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QUARTERLY PROGRESS REPORT

FOR PERIOD ENDING APRIL 30,1950

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METALLURGY DIVISION

J. H. Frye, Jr., Director

QUARTERLY PROGRESS REPORT

for Period Ending April 30, 1950

Edited by

W. H. Bridges and E. C. Miller

DATE ISSUED AUG 4 1950

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SUMMARY

Hanford Slug Program. The thermal cycling of gamma-extruded and alpha-rolled bare uranium rods has shown no significant change in dimensions with thermal gradients, center to outside, in the range 220° to 350°C. It is consequently concluded that thermal cycling does not duplicate Hanford reactor distortion.

Thorium Program. Thorium alloys containing 2%, 4%, 6%, and 8% of Be, Si, Ce, Ti, Zr, In, Nb, Mn, and Al have been prepared and are being tested for workability. Ce, Mn, Cr, and Be produce no significant change in hardness over annealed thorium. Si, In, and Al increase the hardness, Nb decreases the hardness, and Ti and Zr decrease the hardness considerably. Pure thorium has been determined to have a modulus of elasticity of 10.3×10^6, a Poisson's ratio of 0.27, and a shear modulus of 4×10^6. In general, tensile tests yielded the following data:

60-65% elongation in two inches
69-76% reduction in area
30,000-35,000 psi tensile strength
63,000-69,000 psi ultimate strength
20,000-26,000 psi yield point

Materials Testing Reactor Component Development. The yield of sound fuel plates for the Materials Testing Reactor Mock-up has been greatly improved, allowing the completion of the fuel assembly units without excessive rejections. Plans for the fabrication of the fuel sub-assemblies and control rods for the Bulk Shielding Facility are being made. The beryllium reflector sections for the Materials Testing Reactor made of hot pressed metal were drilled by the gun barrel technique equally as well as extruded metal.

The creep tests on lead have been redesigned and curves for temperature and stress at 100°, 150°, and 200°C and at 30, 45, 60, and 90 psi are to be obtained.

ANP Materials Program. The exposure of various materials to static lithium and bismuth at 1000°C to select and study materials suitable for the construction of a high temperature, liquid metal cooled reactor has shown cobalt, nickel, L605, Vanadium, and 1040 steel in lithium, and chromium, cobalt

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and L605 in bismuth, badly attacked. The liquid coolant program is expanding to study the corrosion effects of lead, sodium, sodium hydroxide, and other possible coolants at this and other temperatures. Dynamic corrosion systems are under study and it is expected that thermal convection loops will be in operation shortly.

Radiation Effects Program. An investigation of the applicability of the "thermal spike" theory to the anomalous behavior of certain irradiated alloys has been started. The activation energy for the annealing out of radiation defects in a copper-aluminum alloy approaches closely the activation energy for the diffusion of aluminum in copper.

An apparatus capable of measuring creep under irradiation is being developed, and two different experiments for study of the effects of radiation on high temperature, liquid metal systems are being designed.

Preparation of CP-3 Fuel Rods. A study of the variables in the extrusion of 2% uranium-aluminum alloys has developed the optimum extrusion conditions of die design, billet and container temperature and extrusion rate.

FUEL SLUG STUDIES

HANFORD SLUG PROBLEM

The cycling of gamma-extruded and alpha-rolled uranium in the form of bare rods has not shown any significant changes in dimensions when the thermal gradient was of the order of 220°C, *i.e.*, a center temperature of about 290°C and a surface temperature of about 70°C (see Table I, test rod numbers 33 and 38). Similar rods of alpha-rolled uranium did not show appreciable distortion when they were cycled as many as 235 times with a center temperature of about 450°C and a corresponding thermal gradient of about 350°C (see Table I, rod number 39). The present data indicate that thermal cycling will not duplicate the distortion experienced in the Hanford reactor even at temperatures as high as 300°C in the center of the rod.

Attempts have been made during the past quarter to conduct cycling experiments at center temperatures around 500°C, since it was felt that this condition would produce distortion if it were possible to do so under experimental conditions. The major portion of the work has been carried out with alpha-rolled material. Alpha-rolled unannealed uranium bar was selected since it represents the structure most likely to produce distortion. The test rods were made by machining a reduced test section 15 in. long in a 1-5/8 in. bar. The resulting rod was then located so that the test section was near the exit end of the test unit to facilitate temperature measurements. The short test section was used in a 34 in. long bar to reduce the total load on the electrical contacts. The contacts were left as large as could be accommodated in the present test units. Long copper sleeves were shrunk on at the contacts to increase the area. After machining, rod numbers 33, 37, to 41 were electropolished, then placed overnight in a 20% $CuSO_4$ solution to deposit a thin layer of copper on the surface of the uranium. The plated rod was then installed in the cycling unit. The cycle included a 7½ min increase in power input to the peak, 7½ min at full power input, and 5 min for cooling.

The data presented in Table I indicate no distortion in gamma-extruded uranium, rod 33, after about 860 cycles at approximately 300°C. Cycling of an alpha-rolled test rod, No. 38, of approximately the same diameter (3/4 in.), did not show appreciable change after about 900 cycles at approximately 300°C

TABLE I

Thermal Cycling of Uranium Rods

ROD NO.	TYPE OF MATERIAL	NO. OF CYCLES	LENGTH OF CYCLES (min)	END LOAD (lbs)	CENTER TEMPERATURE (°C)	SURFACE TEMPERATURE (°C)	TEMPERATURE GRADIENT (°C)	*** CHANGE IN LENGTH	AVERAGE INITIAL DIAMETER (in.)	DIAMETER AFTER CYCLING (in.)	COOLING WATER VELOCITY (ft/sec)	CURRENT INPUT (amp/sq in.)	POWER INPUT (kw/ft)	REMARKS
33	γ-Extruded	0-115	20	5100	*275	55			1.203	1.204	32.3	11700	26.2	Rod was sprayed with "Spar Varnish" which peeled off. Arced under contacts.
		115-981	20	880	*290	75	215		0.7540	0.7537	25.6	18200	23.6	Contacts and center section were turned down. Copper sleeves installed on contacts. 0.05% $CuSO_4$ added to cooling H_2O. Uranium electropolished and chemical replacement Cu on bar from $CuSO_4$. Rod failed in contacts. No change in test section.
35	α-Rolled	0-100	20	4440	*400	60	295		1.1008	1.1007	29.3	13300	29.3	Small cracks appeared on surface. Cooling water was 0.1% $CuSO_4$.
		100-330	20	3470	*370	75	295		0.9994	1.0020	28.7	15800	31.0	Rod failed from corrosion cracking.
36	α-Rolled	0-42	20	830	*400	130			1.0010	**	28.5	15700	37.4	Rod split longitudinally over entire length of test section.
37	α-Rolled	0-133	20	910	375	80	295		1.0012	1.0002	29.9	16000	36.8	Electropolished and chemical replacement Cu plate. Small crack developed near exit end.
		133-319	20	1000	365	85	280		0.8785	0.8772	27.1	18300	34.3	Failed in contact. Extreme warp believed due to contact failure.
38	α-Rolled	0-296	20	750	390	80	310		1.0015	1.0000	26.8	15600	34.6	Electropolished and chemical replacement Cu plate. Small blisters appeared.
		296-1200	20	866	300	72.7	226		0.7503	0.7500	26.8	18415	24.6	Turned blisters off and replated. Rod still in fair shape. Some small cracks in test section.
39	α-Rolled	0-235	20	965	445	95	350		1.0035	1.0027	27.3	17500	44.9	Electropolished and chemical replacement Cu plate. Small crack developed in test section.
40	α-Rolled	0-34	20	1000	500	65	435		1.3586	1.3573	42.2	13700	51.0	Electropolished and chemical replacement Cu plate. Failed in contacts.
41	α-Rolled	0-23	20	950	465	60	405		1.3573	1.3560	38.2	13400	42.4	Same as No. 40 rod.

* Estimated Temperature

** Unable to measure

*** Length measurements were not consistent due to slight bows, etc., in the 18 in. span between center punch marks.

center temperature. (The No. 33 test rod had been cycled previously for 115 times at about 275°C center temperature, and the No. 38 test rod has been cycled previously for about 300 times at an average center temperature of 390°C.)

It was felt that distortion might be obtained if the surface temperature of the test section was raised to produce a smaller thermal gradient while the center temperature was maintained. This condition was used with No. 36 test rod. The rod split from corrosion as shown in Fig. 1, before any distortion data was obtained.

From the experience gained in the testing of rods 35, 36, 37, and 39, it was evident that a larger diameter test section would have to be used to overcome corrosion if the desired 500°C center temperature was going to be maintained for any large number of cycles, so test rods 40 and 41 were machined to about 1.36 in. diameter. During testing these test rods showed no appreciable corrosion or distortion after 34 and 23 cycles, respectively, previous to failure in the contacts.

It is believed that center temperatures of 500°C could be maintained if the thermal gradient test units were rebuilt with larger contacts. However, it appears very unlikely that even a 500°C center temperature would duplicate Hanford distortion. Since the relative amount of distortion obtained at Argonne Laboratory by externally heating to 300°C is small compared to that obtained when the samples are externally heated to 500°C, a temperature of 500°C might be more nearly equivalent to the temperature spikes obtained in pile operation. However, it is felt that such major redesign work is not warranted at this time.

It is planned to continue the study to include the cycling of one beta-treated test rod at approximately 300°C center temperature and to hold a fixed temperature of about 300°C center temperature on one alpha-rolled rod for approximately 400 hours. It is not anticipated that these tests will produce distortion but it is felt that these should be included for a complete study. This problem will be terminated after completion of these two remaining tests.

ORNL (CLINTON) SLUG PROBLEM

Fifteen Al-Si bonded ORNL-size slugs, which have operated in the ORNL reactor for five months, have shown no evidence of failure or irregularities as determined by visual inspection. These slugs have been operating at a maximum temperature of 330°C.

BARE URANIUM ROD
Split after 42 cycles – Input 37.4 K.W. – Current density 157,000 amps sq. in. – Calculated surface temp. 130°C – 830 lbs. end load – 0.005% $CuSO_4$ used for cooling

FIG. 1

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Technical assistance in the form of information about Hanford canning techniques has been given to the AEC and interested personnel at Y-12 in connection with the possible set-up of canning facilities at Y-12 to produce the required aluminum-silicon bonded slugs for the ORNL reactor.

Some further work has been done on determination of diffusion rates in U-Al sandwiches. The results have not yet been thoroughly analyzed, but seem to be the most consistent yet obtained.

