HW-22572

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Prepared for the U.S. Department of Energy under Contract DE-AC06-76RLO 1830

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#24. KL Sanborn - RL Beede
#25. RE Hueschen - LF Higginson
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November 1, 1951

TECHNICAL ACTIVITIES REPORT
RESEARCH AND DEVELOPMENT & 234-5 METALLURGY GROUPS
METALLURGY - FILM TECHNOLOGY UNIT
October 1951

(Included 1 Unclass. graph)

To
FILE

By W. L. Schallick
O. J. Wick

[redacted]

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The September Technical Activities Report of the Research and Development and 234-5 Metallurgy Groups is HW-22280.

P-10 DEVELOPMENT (NDA TF-5)

I. DuPont Assistance Program - J.A. O'Rourke

Several satisfactorily etched surfaces have been obtained on a sample of the 10% Li - 90% Al alloy submitted by DuPont. The best technique developed thus far for etching involves briefly swabbing the surface of the polished samples with a dilute solution of acetic or oxalic acid. The surface is then washed with ethyl alcohol and rapidly covered with a film of 3-in-1 oil. Electrolytic etching techniques have been used successfully in a few instances but the quality of an etch produced in this manner leaves much to be desired.

Analyses of the gas contents of four of the DuPont 10% Li slugs have been completed and the results are tabulated below. Since both exposed ends of the slugs were sanded just before each analysis, the results given for the 400°C surface fraction are only indicative of the quantities of surface gases adsorbed on the aluminum cladding and the freshly cleaned end surfaces. It can readily be seen that the volumes of dissolved gases are quite low and should not prove troublesome insofar as isotopic contamination of the product is concerned.

Slug No. (DuPont)	400°C Surface Fraction (cc/100 g)		600°C Dissolved Fraction (cc/100 g)	
	H_2	Non-H_2	H_2	Non-H_2
33	0.13	0.014	0.22	0.045
34	0.15	0.051	0.24	0.000
61	0.89	0.086	0.45	0.000
81	0.41	0.026	*---	0.000

* Gas lost due to break in line.

In the near future two additional slugs are to be analyzed without polishing the slug ends to ascertain the quantity of adsorbed or chemically bonded gas on the exposed alloy surfaces.

II. 108-B Metallurgical Laboratory - J.A. O'Rourke

The testing of equipment to be installed in the metallurgical laboratory, 108-B Building is continuing. Good photomicrographs of material have been made at magnifications from 1X up to 90X with the Bausch and Lomb model L camera. Using this camera in conjunction with the metallurgical microscope good photomicrographs have been obtained of samples at magnifications up to 750X. At higher magnifications some definition is lost due to chromatic aberration, but some of this difficulty may be eliminated by substituting Huygenian eyepieces for the acromatic eyepieces presently being used.

III. Corrosion Testing of P-10 Slugs - J.A. O'Rourke

A number of P-10A slugs are currently being sized into cans to minimize the width of the annulus between the can and the slug. These pieces, together with an equal number of regular P-10 slugs, will be penetrated at the cap end and will be placed in an autoclave and run until failure occurs. Any effects of sizing on the corrosion rates of the pieces are to be noted by frequent inspection during the test.

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IV. Thermal Cycling of Pd Tubing - WA Bleckert, JA O'Rourke

The apparatus for the thermal cycling of Pd tubing in hydrogen atmospheres has been assembled and tested. Preliminary studies indicate that currents of 100 amp or more will be necessary to heat the tubing to the 500°C temperature desired for this test. Since the present copper lead wires to this apparatus will not carry this magnitude of current new leads are to be installed.

V. Studies of Extraction Tube Throat Deposits - MJ Sanderson

X-Ray diffraction patterns were made to identify the components of two samples taken from a furnace extraction tube which were submitted by John O'Rourke. The samples proved to be a mixture of Pb, Li, Pb_3Li_{10} and $LiCO_3$.

VI. Weight Loss and Solution Potential Data on P-10 Alloy Exposed to Pile Process Water at the 105-D Flow Laboratory - CH Pitt RS Dalrymple

After two weeks exposure in Pile Process Water at 20°C, 70°C, and 90°C, the following corrosion rates were found for heat treated and non-heat treated P-10 alloy:

Sample	Average Corrosion Rates in Inches Per Month at 20°C	at 70°C	90°C
Non-heat treated	0.00002	0.00028	0.00955
Heat treated	Nil	0.00003	0.00087

The average solution potential of duplicate samples of non-heat treated and heat treated P-10 alloy in Pile Process Water at the temperatures of the test are as follows:

Sample	Solution Potential Referred to a Saturated Calomel Reference Electrode - Volts 20°C	70°C	90°C
Non-heat treated	-0.68	-1.15	-1.55
Heat treated	-0.76	-1.26	-1.55

The non-heat treated samples were extensively attacked during the two week exposure. The type of attack was exfoliation, that is, grain boundry attack in which the corrosion products because of their volume expand the physical size of the metal. The greatest portion of the attack was confined to an annular section located near the periphery of the samples. This indication of segregation was not noted on the heat treated pieces.

Reports Issued:

1. Effect of Solution Heat Treatment on Hydrogen Pickup of Aluminum Lithium Alloys, J. W. Goffard, HW-21922.
2. KAPL-ANL-HW Cooperative Testing for Hydrogen in Metal Samples, J.W. Goffard HW-21923.
3. Solution Potential of "Heat-Treated" and "As-Extruded" P-10 Alloy, Y.B. Katayama, HW-22407.

URANIUM METALLURGY (HDA TP-6)

I. 3730 Metal Fabrication Laboratory - L.J. Lucas

The Metal Fabrication Laboratory has been reactivated on a full time basis.

