenum Alloys

Samples of Hastelloy B welded plate have been aged for 200 hr at 1100, 1300, 1500, and 1650°F. Hardness traverses across the weld are being made on each sample and on an as-welded sample, and the results are being recorded graphically. This data should present an important means of determining the aging characteristics of nickel-molybdenum alloy weldments.

Fabrication of Strain-Cycle Rig Components

A Coast Metals No. 52 brazed strain-cycle rig was fabricated for Mr. D. P. Gregory of the ANP Heat Transfer and Physical Properties Section.

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High-Conductivity Fin Radiator Fabrication

A report has been completed describing the results of temperature rate-of-rise studies on York brazing alloy rings. A metallographic investigation of the brazed joints prepared in the study revealed that a large amount of boron and/or silicon had left the alloy and entered the stainless steel fin-cladding. Since experience has shown that Inconel is less prone to this type of intermetallic diffusion, a test specimen containing Inconel-clad copper fins was prepared and subjected to the York rate-of-rise (100°F/hr). As was expected, better melting of the braze ring was obtained. Metallographic studies on this sample will be conducted to determine the degree of brazing obtained.

General Service

Twenty-seven type 347 stainless steel gaskets were stress-relief annealed in dry hydrogen for the HRP Program.

MECHANICAL PROPERTIES

The work of the Mechanical Properties Group is summarized in the attached table.

T. Hikido
Lt. T. Hikido

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Temp. °F	Environment	Stress, psi	Hours in Test	% Elongation	Comments
INCONEL					
1500	Argon	3,500	1343	5.08	Annealed 2050°F, 1/8-in Spec.
1500	Argon	3,500	1344	7.33	Annealed 2050°F
1200	Argon	16,000	48	1.44	Annealed 2050°F, 1/4-in Spec.
1500	Argon	3,500	695	7.44	Re-annealed 1650°F
1500	Argon	3,500	1245	6.03	Annealed 2050°F, 1/4-in Spec.
1200	Argon	16,000	312	6.19	Annealed 2050°F, 1/8-in Spec.
1500	Argon	3,500	72	0.57	No decarb., Coarse grain
1300	Air	10,000	2711	14.12	Fine Grain
1500	Air	4,000	4580	8.21	Coarse grain, 0.020 Spec.
1500	Air	3,500	1004	6.65	Fine grain, 0.020 Spec.
1650	Air	2,000	3714	12.57	Coarse grain
1650	Air	1,500	2926	Out of field	Fine grain
1500	Nitrogen	3,500	192	0.73	Fine grain
1500	Nitrogen	3,500	3864	4.18	Annealed 1650°F
1500	Fused Salt	3,500	1744	8.24	Fine grain, 0.450 sq.
1500	Fused Salt	2,590	2537	19.71	0.020-Fine grain, NX3748 ^x
1500	Fused Salt	3,500	2705	21.71	Fine grain (Bar Spec.) ^x
1500	Fused Salt	3,500	691	5.51	Coarse grain
1500	Fused Salt	2,000	19	--	Coarse grain
1500	Sodium	4,000	306	3.18	Fine grain
1500	Sodium	2,500	3378	7.31	Fine grain
1300	Sodium	12,000	303	19.18	Fine grain
1300	Sodium	12,000	455	36.51	Fine grain ^x
1300	Sodium	10,000	2057	33.29	Fine grain, NX8004
1500	Fused Salt	3,500	1697	12.42	0.250 Rod, Fine grain
INCONEL "X"					
1500	Air	20,000	576	1.93	Aged and treated
HASTELLOY "B"					
1500	Argon	20,000	57	38.13	Annealed 2100°F, 1/4-in Spec.
1800	Argon	5,000	43	1.70	Annealed
1800	Fused Salt	3,000	186	1.16	Annealed
1800	Fused Salt	2,000	3570	2.51	Annealed
HASTELLOY "W"					
1300	Argon	20,000	5443	2.96	Annealed 2100°F
1600	Argon	5,000	2376	7.15	Annealed 2100°F
1650	Fused Salt	5,000	1196	6.26	Annealed

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Temp. °F	Environment	Stress, psi	Hours in Test	% Elongation	Comments
1500	Fused Salt	8,000	1362	6.13	Annealed
1300	Fused Salt	25,000	2368	7.97	Annealed
1800	Fused Salt	2,000	258	3.46	Annealed
1800	Fused Salt	2,500	2056	9.90	Annealed
HASTELLOY "X"					
1500	Fused Salt	7,957	3929	14.92	Annealed
1500	Fused Salt	6,000	2587	4.72	Annealed
Nickel-Molybdenum Alloys					
1500	Fused Salt	8,000	187	28.80	Annealed 1/2-hr 1800°F, VT-28
1500	Fused Salt	8,000	325	73.40	Annealed 1/2-hr 1800°F, VT-27*
1500	Fused Salt	8,000	41	1.06	Annealed 1-hr 2100°F, B-3278
1500	Fused Salt	8,000	162	25.71	Annealed 1-hr 2100°F, VT-22
1500	Fused Salt	8,000	204	68.97	Annealed 1/2-hr 1900°F, VT-29
1500	Fused Salt	8,000	43	1.67	Annealed 1/2-hr 1900°F, VT-26
VT-22 = 10 Fe-10 Mo-5 Cr					
VT-26 = 1.5 Ti-1 Al-16 Mo					
VT-27 = 1.5 Ti-2 Al-16 Mo					
VT-28 = 15 Mo-2 Nb-2 W					
VT-29 = 1.5 Ti-1 Al-5 Cr-16 Mo					
B-3278 = 20 Mo-7 Cr-2 Al-0.8 Mn-0.12 C					
Beryllium					
1300	Sodium	5,000	89	Approx. 0.2%*	
Niobium					
1500	Argon	20,000	576	1.55	Clad
Lithium-Magnesium					
93°C	Air	100	1702	0.07	Phosphated
93°C	Air	200	1702	0.33	Phosphated
93°C	Air	400	1702	4.05	Phosphated
93°C	Air	600	864	22.75	As received
93°C	Air	800	647	22.51	Old heat
Lead-Copper (Copperized Doe Run - 0.06% Cu)					
110°C	Air	50	2255	0.08	As cast
110°C	Air	100	2255	0.16	As cast
110°C	Air	20	2255	0.05	As cast
110°C	Air	20	2255	0.05	As cast
110°C	Air	100	2255	0.47	As cast
110°C	Air	50	2255	0.03	As cast

*Rupture

**Discontinued

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