In connection with the blistering problem in MTR fuel elements, a gas sampler has been devised to permit sampling of the gas contained in these blisters. It is planned also to use this device to sample gas in blisters produced on ORNL slugs by heating in the absence of radiation to temperatures of 450 to 550°C.

THORIUM PROGRAM

VACUUM ARC MELTING

Work is proceeding on the development of techniques for vacuum arc melting of thorium in a water-cooled copper crucible. A new vacuum system has been connected to the arc furnace and is now in operation. High speed recorders have been installed to record current, voltage, and pressure continuously. Two melts of thorium were prepared and all equipment functioned satisfactorily. Electrodes of 1/2 in. diameter Ames rolled thorium were used. A current of 800 amperes, with a potential of 20 volts, was required to melt 300 grams of metal in 30 seconds. Pressure during melting was about two microns, and about 19 cc of gas were liberated during melting. The resulting billets had bright surfaces with only a slight film over part of the top. It is planned to study possible changes in analysis and microstructure produced by arc melting.

ALLOY PREPARATION

Forty-six thorium alloys were prepared by vacuum induction melting Ames bar in ZrO_2 crucibles and allowing the metal to solidify in the crucible. Alloys were prepared with 2%, 4%, 6%, and 8% additions of the following elements: Be, Si, Ce, Ti, Zr, In, Cb, Mn, and Al.

The ingot was removed from the furnace and a flat surface was ground along the entire length and polished for metallographic examination and hardness traverses as tests for homogeneity.

Extruded thorium bar may be rolled readily, so the first attempt at working the alloy was by cold rolling the ingots without scalping and as solidified. Four alloys, 2% Zr-Th, 2% Be-Th, 2% Cb-Th, and 2% Si-Th, were rolled in this way and each cracked without appreciable reduction. The next series of alloys, consisting of ingots of pure thorium and 2% alloys of Mn, Al, Ti, Ce, In, and Si were given a soak-anneal in vacuum for approximately 18 hours at 700°C and then scalped to produce billets 0.875 in. in diameter.

The billets were cold rolled in round rolls. All of the specimens cracked badly after a few passes and rolling was discontinued. The only alloy showing appreciable ductility was a 2% Ti-98% Th alloy which was rolled flat (approximately 70% reduction) with severe edge cracking. Since control samples of thorium which were melted in the same manner as the alloys had picked up impurities (probably ThO_2) and cracked as badly as the other specimens, it was decided to break up the cast structure by a preliminary hot forging operation followed by cold rolling. A number of samples were scalped and jacketed in copper cans. Since thorium forms a brittle Th-Cu interface at elevated temperatures, the surface of the billet was coated with a BeO paint. This necessitated an additional vacuum anneal to drive off all moisture, after which the cans were welded.

When it was attempted to forge the first specimen, a 2% In-Th alloy, the weld broke and allowed some oxidation. The specimen was found to have cracked badly even though forged at 900°C. Rather than forge more specimens in the same type cans, the design of the copper can was changed so that the welded portion would not be subjected to the impact of forging. The inside surfaces of the can are to be chromium plated to prevent the formation of the Th-Cu interface. These specimens have not yet been forged.

The effects of the alloying elements upon the hardness of thorium are summarized in Table II. The hardness of the original thorium in the fully annealed condition is approximately R_b62. It may be seen that Ti and Zr decrease the hardness considerably, as does Cb to a lesser extent. On the other hand Si, In, and Al produce increases in hardness. The addition of Ce, Mn, Cr and Be resulted in little if any change in hardness.

The aluminum alloys spontaneously disintegrate after standing exposed to the atmosphere. A study is being made of this phenomenon and it appears to be due to the corrosion of a Th-Al compound.

X-ray diffraction patterns were obtained on both the solid material and the disintegrated material and showed complicated patterns which have not been fully interpreted. The complexity of the disintegrated specimen suggests the presence of a number of corrosion products in addition to the compounds formed in the alloy.

A new tilting vacuum furnace has been completed and is now in operation. The metal is super-heated 100-200°C and cast into a cold mold of zircon. The

ingots are of much better quality and larger than those made previously. More alloys will be made for hot forging to be followed by cold rolling.

Ingots are now being cast in suitable sizes for extrusion. Suitable tools have been made to allow extrusion of 1½ in. diameter billets.

TABLE II

Effects of Alloying Additions on the Rockwell B Hardness of Thorium

Average R_b hardness of starting Th = 62

ALLOYING ELEMENT	ALLOY CONTENT			
	2%	4%	6%	8%
Be	77	80	--	92
Si	92	98	102	80
Ce	60	57	52	60
Ti	2	8	4	37
Zr	24	--	41	36
In	80	85	86	80
Cb	54	46	62	--
Mn	65	60	71	70
Al	94	96	--	--

WORK HARDENING AND RECRYSTALLIZATION STUDIES

Studies of the changes in hardness associated with the rolling of Ames thorium and Westinghouse powder compact are being continued. The Vickers diamond pyramid hardness of the Westinghouse material increased from 65 for annealed material to 125 for specimens cold rolled 95.5%. On the other hand, Ames metal increased in hardness from 69, as annealed, to 110 after cold rolling 95%. There is considerable scatter in these hardness data. An apparent

anomaly exists in the curves of hardness versus cold work for these specimens. There is a significant drop in hardness at 80% cold work for the Ames metal and at 85% cold work for the Westinghouse powder compacts. Further work on similar specimens is planned.

MECHANICAL PROPERTIES

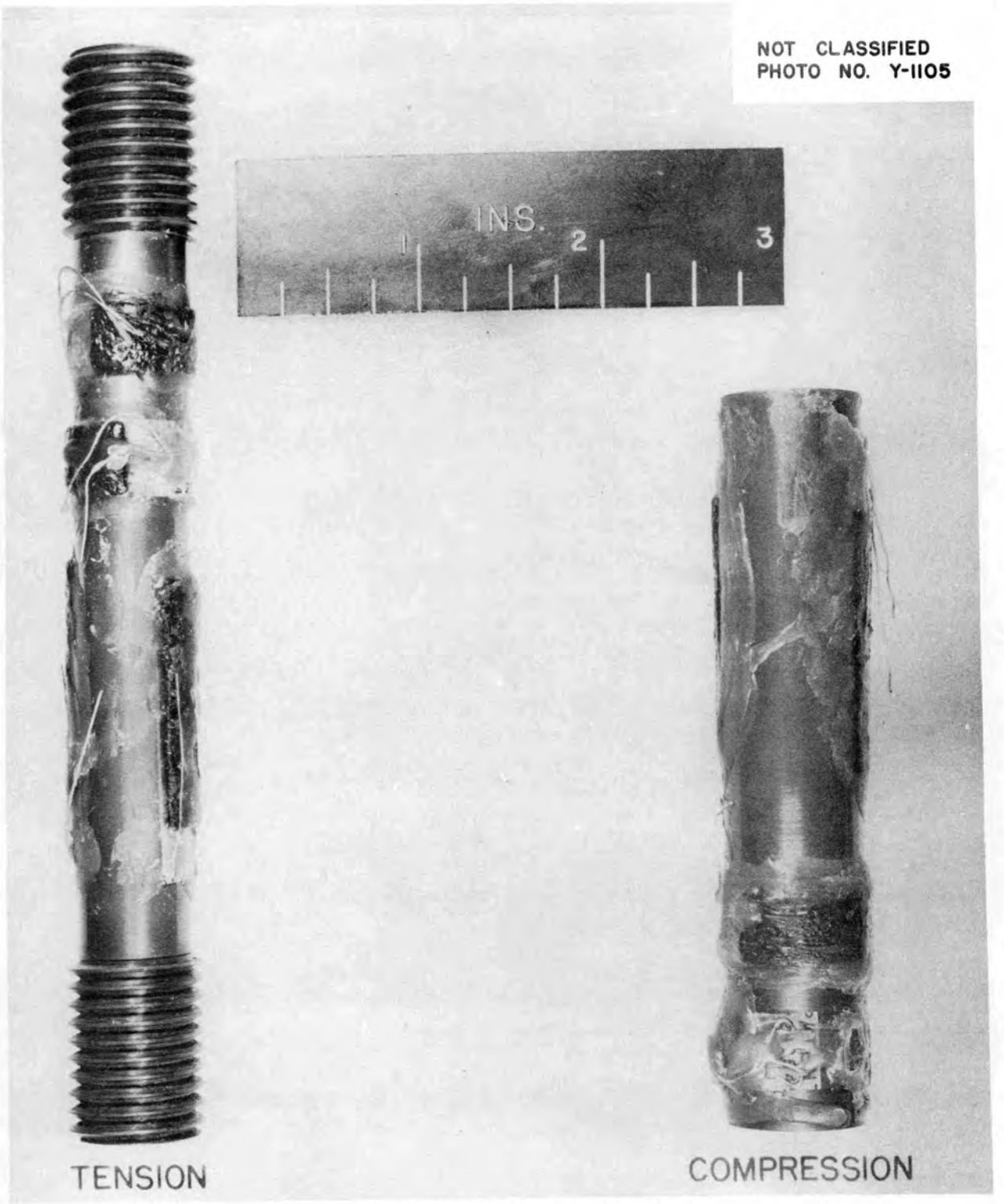
During the past quarter, the following mechanical properties have been determined: modulus of elasticity, Poisson's ratio, tensile strength, percentage elongation, and reduction in area. The material used was obtained as scrap from the thorium bars prepared for irradiation tests at Hanford. This metal was cast at Ames, forged at Battelle, rolled and machined at Westinghouse. A typical analysis of this material was as follows: 0.07% C, 0.15% O_2, 0.07% N_2 and traces of Ca, Fe, Si and Mg.

At Oak Ridge, the rods were annealed for one hour at 750°C, followed by rolling from 1¼ in. diameter to 13/16 in. diameter with a reduction of 0.020 in. per pass. Samples from this rolled stock were used for preferred orientation studies. It was found that there existed a poorly developed 210 fiber texture and a 111 fiber texture even less developed. Average grain size was found to be .04 to .05 mm.

The rolled bars were machined into tension and compression bars. After machining, the test bars were annealed at 800°C for 1/2 hour, polished, and four type A-1 SR-4 strain gauges were applied. The four strain gauges were placed on the specimen so two gauges would read the longitudinal strain and two gauges the lateral strain, Fig. 2. All precautions for the application of strain gauges were followed.