Facilities are available for the rolling, forging, melting, heat treating, and machining of uranium and other metals. In the near future it is hoped that press forming, extrusion, and drawing will be added. Technical assistance is offered with regard to the designing of tools, dies, special machinery etc., and the routing, expediting and follow up necessary in the fabrication of such items.

II. Powder Metallurgy of Uranium - J.H. Bach

A report, HW-22359 was issued discussing the technical and economic advantages of producing Hanford fuel slugs by powder metallurgical methods. The report also suggests an overall program to test the feasibility of the proposed method. The first step of this program, concerning in-pile testing of hot pressed slugs, was initiated during a visit to Sylvania Laboratories on October 10, 1951, and is discussed in trip report HW-22442. It was decided that Hanford will receive 106 slugs prepared by various powder metallurgical processes; of these, 100 will be alpha canned and pile tested. Since the metal thus prepared has a very small grain size and is randomly oriented, it is expected to have better structural stability than rolled and heat treated metal.

III. Rolling Process Development - W.T. Kattner - J.W. Riches

A program for the development of a uranium rolling process and of other fabrication processes is being directed by personnel of the NYOO-AEC for production of rods and slugs in the Fernald, Ohio, Feed Materials Preparation Center. Two experimental rollings in this program were observed by Hanford representatives (HW-22347 and HW-22474).

Billet roughing is done in a 22 inch reversing mill at Allegheny-Ludlum, Watervliet, New York. Two sets of rolls have been tried for this purpose, one consisting of a pass sequence of squashed octogons and the other of ovals, squares, and rounds. Both of these designs, have been found to be unsatisfactory in that the reductions have been too severe and that the uranium bars need to be guided to prevent their rotation in the passes. Overfills and underfills were frequently produced using these roughing rolls. These defects result in production of discontinuities in the bars. A third set of Birdsboro designed roughing rolls conforming more nearly to the passes established by usage at Joslyn & Simonds is to be tried October 26 in preparing 100 rods for DuPont. If this experimental rolling proves satisfactory some Hanford diameter rods will be rolled using similar schedules, probably in November.

The rolling of the finished rods is done at the Lackawanna, New York plant of the Bethlehem Steel Company in a six stand continuous mill. Indications are that uranium rods produced from machined bars in such a mill after a salt bath preheat are quite uniform in quality, with regard to characteristics such as diameter uniformity, grooves, folds and seams in the metal, the amount of recrystallization, and orientation.

Both salt and lead baths have been employed in this development program for preheating of billets and roughed rods. The lead tends to alloy with exposed uranium while there is no apparent attack by the molten lithium-potassium carbonate mixture. The lead does not cling to the billets or rods except where alloying has occurred. Billets preheated in a bath of this metal tend to heat up considerably due to surface oxidation during the rolling. On the other hand, since the molten salt clings to the rod surfaces, the rods thus preheated may be rolled much more rapidly and with better temperature control since the oxidation is greatly reduced. This fact indicates that salt may be used advantageously at Simonds. Seven rods rolled from salt at Lackawanna have been received for a test to determine whether the lithium is removed in the chip recovery process. Data regarding clean up and yields will also be obtained on these rods.

An informal request has been received from NYOO for four to six twelve foot long gamma extruded rods for rolling in the continuous mill. At least four such rods will be shipped when a formal request is received from the AEC.

IV. Uranium Quality - W.T. Kattner - J.W. Riches

Three tests on eggs from Mallinckrodt had TDC values of 18, 20, and 23. Since these TDS values exceed the working maximum of 17, the rods will be given special consideration upon arrival at Hanford.

About twenty percent of the rods held in the Metal Preparation Section inventory are for a Special Correlation Test proposed by the AEC-NYOO. A suggestion was made that the AEC send no more material for this test until better information regarding the data wanted from these rods is obtained from AEC-NYOO. Representatives from the Metals Preparation Section, Mathematics, Pile Applications, and Metallurgy concurred in this suggestion. After these data are obtained and evaluated the Special Correlation Test program may be rescoped.

Observations of billets at Simonds Saw and Steel during the September rolling revealed that only about 15 per cent of the billets were free from cold shuts, scabs, and porosity. It is reasonable that the recent poor yield experience with rods rolled from Mallinckrodt billets may be attributed to these poor billet surfaces. This matter was discussed with the personnel in the AEC-NYOO who are taking an active interest in improving rod, and perforce, billet quality.

V. Mechanical Properties of Uranium R.E. Hueschen - C.L. Pleasance

The following results have been obtained on the five virgin, transformed (triple-dipped) uranium tensile specimens.

Spec	Ultimate Tensile Strength-Psi	Yield Strength (.2% offset)-psi	Modulus of Elasticity-10^6 psi	Elongation in 2"-%	Reduction in Area %	Hardness Rockwell B
TR 3	71,500	29,000	23	5	7	91
TR 4	83,500	30,000	25	9	10	87
TR 6	83,000	29,000	22	10	8	89
TR 6*		31,500	23			
TR 8	81,500	29,000	23	8	9	89
TR 10	83,000	30,500	25	13	8	91

* A SR_4 strain gage was also used to measure the strain induced in sample TR-6.

The low values for ultimate strength and ductility of specimen TR-3 could be due to an undetectable void or crack which produced a stress concentration. However, the grain size near the break, after the specimen had been stressed in tension was approximately .150 as compared to a grain size of approximately .200mm for all the other specimens. Since only one specimen is involved it is not possible to drew any conclusions as yet. However, specimens with varying grain diameters will be obtained for testing. The degree of preferred orientation of specimens will be obtained in an attempt to correlate b-axis alignment, prior to triple-dip heat treatment, with ductility.

Three as rolled, untransformed uranium specimens have been tensile tested. One specimen broke in the threads due to stress concentration produced by machining the threads too deep. The properties of the other two untransformed specimens are as follows:

Spec	Ultimate Tensile Strength-psi	Yield Strength .2% offset-psi	Modulus of Elasticity-10^6 psi	Elongation in 2"-%	Reduction in Area %	Hardness Rockwell B
UN 2	108,000	36,000	20	19.5	13.6	91
UN 3	107,000	30,000	21	16.4	17.4	92

The microstructure study showed both samples to be recrystallized uranium with a grain size of approximately .045mm. Orientation values will also be obtained to correlate preferred orientation with per cent elongation. Compression as well as tension testing will continue on this material. In an effort to obtain an exact value for Poisson's Ratio a method of using a series of strain gages at right angles to each other is being tried. It will first be tried with steel and if this is satisfactory it will be used on both compression and tension specimens of uranium. If this method is feasible, it is believed that high-temperature strain gages can be obtained for determining Poisson's Ratio (as well as measuring strain) at elevated temperatures. Remelt uranium will also be tested as well as as-rolled uranium exhibiting a cold worked structure. Thus far only samples cut from the center of a rod have been tested; further study will include cutting of specimens displaced from the center of a rod.

VI. Detection of Per Cent Transformation of Uranium Slugs-Metals Comparator Method - R.E. Hueschen - L.F. Higginson

The General Electric Metals Comparator has been received. The Metal Comparator works on a bridge principle, with variable resistors balanced against the coil with the sample inserted in the core. Any change in internal impedance of the test sample causes deflection of a microammeter; the impedance varying with eddy current losses on a non-magnetic material such as uranium. The reliability of this Comparator in detecting the volume per cent transformation of bare slugs will be determined. An impedance reading versus volume per cent transformation curve will be obtained with the variables involved, such as orientation, grain size, cracks or voids, oxide layer, and slug diameter correlated. A testing program for this work was outlined by R.E. Hueschen in HW-22361.

Seventeen remelt and seventeen virgin uranium slugs which were acceptable for canning were obtained from the Manufacturing Department. Thirty-four rejected slugs (slugs with holes or cracks greater than 1/16" deep) were also

obtained. Comparator readings of all these untransformed, as-machined slugs were taken with an arbitrarily chosen virgin slug used as a standard. Preliminary readings show a marked increase in impedance for a badly cracked slug. In general, the rejected slugs showed more impedance than those slugs accepted by Manufacturing Inspection. In other words, a cracked slug would be more likely to be rejected by the Comparator than a slug with no cracks, both slugs having the same per cent transformation. Six completely transformed slugs were obtained for preliminary study. All of these six slugs showed much less impedance than any of the untransformed slugs. This is in line with findings in the KAPL 460 report, by D.W. White. A number of selected slugs will now be partially and completely transformed by the salt bath method and Comparator readings will be taken after heat treatment and again after pickling.

VII. Thermal Expansion-Automatic Recording Dilatometer - R.E. Hueschen

A new Leeds and Northrup slide-wire has been ordered to extend the measuring range of the present Micro-max recorder to 1000°C so that the gamma phase can be investigated. Samples taken adjacent to the specimen used in the mechanical property study will be run as soon as the Brown "Electronic" recorder is repaired. The following data has been compiled from dilatometeric runs made by R.B. Socky on alpha rolled triple-dipped uranium. This data has been documented in the form of a letter to M.W. Carbon, HW-22465.

VIII. Linear Coefficient of Thermal Expansion Data - R.E. Hueschen

Sample	Linear Coefficient of Thermal Expansion* Alpha Uranium- 10^{-6} in/in °C		Alpha-Beta Transformation Temperatures- °C		Linear Coefficient of Thermal Expansion- Beta Uranium- 10^{-6} in/in °C	
	Heating	Cooling	Heating	Cooling	Heating	Cooling
T 1	16.5	17.2	662	641	22.7	23.3
T 2	15.8	21.3	657	645	22.3	21.0
T 3	16.2	18.8	663	648	22.7	18.9
RT-L 1	20.1	16.2	661	648	22.3	22.3
RT-L 2	17.1	16.6	659	648	22.8	22.9
RT-L 3	17.8	17.7	660	649	23.5	23.6

* This is an instantaneous coefficient taken at the true median (312.5°C).

Dimensional Changes on Heating Alpha Rolled, Triple-Dipped Uranium From Room Temperature to 720°C

Sample	Expansion--Alpha to Beta Transformation- 10^{-3} in/in	Contraction--Beta to Alpha Transformation- 10^{-3} in/in	Net Change--Room Temp. to Room Temperature 10^{-3} in/in
T 1	3.0	3.8	-0.6
T 2	2.0	3.6	-5.0
T 3	2.6	3.5	-0.2
RT-L 1	2.3	3.7	+0.7*
RT-L 2	2.7	6.5	-2.5
RT-L 3	3.0	4.5	-1.5

* The large alpha coefficient on heating of specimen RT-L 1 indicates initial random orientation. Continued study is needed to establish the validity of this net expansion. It is believed possible that the degree of preferred orientation prior to the triple-dip (beta) heat treatment could be correlated with these net changes.

IX. Processing of Uranium Rolled at 300°C to 600°C - L.A. Hartcorn

Approval for pile irradiation of this material has been delayed outside of Metallurgy since August 1951.

X. The Effects of Fabrication Procedures on the Pile Behavior of Uranium - L.A. Hartcorn

The literature survey being conducted is about 60 per cent completed. The data will be presented at a forthcoming Technical meeting of Metallurgy Personnel on November 28, 1951.