The specimens were tested in the 120,000 lb Baldwin Southwark tensile testing machine (Fig. 3). Lateral strain measurements were made with a SR-4 Wheatstone Bridge Control Box; longitudinal strain measurements were made with an SR-4 Type K Portable Strain Indicator.

After zero load readings were taken for all four gauges, the specimen was loaded to 100 lbs, the load was released, and zero readings were checked. The specimen was then loaded at intervals of 200 lbs up to 1000 lbs; above 1000 lbs the intervals were increased to 250 lbs. At each interval all four gauges were read and recorded with the load at that interval. The error in



Thorium specimens showing the SR-4, Type A-1 Strain Gages attached.

FIG. 2

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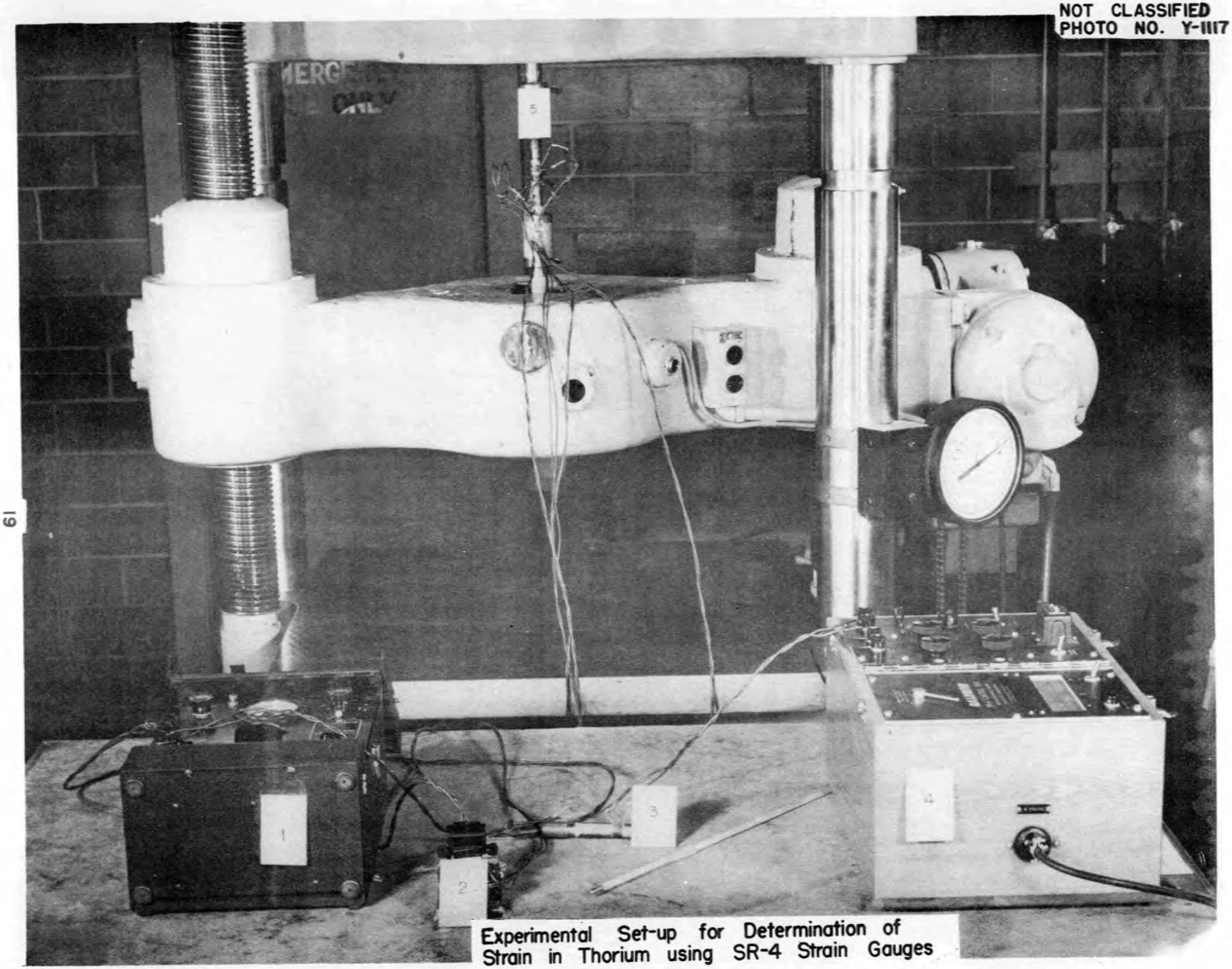


Experimental Set-up for Determination of Strain in Thorium using SR-4 Strain Gauges

FIG. 3

reading the load was ± 1 lb up to 1000 lbs load and ± 5 lbs above the 1000 lb load. The error in the strain measurement $\pm 5 \times 10^{-6}$ in./in.

From the values of stress and strain determined in these tests, load-elongation curves were plotted. The values of longitudinal strain obtained from both gauges and the average strain were plotted at the different stress values. The curve of the average longitudinal strain plotted as function of stress gave a good straight line from which the modulus of elasticity could be calculated.

Poisson's ratio was calculated at each stress level by dividing the the average transverse strain by the average longitudinal strain. The values at the different stress levels were averaged for the value of Poisson's ratio for the individual specimens.

The shear modulus was calculated from the interrelationship of Poisson's ratio and modulus of elasticity,

G = shear modulus,

E = modulus of elasticity ,

U = Poisson's ratio,

$$G = \frac{E}{2(1 + U)}.$$

Only three values of shear modulus were calculated, one from average tension test data, one from average compression test data, and the overall average using values from both tests.

The values of modulus of elasticity, Poisson's ratio, and the shear modulus are given in Table III. The results for the modulus of elasticity are in good agreement. The values of Poisson's ratio show considerable scatter. This was especially true in the compression tests. Possible explanations are: nonaxiality of loading, gauge errors, and flaws in the specimens.

Torsion tests for determination of shear modulus will be made on samples from the same lots of thorium. These values for the shear modulus and the data for the modulus of elasticity will be used to calculate Poisson's ratio. The resulting data will permit an accurate check of Poisson's ratio as determined above.

TABLE III

Modulus of Elasticity, Poisson's Ratio and Shear Modulus of Thorium

	SPECIMEN NUMBER	MODULUS OF ELASTICITY	POISSON'S RATIO	SHEAR MODULUS (calculated)
(TENSION TEST)	A-164	10.0×10^6	.294	
	A-166	9.9×10^6	.247	
	A-180	10.4×10^6	.262	
	A-183	9.92×10^6	.263	
	A-174	10.16×10^6	.287	
	A-179	10.44×10^6	.241	
	A-183	9.98×10^6	.322	
	Average	10.1×10^6	.274	3.97×10^6
(COMPRESSION TEST)	A-155	11.1×10^6	.210	
	A-164	10.16×10^6	.261	
	A-166	10.1×10^6	.344	
	A-179	10.5×10^6	.383	
	A-180	10.6×10^6	.267	
	A-183	10.2×10^6	.068	
	Average	10.4×10^6	.255	4.16×10^6
Overall Average		10.3×10^6	.265	4.06×10^6

Following the stress-strain tests, the tensile bars were remachined into conventional .505 in. diameter tensile test bars. After being annealed at 800°C for 1/2 hour, the bars were pulled in the tensile testing machine at a constant cross-head speed of 0.1 in. per minute. The medium low range of the 120,000 Baldwin Southwark machine was used with Peters extensometer No. 6106M. A load-elongation curve was drawn for each specimen and data were obtained for a true stress-true strain curve. Typical examples of the specimens after fracture are shown in Figs. 4 and 5.

The results of the tensile test are given in Table IV. In comparing these data with previous results, it is seen that the lower carbon content of the material used in the present tests is reflected in lower tensile strengths and higher ductility values. The modulus of elasticity determined in the tensile test compares favorably with the modulus determined using strain gauges.

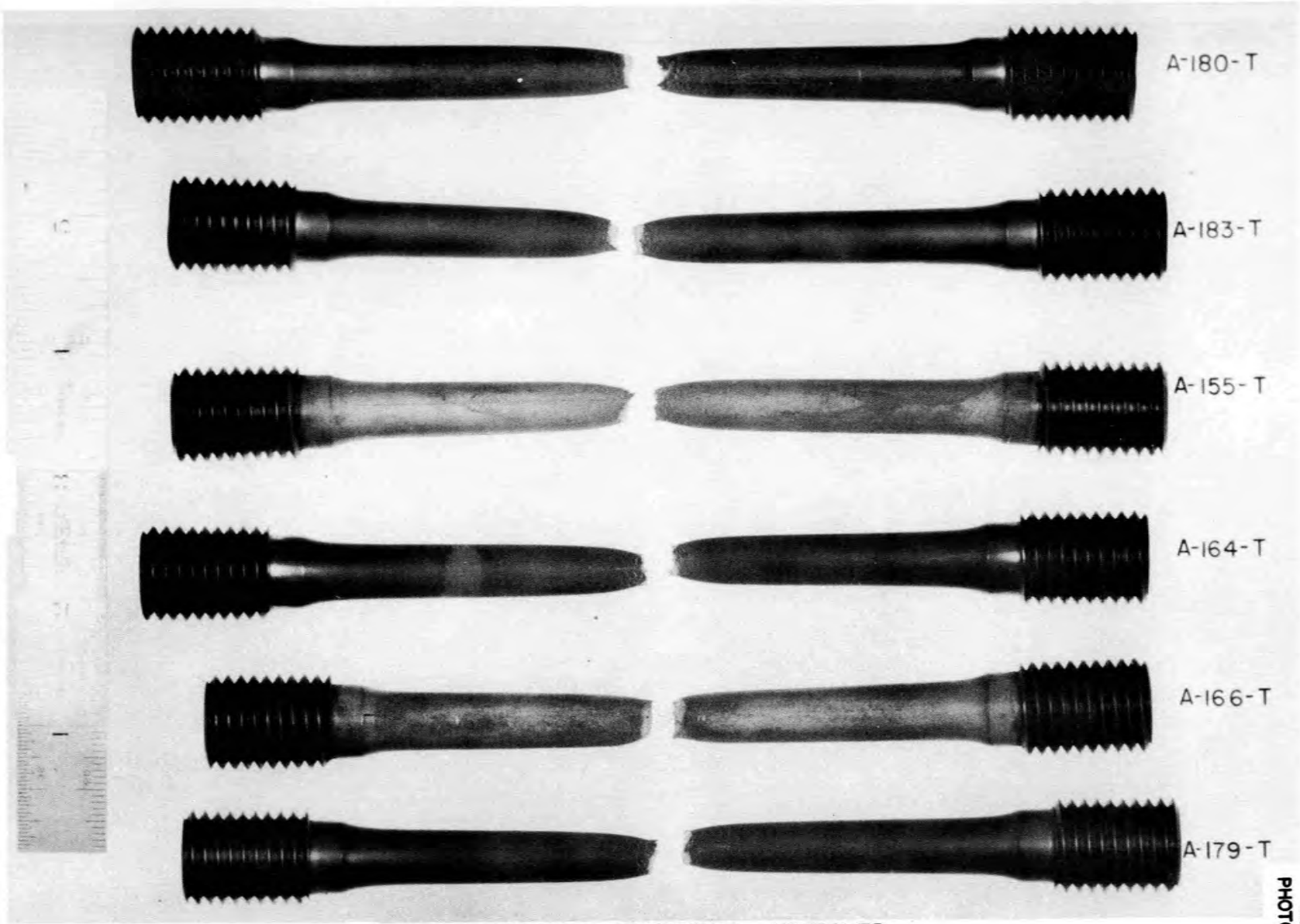
A load-elongation curve from a thorium tensile test is given in Fig. 6. This curve has a pronounced "yield point" comparable to yield points seen in load-elongation curves of low-carbon steel. Preliminary results from compression tests on thorium show this yield point also.