XI. Orientation Studies - M.J. Sanderson

Production Test 313-105-1-M is designed to test the pile behavior of uranium slugs of known crystallographic orientation. The material to be used in this will consist of six slugs from each of the following rods:

1. "Hot Rolled" Lockport Material (Q 6569 - Q 6570)
2. "Cold Rolled" Lockport Material (Q 6593 - Q6594)
3. "Continuously Rolled" Joslyn Material (Q 8587)
4. "Low Temperature Rolled" Lockport Material (Q 7411 - Q 7412)

This material has been machined to standard size Hanford slugs and identified by appropriate stamping. Adjoining each slug a 3/8" wafer and a one inch wafer were collected for orientation studies and thermal cycling experiments. The results of the thermal cycling tests to be conducted at KAPL will be correlated with the actual pile behavior noted for this material.

The "hot" and "cold" rolled Lockport material shows a preferred orientation with the (020) pole parallel to the rod axis. The "psi" values obtained from wafers which were cut such that the surfaces examined were inclined 5°, 10°, 15°, 20°, 25°, 30°, and 35° to the rod axis indicates that this is the only orientation noted for this material thus far. The results of orientation studies on the remaining material should be completed within a week.

METALLURGY OF HANFORD STRUCTURAL MATERIALS RDA TP-7

I. A Technique for the Study of Local Anodic and Cathodic Areas on Metal Surfaces - K.L. Sanborn

In a paper presented before the Electrochemical Society in Detroit, October 11, 1951, a technique was described by J.B. McAndrew and associates of the Armour Research Foundation, for the study of local cell elements on metal surfaces. The technique is particularly well adapted to the study of canned slugs and will be described in a forthcoming report. Details have been discussed with R.S. Dalrymple who plans to evaluate the technique for possible applications in his work.

II. Corrosion Tests Recuplex Program - K.L. Sanborn

Corrosion tests in support of the design of facilities for the Recuplex process are well advanced. Proposed alterations in the flowsheet may require re-evaluation of conditions to be met in the dissolver. Boiling tests in CAW from combined feeds have extended to six 48 hour runs for

samples exposed in the liquid and three 48 hour runs for samples exposed in the vapor. Corrosion rates of the order .0003 inches/month or less have been recorded for sensitized 309 SCb, 347, and 304 ELC stainless steels in this environment. Corrosion rates were nil for types 304 ELC (sensitized) and 316 (as-received) in an agitated two phase system simulating CA column conditions at the feed point. The test program outlined in HW-21988, Proposed Corrosion Testing Program for Recuplex Program, will be complete November 1, 1951 except for two parts for which flowsheet alterations are pending.

III. Blend Tanks C02, 003, UR Tank Farm - K.L. Sanborn

Laboratory work on this problem has been completed and the final report is in rough draft form. The welds were found to be satisfactorily resistant to the process environment.

IV. Splash Ring Failures, 200 Area - K.L. Sanborn

Work will be continued on the preparation of photomicrographs to be included in the report on this problem.

V. Cast Melt Pots UO_3 Process - K.L. Sanborn

No information has been received concerning the heat treatment of the cast melt pots. Corrosion tests are scheduled for completion November 1, at which time an attempt will be made to correlate corrosion resistance and heat treatment.

VI. Influence of the Presence of Mercuric Ion on the Corrosion of 200 Area Dissolvers - K.L. Sanborn

A study of the effect of the presence of small amounts of mercuric ion on the corrosion of dissolvers in the 200 Areas was undertaken during the month. Tests are being conducted on welded and unwelded type 309 SCb in solutions containing 10^{-2} and 10^{-3} molar mercuric ion and in a control solution containing no mercuric ion.

VII. Corrosion Test Program, 200 Area Waste Storage Facilities -K.L. Sanborn
R.L. Beede

Corrosion tests were further delayed pending arrival of DeKhotinsky type thermo-regulators. It has been decided to start the tests using Variacs, with which it is anticipated the temperature can be controlled to within $\pm 3^{\circ}C$. The test program will start by November 1.

VIII. Anodizing of Aluminum Slug Jackets, A Survey - D.L. Zimmerman

A literature survey of past and proposed work on the anodizing of aluminum slug jackets has been completed. The first test carried out at Hanford Works indicated that the artificial oxide coating produced on 2S aluminum by the conventional anodizing process slowly dissolves when exposed to Pile Process Water at elevated temperatures. More recent experiments employing anodized dummy slugs, and at a lower temperature, indicate that these slugs have good corrosion and abrasion resistance and that the dying of anodic coatings with inorganic pigments for identification purposes may be feasible.

A more complete program of study has been proposed and includes the investigation of the corrosion and abrasion resistance of various types of anodic coating, the testing of several inorganic pigments, and the determination of the effect of anodic films on pressure drop in Process Tubes. The details of this survey are contained in Document HW-22444, Anodizing of Aluminum Slug Cans.

IX. Impurities in Aluminum Alloys and their Effect on the Corrosion Resistance of 2S Aluminum - D.L. Zimmerman

A literature survey was performed on this topic and reported to W.L.Schallid in a letter dated October 15, 1951. A list of impurities, obtained from recent spectrochemical analyses of aluminum slug cans and caps, (not commercial 2S aluminum) and single electrode potentials of a few of the alloy constituents resulting from impurities in aluminum, were included.

X. Instrumentation - R.S. Dalrymple - Y.B. Katayama - D.L. Zimmerman

Several instruments pertinent to pipe line corrosion survey work have been ordered. Development work has begun within the Corrosion Group on instruments and methods required for the expanded electrochemical studies program.

XI. Effect of Thermal Gradients on the 2S-72S Galvanic Couple in Pile Process Water - R.S. Dalrymple - Y.B. Katayama

An investigation is in progress to determine the effect of thermal gradients on the 2S-72S aluminum galvanic system in Pile Process Water. Preliminary experiments indicate that the 2S aluminum component will change from cathodic polarity to anodic polarity when the temperature differential between the 2S and 72S electrodes (temperature of 2S minus the temperature of 72S) goes from negative to positive. The transition point varies with the temperature of the 72S electrode. The exact transition point will be determined as a function of the temperature of the 72S electrode. The current flow between the two electrodes increases rapidly and non-linearly as the temperature differential increases positively. From the current flow, the corrosion rates will be calculated at the various temperature differentials.

XII. Corrosion of 2S Aluminum in Potassium Tetraborate Solution - R.S. Dalrymple
Y.B. Katayama

The corrosion rate of 2S aluminum in 10% potassium tetraborate solution and in filtered water from the 100 Areas was determined by immersing duplicate samples in the two electrolytes at 40°C for a period of two weeks. The effect of 1000 ppm of potassium dichromate, on the corrosion rates, in the above solutions was also determined. Results indicate the following:

1. 2S aluminum has an appreciable corrosion rate in potassium tetraborate
2. The addition of 1000 ppm of potassium dichromate to the above system reduces the corrosion rate by a factor of approximately twenty.
3. During the period of the test, 2S aluminum in filtered water, with or without 1000 ppm potassium dichromate, was not attacked.

XIII. The Influence of Impurities on the Solution Potential of Weld Beads on Canned Slugs - R.S. Dalrymple - Y.B. Katayama

Spot solution potential measurements along the weld bead on canned slugs are being contemplated. Spectrographic analyses are to be obtained on those areas where solution potentials will be measured in order to determine the impurities present. A correlation will be attempted between the solution potential and the spectrographic data.

XIV. Specific Resistance of Influent Pile Process Water - Y.B. Katayama

Graphs were drawn showing the daily fluctuation of the specific resistance of influent Pile Process Water from tank #4 at 190-H Building at temperatures of 25°C, 50°C and 90°C. The graphs and an accompanying letter were submitted to W.R. Conley, Jr. to determine if a correlation exists between specific resistance and raw river water composition, chemical feed, or other pertinent data.

XV. Solution Potential Measurements on Process Tube Samples - C.H. Pitt - R.S. Dalrymple

A type K-2 Leeds and Northrup potentiometer has been set up in the Radiometallurgy Building at 100-B Area to make solution potential measurements on samples of irradiated process tubing. The samples are to be located in the solution behind lead shielding so that the operator of the equipment will be protected from radiation. A N/10 calomel reference electrode with an attached salt bridge is being constructed for use with the equipment. The reference electrode will be placed on the 'safe' side of the lead shielding to prevent possible radiation effects on the standard cell. Preliminary measurements are now in progress.

XVI. Examination of a Nozzle Assembly with a Stuck Cap Supported Shielding Piece C.H. Pitt - R.S. Dalrymple

After removing the shielding plug from the nozzle by means of an hydraulic press, the shielding piece and the nozzle were visually examined for corrosive attack. In addition, the nozzle was cross-sectioned through one of the pits and metallographically examined. A report on this examination is now in preparation.

XVII. Examination of Rear Sections of Process Tubes 0890-F and 0893-F - C.H. Pitt - R.S. Dalrymple

Because of suspected leaks in the rear sections of the captioned tubes, it was decided to slit open the last 7-9 feet of each tube to determine the interior condition. Visual examination of the uncleaned pieces revealed only the usual brown film.

Chemical removal of the brown film from one of the tube sections revealed extensive pitting of the 7.S cladding especially between the ribs. The pitting did not appear to extend below the cladding. No perforations were found in the sections examined.

XVIII. Flow Cup Tests at 100-D - R.S. Dalrymple

Because of space considerations it has become necessary to remove the Flow Cup facility from the 105-D Corrosion Laboratory. As suitable space is not immediately available, the current corrosion tests will be terminated after approximately seven months exposure. At that time, a report will be issued summarizing the results of the investigation. Possible sites for an expanded corrosion laboratory in the 100 Areas are being investigated.

XIX. Properties of Boron Stainless Steel Safety Rods for C Pile - R.E. Hueschen C.L. Pleasance

Three samples of 2% boron stainless steel from the Electric Steel Foundry Co. were tested for the Reactor Unit of the Design and Construction Section. The tests were made to determine whether the material met General Electric's specification HW-4794. The samples were machined into five specimens; two ASTM 5/16" round tensile bars and three flat specimens with a 0.125" x 0.375" cross-section. A Baldwin SR_4 gage was used to record strain. The specifications for this material and the results of the tests are given in the following table:

Specimen	Specimen Type	Ultimate Strength-psi	Yield Strength Psi(.2% offset)	Elongation-% (2 inches)	Rockwell Hardness *
KB 3-1	Round	69,500	49,000	1.2	Rc 21 (Brinell 231)
KB 3-2	Round	58,500	49,000	1.2	Rc 21
KB 3-2	Flat	55,500	49,000	1.2	Rc 21
KB 2-2	Flat	60,000	43,000	2.3	Rc 20(Brinell 226)
KB 2-2	Flat	64,000	48,500	1.6	Rc 20
G.E. Spec.	Flat or Round	81,000**	50,000**	1.5**	Rc 32 (Brinell 300)

* All hardness values quoted are for information purposes only.
** Minimum Values

At a subsequent meeting with representatives of the steel company, it was decided to change the fabrication of the centrifugally cast boron stainless-steel pipe from sand-molds to permanent type molds. Permanent molds were used by the United States Pipe Co. in casting the vertical safety rods now in use.

A sample of pipe cast in a permanent steel mold will soon be received for testing. In addition to the original tests, an analysis of the micro-structure in the new sample will be made and compared with samples of the material presently in use on the project.

XX. Zirconium - J.H. Bach

R.C. Dalzell, AEC Washington, called a meeting to discuss the progress made on the evaluation of various Zr-Sn alloys. The sense of the meeting may be summarized as follows:

(1) The corrosion resistance of good zirconium is not improved by tin additions.
(2) Although the corrosion resistance of inferior grades of Zr are improved by the addition of Sn such alloys are still inferior to the best grade of crystal bar now produced by WAPD.
(3) Even small additions of Sn will decrease the ductility and workability of zirconium considerably.