Bars are now being fabricated to determine the effect of cold work, and cold work plus aging, on the yield point of thorium. Experimenters have found that cold work will remove the yield point of low carbon steels but the yield point will return if there is a time delay between testing and cold working. The yield point of thorium will also be checked using high purity thorium prepared by the iodide decomposition process.

PREPARATION OF THORIUM FILAMENTS

In order to have the most pure grade of thorium available for test, Battelle Memorial Institute has been requested to prepare thorium rods by iodide decomposition on a hot filament. To attain maximum purity, it is necessary to use thorium wire for this filament. The present work was concerned with the preparation of such wire.

Two thorium wires of .075 in. diameter have been made from Ames metal plate. The history of this material is unknown; the analysis is as follows: 0.12% C, 0.10% O_2, 0.010% N_2 and traces of Fe, Ca, Mg, Ni, Pb, and Si. A



THORIUM TENSILE SPECIMENS ANNEALED
½ HOUR AT 800°C BEFORE TEST

FIG. 4

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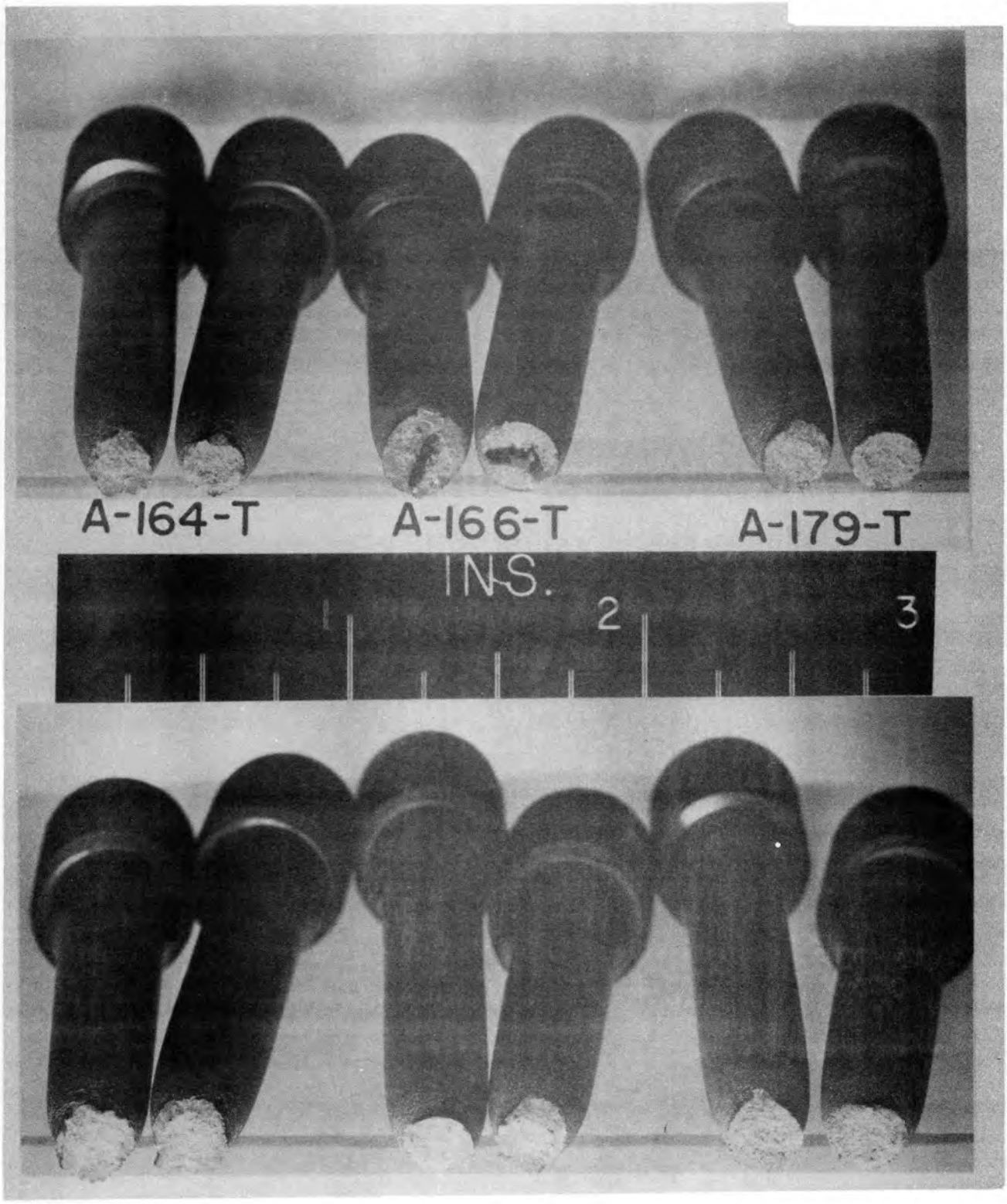
Y-128



Fracture of Thorium Tensile Specimens

FIG. 5

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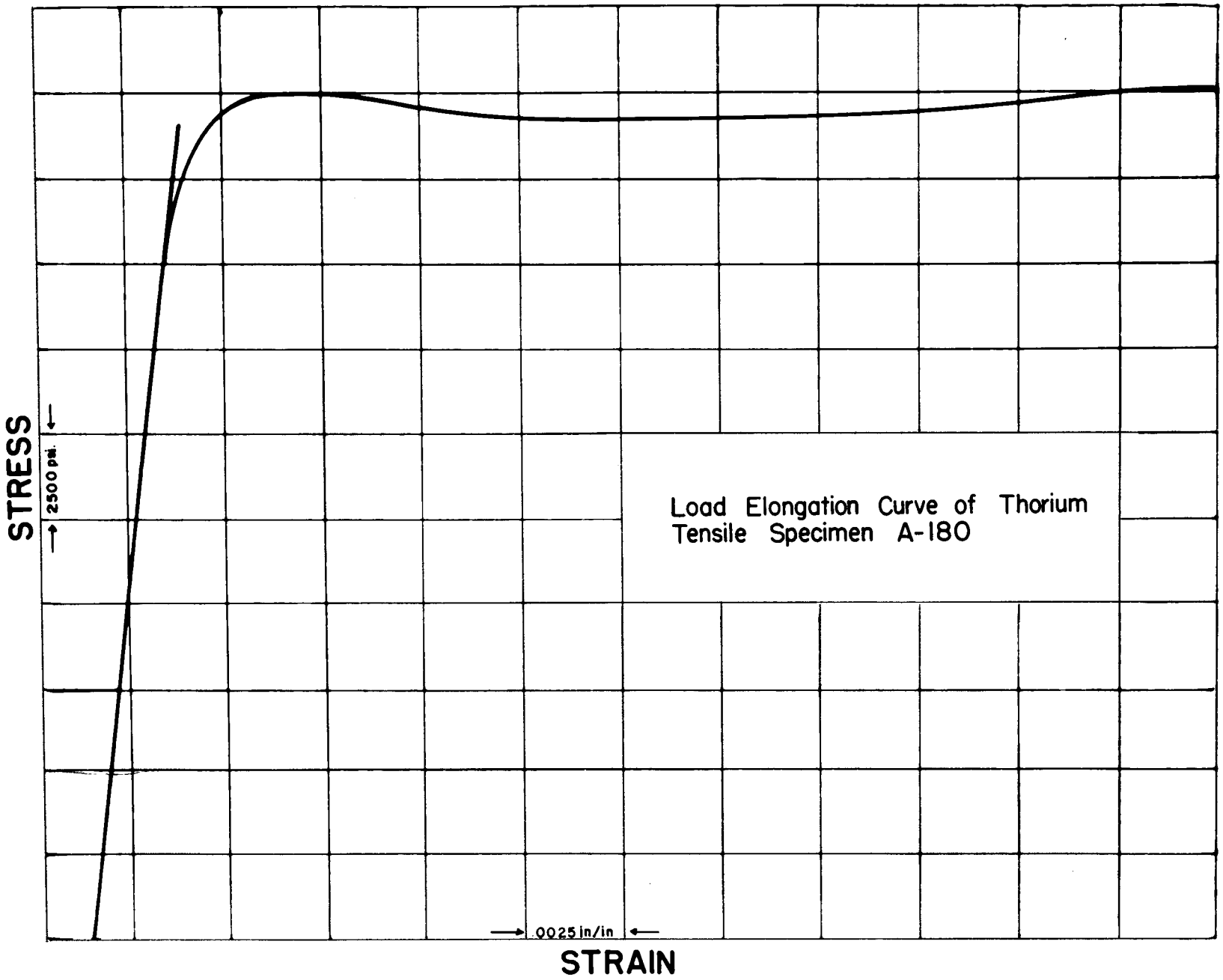