(4) USBM ingots still contain more impurities, particularly graphite, than any other grade of metal. This severely limits the workability of this type of zirconium.

KAPL has shipped 20 lbs. of grade II [illegible] to HAPL. Two [illegible] experiments on process tube fabrication. We have requested KAPL to send a small amount of this metal to Hanford for 305 reactivity testing.

XXI. Aluminum - J.H. Bach - P.J. Pankaskie

A thorough review of all available aluminum alloy data was started to determine the feasibility of obtaining process tubes with better mechanical and corrosion properties. A Trip Report, HW-22213 was issued on the consultations with Dr. F.N. Rhines of the Carnegie Institute of Technology and various Alcoa metallurgists.

XXII. Experimental Equipment - J.H. Bach - P.J. Pankaskie

Detail design on the "Uniscan" unit has been completed and construction will be started as soon as the necessary parts and raw material can be obtained. Construction was started on the pressure creep apparatus. All the equipment for it, except the high pressure gages, is now on the project.

Reports Issued

1. Flow Laboratory Corrosion Tests of Various Metals and Alloys in Pile Process Water, R.S. Dalrymple, HW-22365.
2. Thickness of 72S Aluminum Cladding on Hanford Process Tubes, L.A. Hartcorn HW-22406.

RADIOMETALLURGY - FACILITIES (RDA TP-8 PART I)

I. Double Crystal X-Ray Spectrometer - M.J. Sanderson - W.V. Cummings, Jr.

Numerous experimental trials have been conducted using the double crystal spectrometer in its original design. Various changes in the collimating system and the geometry of the unit have proven unsuccessful in producing a suitable spectrum of a uranium sample. The inefficiency of the quartz crystal diffracting the beam out of its original plane makes it impossible to record the less intense diffracted beams. It seemed advisable to modify the design so that the double diffraction process occurs in the same plane. Using this method with the crystals placed in the "parallel" position has given encouraging results. A spectrum of a uranium sample has been reproduced and "psi" values calculated from this pattern are in good agreement with those which were obtained from the same sample from a single diffraction process.

Several improvements of the design are felt necessary. An integrating sample holder similar to the one being used on the Phillips unit will be ready soon and should give a more reproducible pattern. In order to increase the intensity of the second diffracted beam the use of a bent crystal for focusing and better collimating slits are being considered. The problem of adequate shielding for the G.M. tube is also being studied.

the slug. Water quenching from 750°C resulted in an average warp of 0.003 inches from the center line of the slug.

Water quenching from 683°C reduced the grain size around the periphery of the slug; however, the grain size at the core is comparable to triple dip canned metal.

V. Etchants and Cleaning Agents for Use in the Canning Process - R.S. Dalrymple, D.L. Zimmerman

Laboratory testing of a number of commercial aluminum etchants and modifications of these etchants is nearly complete. The most promising of the formulations are to be more closely evaluated as to the effect of temperature on rate and type of attack. Following completion of the laboratory tests a report will be issued.

VI. Non Destructive Inspection of Canned Slugs by X-Ray Techniques - M.J. Sanderson

The feasibility of using some x-ray diffraction technique for measuring the Al and/or Al-Si thickness of canned fuel material is being considered. The receipt of a silver target tube for the XRD unit provides the necessary tool for experimental work on this project.

VII. Beryllium Additions to Aluminum-Silicon - L.A. Hartcorn, L.S. Reed

The microstructures of Al-Si samples with and without additions of beryllium were examined metallographically. No definite change was noted in the appearance of the eutectic alloy and no refinement of the primary aluminum crystals in the matrix material was accomplished. From this investigation it appears that the advantages of adding small percentages of beryllium to the aluminum-silicon alloy are confined to the non-oxidizing characteristic of a molten bath when left undisturbed in air.

The health hazards involved in using beryllium additions to aluminum-silicon alloys are still questionable. A rather thorough search for information on this subject revealed no conclusive quantitative results on dosage, particle size or percent of beryllium in the alloy which could be considered objectionable or fatal from a health standpoint. Investigators do agree, however, that the hazards are serious.

VII. The Effect of Temperature on the Grainsize of Triple-Dipped Uranium - L.A. Hartcorn - L.S. Reed

Ten alpha-rolled uranium slugs were triple-dip canned using the normal time cycle but at various bronze bath temperatures, to determine the effect of temperature on the grain size. Five control slugs were heated to a temperature of 739°-741°C; the remaining five were heated to 739°C, 745°C, 750°C, 756°C, and 760°C, respectively. A sample from each slug was examined metallographically and, without exception, the average grain diameter was found to be 0.150 to 0.200 mm. From this limited investigation it may be stated that the grain size of alpha rolled slugs is not appreciably affected by triple-dip canning within the temperature range employed.

VIII. Materials for Tools Exposed to Molten Al-Si - L.A. Hartcorn

We are waiting for additional samples of carburized steel which are being exposed to the environment of molten Al-Si by Industrial Engineering. When these samples are received, a metallographic examination will be made to

determine Al-Si penetration.

PLANT SERVICE WORK

I. Investigation of Connector Screw Failures in the Redox Building-L.A. Hartcorn

Three one-inch diameter connector screws from the 202-S Building were received by Metallurgy for an examination. The connectors, fabricated from type 440-A stainless steel, had failed under the force of impact loading. Specifications for the screw required surface hardening of the 2" hex nut and 1" screw to about 500 Brinell (Rockwell C 50).