FIG. 6

TABLE IV

Tensile Testing of Thorium

SPECIMEN NUMBER	A-155	A-164	A-166	A-179	A-180	A-183
Percent Elongation in 2 in.	60.7	65	45.9	62.6	63.5	64.6
Percent Reduction in Area	71.4	75.2	59.9	69.1	71.3	75.8
Tensile Strength (psi)	31,750	27,570	29,970	34,800	32,680	26,870
Ultimate Strength (psi)	68,940	67,670	40,050	64,300	62,830	67,290
Modulus of Elasticity	9×10^6	7×10^6	9×10^6	10×10^6	10×10^6	13×10^6
Proportional Limit (psi)	22,500	19,000	17,040	21,500	20,500	15,000
Yield Point (psi)	25,800	21,900	22,360	26,500	25,000	18,750

sample .255 in. × .350 in. × 9.5 in. was milled from a .255 in. thick plate. After annealing the rod for one hour at 800°C, the wire was fabricated in the following steps:

Heated in oil at 260°C
Rolled in 3/8 in. pass to make a round bar
Heated in oil at 260°C
Swaged from 0.387 in. to 0.307 in. diameter (20% reduction)
Annealed at 800°C for one hour
Heated in oil at 260°C
Swaged from 0.307 in. to 0.102 in. diameter (88% reduction)
Annealed at 800°C for one hour
Heated to 100-200°C and coated with a water slurry of Stancoat No. 1 (a drawing compound).
Drawn from 0.102 in. - 0.075 in. diameter using a draw plate and the 120,000 Baldwin Southwark tensile testing machine (48% reduction)

The reduction figures given in parentheses represent the total reduction between the 800°C anneals. At first the rods were heated in oil before each pass in the swaging dies. This is not necessary, however, and in the final swaging operations the rods were not preheated. The most difficult part of this procedure is pointing the rods for drawing. This was accomplished by grinding and filing.

MTR COMPONENT DEVELOPMENT

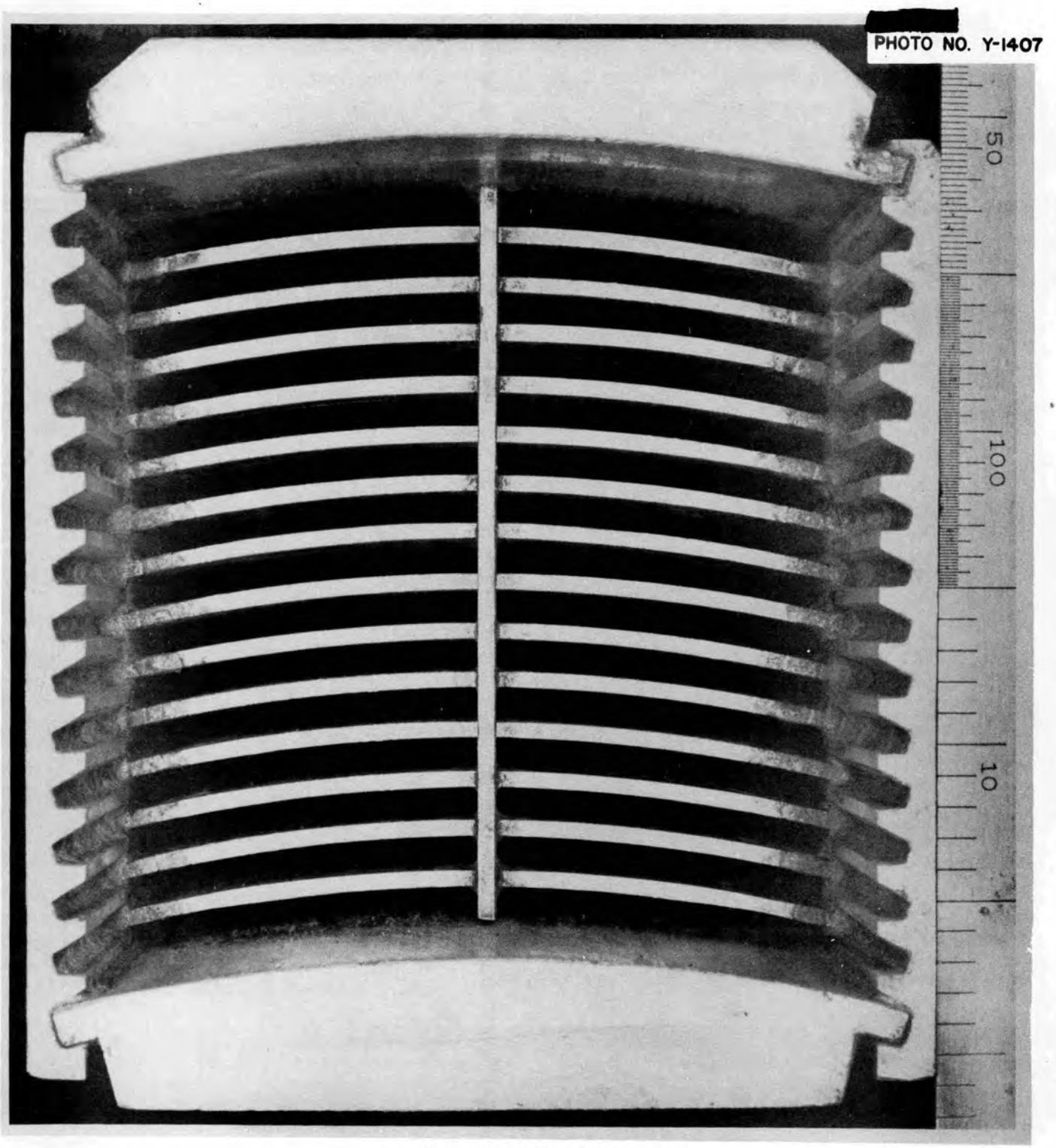
MOCK-UP FUEL PLATES

Two additional natural uranium runs (N25, N26) gave high yields (90%) of blister free plates after treatment with aluminum brazing flux (Eutectic 190) at 1100°C. Accordingly, production of plates containing enriched uranium was resumed, with a modified procedure, incorporating an extra step of heating the clad plate to 1100°F while coated with a slurry of brazing flux. Yields were much improved over those obtained previously. Of 235 plates rolled, 201 did not blister. A listing of individual yields for each run is shown in Table V. During the last five runs, the amount of flux applied to the plates was varied; in the cases of runs N31, N33, and N35, a very heavy coating was applied; a thin coating was brushed on the plates from runs N32 and N34. The former treatment seemed to be best: an average yield of 96% was obtained from runs N31, N33, and N35 while only a 69% yield was realized with runs N32 and N34.

The production of fuel plates for the critical experiment was completed on February 10, 1950. Of a total of 749 plates which had been rolled, 463 were acceptable (61.8%). A breakdown of uranium distribution for the 35 runs is given in Table VI. The value given for U in the slag (55.72 gm) is obtained by difference between the amount charged in the 35 heats and that accounted for by transfers and material remaining capable of being analyzed. It is subject to the errors involved in sampling, weighing and analysis (± 1% of the uranium involved).

MTR MOCK-UP FUEL ASSEMBLIES

Production. Eleven enriched fuel assembly units were produced, completing the commitment of fuel fabrication for the MTR critical experiment. An additional fuel section was prepared for use in a shim safety rod for the experiment. A production technique was adopted which would insure preparation of such units with a minimum of rejections due to exposed uranium alloy. The uranium section, after brazing, is shown in Fig. 7. In addition to a heavy top and bottom plate of aluminum, the safety rod assembly units are narrower than standard sections, and the component plates are also smaller. After



Section of Shim Safety Rod

FIG. 7

TABLE V

Yields of Blister-Free Fuel Plates

HEAT NUMBER	TOTAL PLATES	BLISTERED PLATES	PERCENT YIELD
N25	20	4	80
N26	21	4	81
N27	22	2	91
N28	22	2	91
N29	22	3	86
N30	20	3	85
N31	22	0	100
N32	20	6	70
N33	22	2	91
N34	22	7	68
N35	22	1	97
		Average	85.5

TABLE VI

Distribution of Uranium in Fuel Plate Fabrication

Received as U_3O_8		5749.11 gm
Charged as U_3O_8		5591.38
In samples transferred out	47.61 gm	
In samples stored	40.96	
In acceptable plates	3797.36	
In blistered plates (not recycled)	1555.73	
In alloy scrap from last run	93.98	
In slag (35 runs)	55.72	
		5591.38

fabrication of active plates for standard fuel it was found feasible to select the most nearly centered plates for control-rod sections. With the aid of suitable templates and fluoroscopic examination, enough of these were set aside for further machining operations. All plates thus selected could be milled to dimension satisfactorily.

The previous Quarterly Report (ORNL 583)[1] tabulates the inspection results obtained for the first fourteen enriched fuel assembly units. The key dimensions, Fig. 8, obtained on all 25 units are plotted in Fig. 9. The range of values obtained have been plotted against their frequency; thus, if one assembly shows a range of but 0.003 in. in sagitta, this point is shown as 4%, 0.003 in. (sagitta). Since no assembly shows range greater than 0.026 in. for this dimension, the point 100%, 0.026 in. corresponds to the extremity of this curve.

Cadmium foil was used as a shield-wrapper for storage of the first enriched fuel assemblies produced. Some thought was given to the possibility of cadmium pickup during handling. Filter paper smear tests showed the cadmium

(1) Frye, J. H., *Metallurgy Division Quarterly Progress Report for Period Ending January 31, 1950*, ORNL 583 (Mar. 10, 1950).

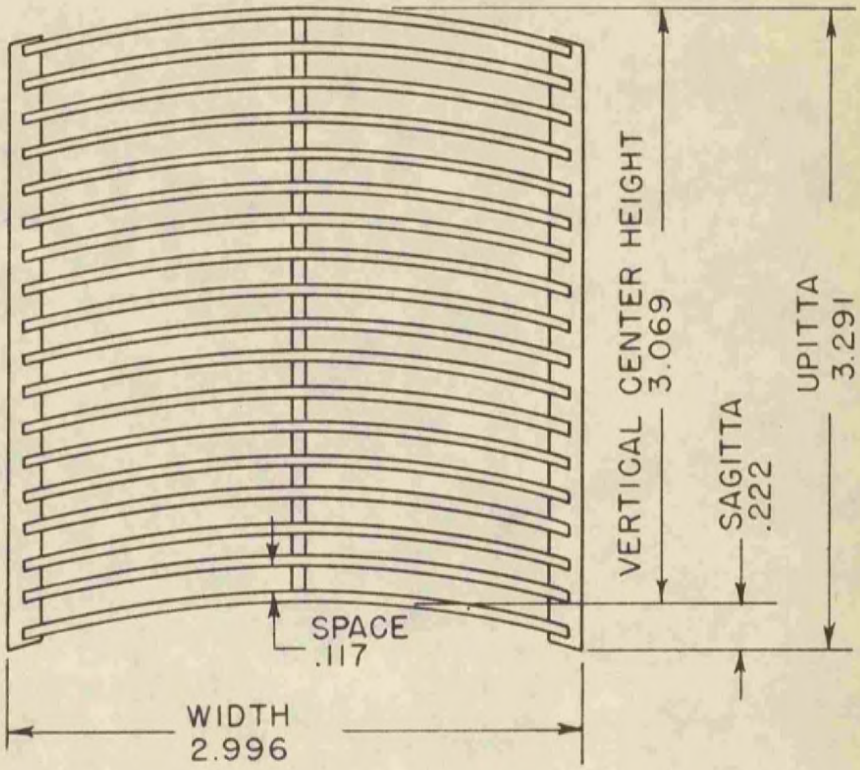


FIG. 8
FUEL ASSEMBLY DIMENSIONS

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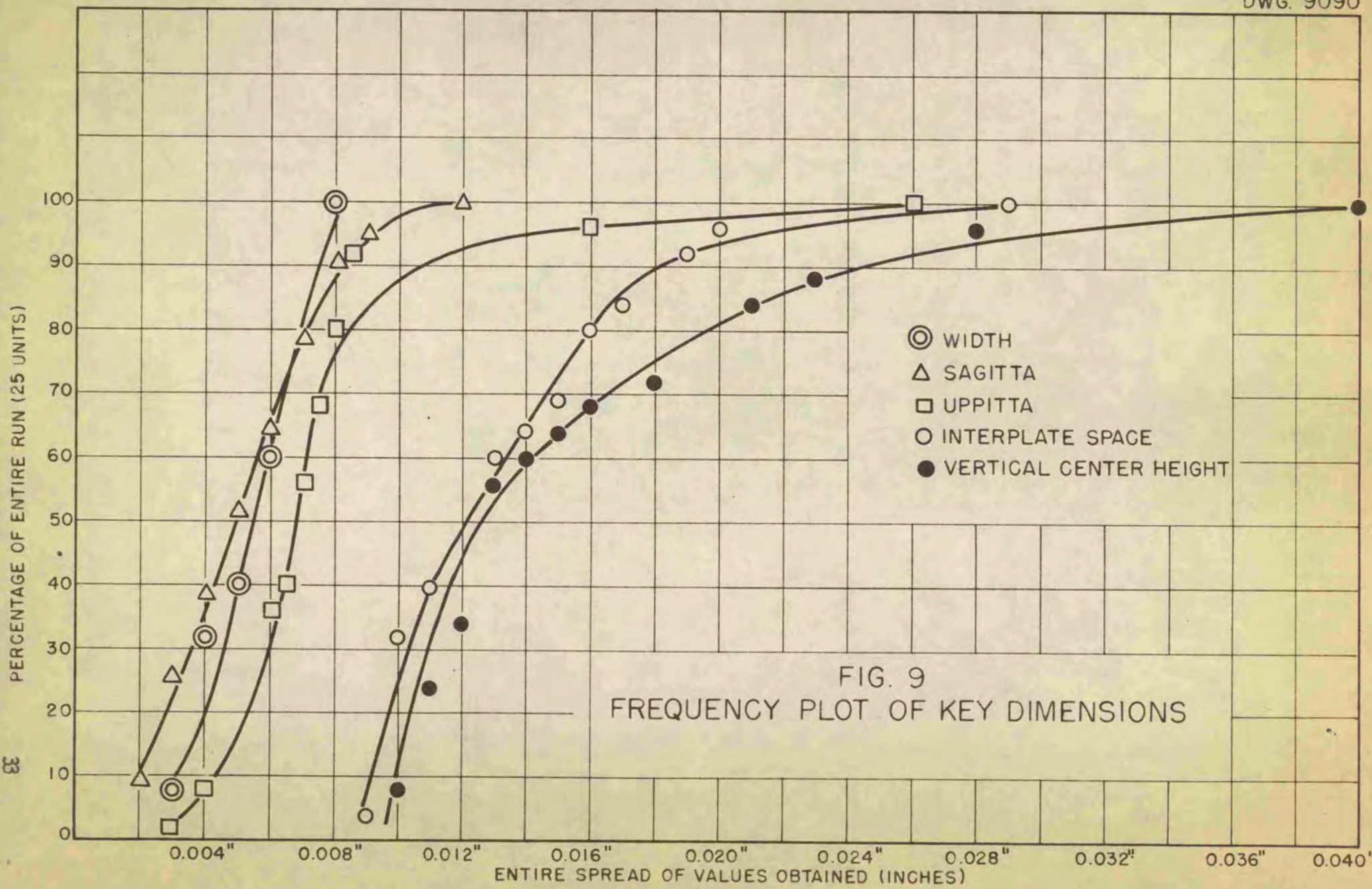

FIG. 9
FREQUENCY PLOT OF KEY DIMENSIONS

pickup by the assemblies to be low. However, subsequent assemblies were wrapped first in paper to avoid direct metal contact.

Brazing Studies. The brazing cycle used in fabrication of fuel assemblies consists of three steps as follows:

1. Dry at 150°C — 2 hours
2. Preheat at 850°F — 50 minutes
3. Braze at 1110°F — 38 minutes

Attempts were made to eliminate either or both of the first two steps as follows:

Assembly 1	Dry:	2 hours at 150°C
	Braze:	45 minutes at 1100°F
Assembly 2	Dry:	2 hours at 150°C
	Braze:	55 minutes at 1110°F
Assembly 3	Braze:	65 minutes at 1110°F (ceramic jig)

Results were very encouraging. In the first case there was insufficient flow of braze metal. In the second trial braze joints were uniform and good. In the third case the assembly was not brazed quite as well as the second but all plates were joined. Further trials will be made with either longer times or higher temperatures.

Casting Studies. To ascertain the relative distribution of uranium in cores and punching scrap, two ingots (N30, N31) were cast and rolled to 0.300 in. Twenty cores were punched from each slab; then, in each case, the cores were melted in one batch and the scrap in another. Duplicate samples were taken from each melt prior to pouring. The results, tabulated below show no significant difference.

ANALYSIS of U
(Percent)

HEAT NUMBER	MELT	CORES	SCRAP
N30	13.41	13.34	13.30
N31	13.43	13.37	13.36

FUEL ELEMENTS

Further trials at plate forming by successive pressing showed that this method would be feasible. In every case, radius of curvature was uniform. There was an additional curve along the length of the plate. This longitudinal bowing could be removed by annealing at 1100°F while the plates were stacked on a form having the proper lateral radius of curvature. Several regular MTR assemblies made with plates which had been formed by successive pressing did not show any deviation from normal plate spacing or other dimensions. Work on long MTR-type fuel elements has been stopped.

Li-Al ALLOY PLATES

Three small sandwiches containing 3.5% Li-Al alloy were prepared. Cores 0.5 in. × 0.34 in. × 0.024 in. were punched and inserted in an aluminum frame 6 in. × 4 in. × 0.230 in. having three holes punched in it. The frame and cores were preheated in a helium atmosphere at 500°C and hot rolled 50% in four passes. Then a 0.120 in. 2S aluminum cover was applied and the assembly was hot-rolled to 0.060 in. Annealing three hours at 500°C produced no visible change in the sandwich. Samples have been submitted for metallographic investigation. Three additional Li-Al sandwiches having the same dimensions as an MTR fuel plate were similarly rolled. One of these exhibited a long blister at one edge of the core after annealing three hours at 500°C. This work has been discontinued.

BULK SHIELDING FACILITY COMPONENTS

Plans are being made for fabrication of 23 fuel sub-assemblies and three control rods for the Bulk Shielding Reactor. Fuel assemblies are to be similar to those fabricated for the MTR critical experiment. Shim and safety rods are of a new design — essentially an aluminum tube filled with a Pb-Cd alloy. It is expected that fabrication will start in June.

BERYLLIUM REFLECTOR SECTIONS

The specifications for the beryllium for the MTR reflector were drawn up and transmitted to the Steering Committee for inclusion in the material request.

They include chemical and metallurgical specifications for both hot-pressed and extruded metal.

During the past quarter, six hot-pressed Materials Testing Reactor (MTR) reflector sections were deep-hole drilled in the Research Shops at ORNL. These full length (40 in.) sections were cut from the first large slab produced in the new horizontal hot pressing furnace recently installed at Brush Beryllium Company in Cleveland, Ohio. Results were encouraging and indicated that hot-pressed metal could be drilled equally as well as extruded metal by the gun barrel drilling technique. Accuracies obtained in drilling the long, straight, 3/16 in. diameter cooling holes were greater than expected.

Practically all earlier drilling work was conducted on extruded stock, and there existed some doubt as to the direct applicability of this technique to the hot-pressed metal. In fact, comparison tests with high-speed drills at High Standard Manufacturing Company last summer showed that the hot-pressed metal required more frequent resharpening than did the extruded metal. This excessive tool wear was presumably due to the additional amount of beryllium oxide present in the hot-pressed metal. This difficulty was remedied with the use of the tungsten carbide tipped tools.

Table VII contains the pertinent data used in deep hole drilling and fabrication of the six sections into standard "A" type reflector units.

Four drill tools, stepped in convenient lengths (16, 28, 40 and 56 in.) to cover the forty-inch range, were used in drilling the first unit. Shorter length drills are required in the beginning to minimize whipping of the tool shaft. Use of too many tools, however, increases the hole run out from straightness; therefore, only two tools were used in drilling the last five units; and this appears to be the optimum number.

The drill tool uses its own prime mover and rotates on an axis independent of the workpiece. "End difference" refers to the maximum runout on the final end resulting from the drill rotating on one axis and the workpiece on another. After drilling, the unit is aligned on the hole axis and the sides are machined paralled to the hole axis. The last two columns in Table VII indicate the accuracy to which the lateral sides can be finish machined with respect to the water cooling channel.