An examination of cross-sectional and longitudinal samples cut from the connectors revealed that the screw portion was hardened throughout. In addition, hardness traverses and macro-etched samples showed that an extremely sharp contrast in hardness existed in the area between the nut and threaded portion of the connectors. It was at this line of demarcation that failure occurred. Unbroken connector screws were also examined and the same hardness pattern was prevalent. In the course of the investigation it was revealed that type 440-A stainless steel has an inherently low impact strength in the hardened condition. Plans are now under way to remove all the 1" connectors fabricated from 440-A stainless steel. A low alloy steel of high impact strength (AISI 4140 steel) is being considered as replacement material for these connectors.

II. Routine Corrosion Studies - K.L. Sanborn

Ten Huey tests on stainless steels being used in the construction of the Hot Semiworks facilities were conducted during the month.

III. Identification of Unknowns by X-Ray Diffraction - M.J. Sanderson

Identification powder patterns of specimens submitted by the Chemical Research Group proved that the material in question was amorphous thus making qualitative analysis impossible by x-ray techniques.

Samples which are presently being collected by the Health Instruments division are being examined for the presence of uranium oxides.

IV. Compression Test of Shielding Material - R.E. Gueschen - C.L. Pleasance

Pile Physics was assisted in compression testing of iron shot embedded in Portland Cement. Six irradiated and six non-irradiated specimens were tested using SR_4 strain gages. After six samples were broken, the tests were discontinued due to erratic results. A different technique will be tried. The tensile machine was also used for the compression testing of masonite.

W. L. Schalliol
Group Head

234-5 METALLURGY GROUP

PLUTONIUM METALLURGY PROGRAM (RDA TP-16)

I. Development of Laboratory Facilities - T.C. Nelson, Jr.

The September Technical Activities Report of the 234-5 Metallurgy Group, (HW-22280, page 16) reported that allocation of space for certain metallurgical activities was pending. A meeting was held between Separations Technology and Pile Technology early in October at which time our request for room 184 and the assembly bay was refused on the basis that existing space would physically house the equipment. However, an offer to allocate part of corridor 8, all of room 176, and corridor 9 was made.

Considerable time was spent by our group in determining the feasibility of locating our equipment in the area described. At a meeting held on October 12 between G.E. McCullough, R. Ward, and T.C. Nelson this plan was submitted as per the attached drawing. Although this space was not the most desirable, nor comparable with the space requested, it was felt that the aforementioned areas could be used when certain construction changes were made. Drawings to this effect were prepared and submitted for comment and approval.

Estimates were obtained, a work order to cover the work was written and turned over to Mr. R.T. Jessen, of 200-W Plant Engineering. Mr. Jessen is currently presenting this proposed alteration to the work authorization committee. They will determine whether operational or construction forces will do the work.

Construction Report: Work is progressing at a more rapid pace with the advent of a large number of workmen on the job. The estimated completion date is still problematical but expected to be sometime in December. A few unexpected problems have arisen such as the waste water drains from the high vacuum equipment. The "hot" exhausts from the mechanical pumps and several other minor difficulties.

Hood 17 and the Tukon hood arrived and are currently being installed. The Kodak 5 x 7 Master View Camera and accessories also arrived during the month.

II. Space Planning - B.O. Reese

The proposed use of Corridors 8, 9, and Room 176 in the 234-5 Building has required a number of layouts of furniture and laboratory equipment to determine an optimum arrangement. Relocation of the present equipment and office space of Rooms 187, 183, and 182 into the new space will provide greater floor space, but with somewhat less convenience of access than at present. The Hauserman panel construction of the building allows a convenient and inexpensive remodeling technique for the new partitions and doorways required. Electrical, lighting, plumbing, and hot ventilation needs have been detailed to allow for tie-in with existing lines as much as possible; for example, the X-ray cooling water and drain lines require only 40 inches of pipe to be laid on the floor from the adjacent dark room facilities.

III. Tensile Test Machine - B.O. Reese

Spherical seat fittings have been designed for the Detroit 20,000 lb. tensile test machine which is to be installed in Hood 17 of the laboratory. A shop drawing is prepared for the A.S.T.M. std. tensile test specimen to be used

for the calibration of the machine against the 60,000 lb. machine at 300 Area, pending the arrival of CR_4 strain gages on order from the Baldwin Lima-Hamilton Company. A supply of both simple and rosette type gages is included in the order to cover future stress analysis programs.

IV. Calibration of Metallograph - L.P. Morgan

Work has been started on the calibration of exposure for the metallograph, using Kodak Contrast Process Ortho sheet film. With the filters removed, a reading is taken at the eyepiece with a General Electric DW-68 Exposure Meter. The filters are replaced and sixteen exposures was made on a single 5 x 7 sheet, differing by a constant increment of exposure time. The development film will be examined with a densitometer to determine the density desired. A chart of exposure time in seconds versus foot candles will be made on log-log paper. From the straight line obtained for each magnification it will be possible to determine the correct exposure time for any meter reading. Because of the low illuminating power of the tungsten filament light source it is planned to use a more sensitive meter. It is apparent that a more powerful light source, such as a carbon arc, will be necessary for higher magnifications.

V. The Thickness of Films on Metals - M.D. Freshley

A method for determining the thickness of thin oxide films which form on metals is being investigated.

The method that is being considered depends on the fact that the character of polarized light reflected at the surface of the metal is dependent on the thickness and nature of the film which is on this surface. Absolute measurements of the thickness depend on a comparison between the reflection of film-covered and film-free surfaces. A metal that is absolutely free from films must be obtained or the true optical constants must be known. If it is not possible to obtain a film-free surface and the true optical constants of the metal are unknown; information of an approximate nature can be provided regarding changes occurring in the thickness and nature of the film.

This method is capable of measuring the mean thickness of a film and does not apply to the measurement of the thickness at a given point. Also, there are other factors which must be considered such as the age and stage of growth of the film and complications due to adsorbed gases.

P. Drude has found that quantitative investigations are limited to films of the order of 20-100°A, which are fairly uniform in thickness and optical density, and which are deposited on such a metal that there exists a sharp interface between the two media.