TABLE VII

Deep Hole Drilling of Hot-Pressed Beryllium

MATERIAL IDENTIFICATION	HOLE DIAMETER (Inches) LOWER END	HOLE DIAMETER (Inches) UPPER END	NUMBER OF DRILLS USED	END DIFFERENCE BETWEEN AXES OF DRILL AND WORK (Inches)	MAXIMUM VARIATION BETWEEN HOLES AND SIDES AFTER MACHINING (Inches) UPPER END	MAXIMUM VARIATION BETWEEN HOLES AND SIDES AFTER MACHINING (Inches) LOWER END
Y4460 BPQRM - 1	0.1885	0.1880	4	< 0.030	0.005	0.004
- 2	0.1870	0.1875	2	0.016	0.004	0.002
- 3	0.1875	0.1870	2	> 0.005	0.001	0.002
- 4	0.1870	0.1870	2	> 0.005	0.000	0.002
- 5	0.1880	0.1890	2	< 0.005	0.001	0.002
- 6	0.1870	0.1875	2	0.000	0.001	0.001
	Drill diameter			0.184 in.		
	Angular speed work piece (counterclockwise)			146 rpm		
	Angular speed of drill (clockwise)			1476 rpm		
	Angular speed of drill with respect to work piece			1622 rpm		
	Drill feed			0.392 in.		
	Coolant			Mixture 50-50 sulphurized oil Varsol, plus 2-5% by volume of Oil Dag		
	Coolant pressure			15-20 psi		

NEUTRON WINDOW-LEAD CREEP TESTS

The lead creep tests mentioned in the last Quarterly Report[2] were discontinued and new tests were started to check the results obtained. In the new tests the deformation is being measured with SR-4 strain gauges. Chemical lead and lead calcium alloys are being held at 150°C under stresses of 30 and 60 psi.

The specimens under the 60 pound stress have been stressed about 1000 hours and are elongating along smooth curves. The loading is not exactly axial so the two sides of the specimen are following different curves; however, they do seem to be approaching the same rate. The differences were in the first stage of the creep. The calcium-containing alloys are elongating more rapidly than the chemical lead. This is not what was expected and work is being started to see if this is a true effect caused by the low stresses at high temperature or if it resulted from poor heat treatment. At present the average creep rate for the chemical lead is about 0.1 microinches per hour or about 0.1% per year. The average rate for the calcium lead is about 0.2% per year.

The specimens under a stress of 30 pounds have not yet (~1000 hours) elongated enough to produce definite results.

Since the exact temperature of the window is not yet available and the results for 30 psi are doubtful, it is desirable to obtain some curves from which the desired values can be extrapolated. To obtain such curves for both temperature and stress, new tests are being set up. These tests include temperatures of 100°C, 150°C, and 200°C and stresses of 30, 45, 60 and 90 psi.

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(2) Frye, J. H., *op. cit*, ORNL 583.

ANP MATERIALS PROGRAM

The objectives of this program are to select and study materials suitable for construction of a high temperature, liquid metal cooled, air-borne reactor, and to develop methods of fabricating these materials into reactor components. The bulk of the reportable information available at this stage of the work is that pertaining to static corrosion sorting tests which are being run to select the materials which are to be subjected to more thorough investigation.

Static corrosion sorting tests have been made for the following metals and alloys:

(1)	Niobium (Columbium)	(7)	Iron (Armco)	(13)	Manganese
(2)	Molybdenum	(8)	Alloy L-605	(14)	Nickel
(3)	Vanadium	(9)	Beryllium	(15)	Zirconium
(4)	Tungsten	(10)	Cobalt	(16)	Titanium
(5)	1040 Steel	(11)	Chromium	(17)	Thorium
(6)	Tantalum	(12)	Silicon	(18)	Uranium

The tests consisted of exposing the above metals to the action of liquid lithium in an evacuated Armco iron capsule for four hours at 1000°C. Similar tests were made using liquid bismuth instead of lithium. The samples were run in triplicate. The lithium was melted in the protective atmosphere of a helium-filled dry box, drossed to remove floating impurities, and cast into inverted hollow capsule plugs, one of which is shown in Fig. 10. After cooling, the plugs were placed in a desiccator before removing from the dry box. In runs using bismuth, the dry box was not used but loading was carried out by charging a small ingot of bismuth previously cast to fit into the capsules. To prepare for a run, the sample was placed in a capsule along with the coolant metal and plug, the plug was cold pressed into the capsule, the capsule evacuated, and the seam between plug and capsule heli-arc welded. The weld was helium lead tested and the capsule and contents heated to the melting point of the coolant while evacuated. The portion of the plug extending above the capsule was then crimped twice, sheared off by a third crimp and spot welded. Heat for the test was furnished by four tilting Burrell Globar tube furnaces,

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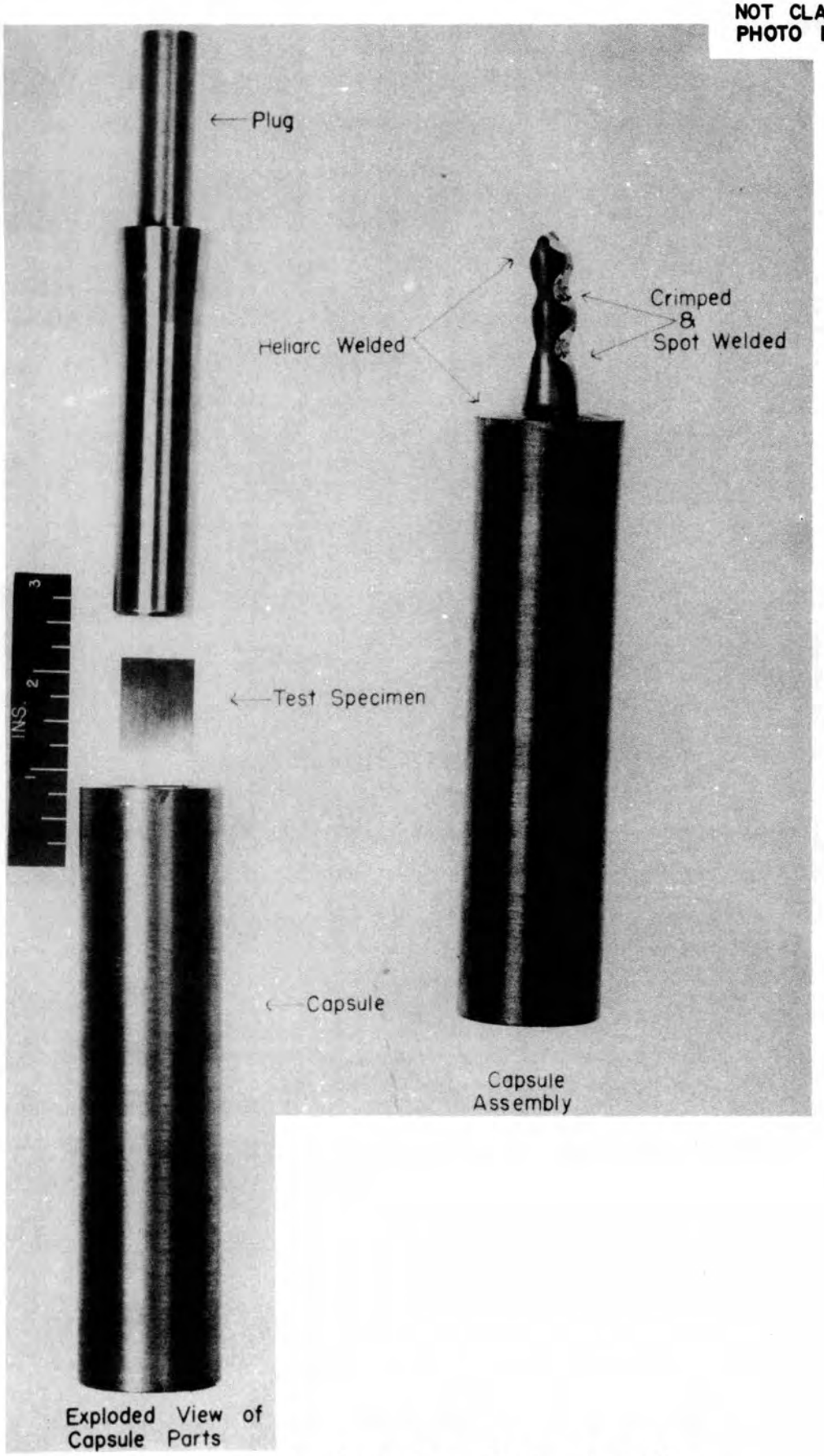

Liquid Metal Corrosion Test Capsule
FIG. 10

each capable of holding two samples. The tilting feature allows inversion of the capsules after each run to drain the coolant metal from the sample. The furnaces were continuously flushed with helium to prevent oxidation of the iron capsule and to help prevent fire in case of container failure. After completion of a heating cycle the capsules were placed in a lathe, grooves cut through the capsule walls, and the plugs removed. This method is preferred to sawing since it results in less contamination of coolant due to chips from the capsule. Lithium or bismuth remaining was removed and the samples were weighed. The metal samples were sectioned and mounted for microscopic study, and surface films, if present, were identified by X-ray diffraction. The lithium and bismuth were analyzed spectrographically for sample components.

The bismuth used was specified as 99.95% pure, supplied by Belmont. Lithium was obtained from the Maywood Chemical Works of Maywood, New Jersey. The manufacturer's specifications indicated a product of 99.5% purity, the remainder being 0.20 calcium, 0.07 heavy metals, 0.03 iron and aluminum, 0.02 nitrogen, 0.02 sodium and 0.009 silicon. Subsequently, lithium with 0.005% sodium, as supplied by the Metalloy corporation of Minneapolis, Minnesota, was substituted.

The data for the various four hour runs at 1830°F are presented in Tables VIII and IX. They represent, with a few exceptions, weighted averages for three specimens. The results are given in terms of weight change per unit of surface area rather than as a time rate of weight change, because corrosion rate is not likely to be linear, and at present we have data representing one time interval only. Samples for test were made up on flats 3/4 in. × 3/4 in. × 1 in. whenever possible. Since the volume of coolant material placed in each capsule was uniform, variation in sample surface is reflected in the column headed "Surface per unit volume of coolant." The value of this ratio for a standard specimen is 0.6.

Corrosion samples from each of the various elements and alloys tested are being studied to determine the nature and depth of attack, thickness of film formation, and possible selective leaching of impurities.