The adaptability of the Bausch and Lomb Research Metallographic Equipment for this purpose is being investigated. This instrument is equipped with a special Elliptical Vibration Compensator with which the state of polarization of the reflected light can be determined. The state of polarization is in turn a function of the film thickness for thick films of the order of 200-400°A. The instructions that were supplied with the Elliptical Vibration Compensator are not too explicit as to its use, so an additional article has been requested.

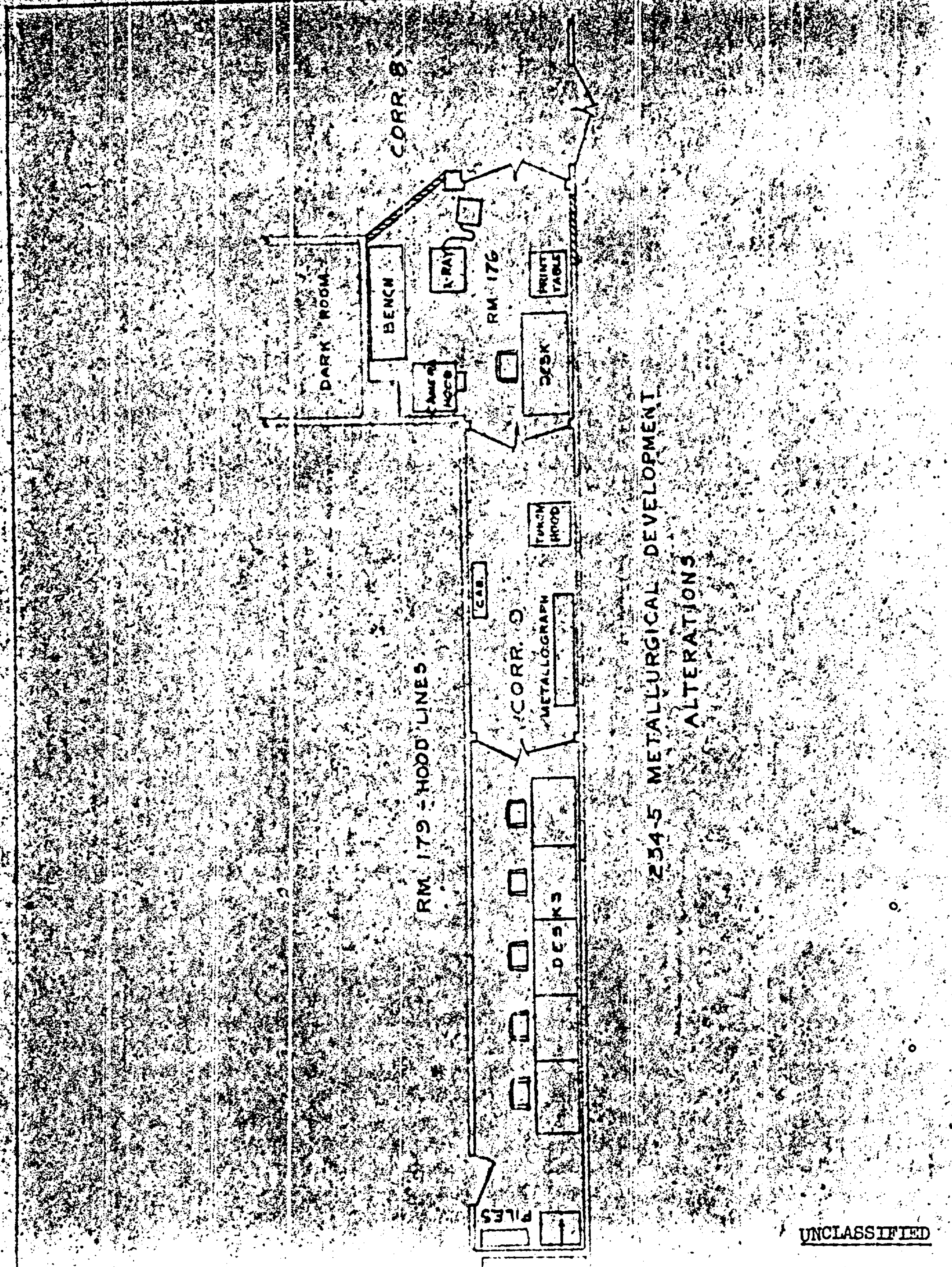
Much investigation remains to be done concerning this method of determining the thickness of films on metals.

VI. Cathodic Etcher - M.D. Freshley, LP Morgan, - B.O. Roose

Electrical etching of specimens has suggested a means of avoiding the extreme oxidation problem anticipated in the laboratory program. The work previously done on cathodic etching at the Ford Foundation and at Distillation Products Industries has been carefully reviewed. The D.P.I. apparatus commercially available has been rejected, since the rubber gasketing, lack of cold trap, and difficulty of flushing the vacuum system suggest probable specimen contamination. The current design project for a cathodic etch apparatus is particularly concerned with the elimination of oxygen, hydrocarbons, and/or other active elements. An inert atmosphere of argon will be provided, with control and instrumentation for rapid flushing. While operating pressures may be of the order of 25 microns, the design anticipates much lower vacuums in the consideration of material vapor pressures. Both AC and DC arcs will be used in order to compare etch quality. Test operation will be on a stand, but eventual hood installation is anticipated in the design. The diffusion pump and AC transformer have been received and some of the instrumentation is on order.

O.J. Wick
Group Head

WLS:OJW:hh


CORR. B
DARK ROOM
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X-RAY
RM. 176
PRINT TABLE
DESK
FUME HOOD
CAB.
CORR. D
METALLOGRAPH
RM. 179 - HOOD LINES
DESKS
FILES
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ALTERATIONS