Transverse metallographic sections were prepared and mounted adjacent to steel backing plates used to prevent rounding of the specimen edge during polishing. All specimens were carefully polished and examined microscopically

TABLE VIII

Corrosion of Materials by Liquid Lithium in Static Tests - 4 hrs., at 1000°C

MATERIAL	SOURCE AND ANALYSIS	RUNS	WEIGHT CHANGE (mg/cm^2) RANGE	WEIGHT CHANGE (mg/cm^2) AVERAGE	RATIO OF SPECIMEN AREA TO COOLANT VOLUME, cm^2/cm^3	REMARKS
Titanium	Remington Arms	3	+ 3.5 to + 4.4	+ 3.5	0.4	Discontinuous adherent film. Identified by X-ray as TiN.
Zirconium	Bureau of Mines	3	+ 0.3 to + 0.5	+ 0.4	0.6	Thin continuous adherent film of ZrN.
Molybdenum	Fansteel	3	+ 0.1 to + 0.4	+ 0.3	0.6	MoC and Mo_2C detected
Zirconium	Bureau of Mines	2	- 0.1 to + 0.1	+ 0.1	0.4	ZrN, same as Zr above
Niobium	Fansteel	3	- 1.9 to + 0.1	- 0.1	0.2	
Iron (Armco)	Corry Steel Co.	3	- 0.2 to - 0.1	- 0.1	0.6 (?)	Uniform attack
Tungsten	Fansteel	3	- 5.6 to - 0.1	- 0.2	0.6	Uniform attack - no film detected
Tantalum	Fansteel	3	- 1.5 to - 1.0	- 1.5	0.6	Uniform attack with thin carbide film formed
1040 Steel	SAE Steel Co.	3	- 2.4 to - 1.9	- 2.2	0.6 (?)	Selective type corrosion, probable carbide solution
Cobalt	Kulite Tungsten Co.	2	- 5.2 to - 4.8	- 4.8	0.6	Penetration attack of sintered metal
Chromium	Univ. of Cincinnati	1		- 5.7	0.7	
L-605	Haynes Stellite Co.	3	- 7.4 to - 6.0	- 6.8	1.3	Selective type corrosion
Vanadium	KAPL	1		- 7.7	0.4	Continuous non-uniform film of VN and V_2N or V_2C
Beryllium		3	- 18 to - 16.4	- 17	0.6	Unidentified discontinuous surface film. Fairly uniform attack
Thorium	Iowa State College	1	- 67.7 to - 45.4	- 56.8	0.6	
Nickel	International Nickel	3	- 100.4 to - 67.9	- 86	0.6	Severe intergranular attack
Manganese	Electro Manganese Corp.	3	Thin strip tested - some solubility noted.			
Silicon		3	Dissolved and deposited on capsule wall.			

TABLE IX

Corrosion of Materials by Bismuth in Static Tests - 4 hrs. at 1000°C

MATERIAL	SOURCE	RUNS	WEIGHT CHANGE (mg/cm^2) RANGE	WEIGHT CHANGE (mg/cm^2) AVERAGE	RATIO—SPECIMEN AREA TO COOLANT VOLUME, cm^2/cm^3	REMARKS
Niobium		2	+ 14.1 to + 24.5	+ 19.3	0.2	Continuous film
Molybdenum		2	+ 9.9 to + 11.0	+ 10.4	0.6	Continuous uniform film plus fairly continuous intermetallic
Vanadium	KAPL	1		+ 9.9	0.4	Heavy continuous film formed some intergranular attack
Tungsten		2	- 39.6* to + 3.7	+ 3.7	0.6	Uniform attack
1040 Steel	SAE Steel Co.	3	- 0.3 to + 4.6	+ 2.6	0.6	Uniform attack with selected corrosion of carbide
Tantalum		2	+ 2 to + 2.9	+ 2.4	0.6	Uniform film over original surface plus penetration layer
Fe (Armco)	Corry Steel Co.	2	0 to + 0.2	+ 0.1	0.6	Uniform attack
U-605	Haynes Stellite Co.	3	- 4 to + 0.1	- 3.5	1.3	Continuous film intergranular type attack
Beryllium		3	- 25.4 to - 14.8	- 21	0.6	Fairly uniform attack
Cobalt	Kulite Tungsten Co.	2	- 30 to - 18.4	- 24	0.6	Severe penetration type of attack
Chromium	Univ. of Cincinnati	1		- 126	0.7	Severe uniform attack
Silicon		3	Some weight Loss noted			
Manganese	Electro Manganese Corp.	3	Thin strip tested - partially dissolved			
Nickel	International Nickel	3	Essentially dissolved			
Zirconium	Bureau of Mines	3	Essentially dissolved			
Titanium	Remington Arms	3	Dissolved			
Thorium	ISC	3	Dissolved			
Uranium		3	Dissolved			

* This value is probably not due to corrosion.

in the unetched condition. Etching, it was felt, would perhaps destroy films and other corrosion effects at the liquid-metal interface.

Figure 11 shows unetched micrographs of the various element tests for four hours in 1000°C (1832°F) lithium.

Pure Metals in 1000°C (1832°F) Lithium

Zirconium-graphite melted (Mag. 1000X) shows good resistance to attack. A thin continuous adherent film, identified by X-ray diffraction as ZrN, was found. Film thicknesses were approximately 0.1 mil and 0.4 mil respectively for two lots of zirconium metal tested.

Cobalt-sintered (Mag. 500X) shows poor resistance to attack. Average corrosion depth is 1 mil. Several deep stringers were noted running to a depth of 6 mils. The intergranular attack is typical of that due to impurities at the grain boundaries.

Tantalum (Mag. 1000X) shows good resistance to 1000°C lithium, with formation of continuous thin TaC film.

Armco Iron (Mag. 1000X) shows extremely good resistance to attack. Edge roughness is presumably due to machining during preparation. All specimens are now being finished on 600 grade metallographic paper.

Columbium (Niobium) (Mag. 1000X) shows good resistance to attack. The gain in weight is attributed to a uniform film formation, approximately 0.1 mil deep. The coating has not been identified as yet.

Molybdenum (Mag. 1000X) shows good resistance to short time exposure. The weight gain is attributed to irregular build-up of Mo_2C and MoC.

Tungsten (Wolfram) (Mag. 1000X) shows good resistance to lithium attack.

Nickel (electrolytic) (Mag. 100X) shows severe intergranular attack and deep penetration.

Alloy L605 (Mag. 1000X) shows poor resistance to attack. Selective leaching of one or more impurities is apparent from the zone of voids immediately below surface layer. Depth of the selective type of corrosion was approximately 0.7 mil.

CORROSION SAMPLES TESTED IN MOLTEN LITHIUM

1000°C FOR FOUR HOURS

EXPOSED SURFACE AT TOP OF PHOTOMICROGRAPH

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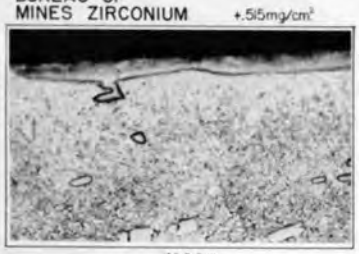
BUREAU OF MINES ZIRCONIUM +.515mg/cm²

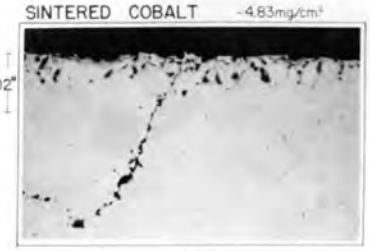


1000X

BUREAU OF MINES ZIRCONIUM +.088mg/cm²

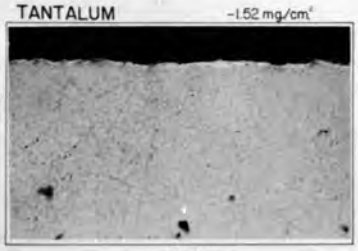
1000X

SINTERED COBALT -4.83mg/cm²



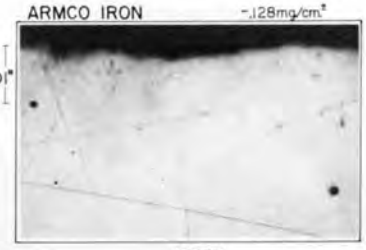
500X

TANTALUM -1.52 mg/cm²



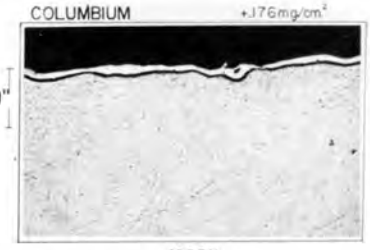
1000X

ARMCO IRON -.128mg/cm²



1000X

COLUMBIUM +.176mg/cm²

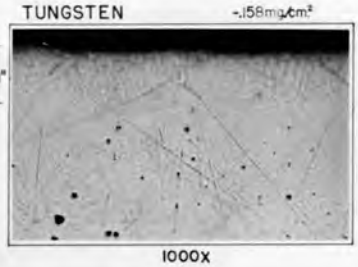


1000X

MOLYBDENUM +.338mg/cm²

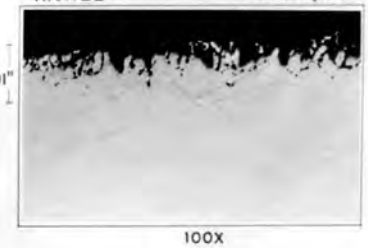
1000X

TUNGSTEN -.158mg/cm²



1000X

ELECTROLYTIC NICKEL -90.92 mg/cm²

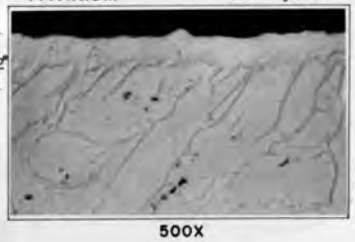


100X

ALLOY L605 -7.36mg/cm²

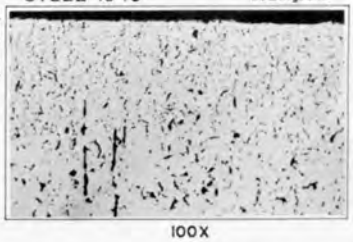
1000X

TITANIUM +3.55mg/cm²



500X

STEEL 1040 -1.92mg/cm²

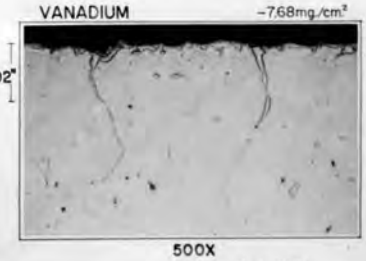


100X

BERYLLIUM -17.24mg/cm²

500X

VANADIUM -7.68mg/cm²



500X

UNETCHED

FIG. 11

R. J. Gray

Titanium (Mag. 500X) shows fair resistance. The thin film formed on outer surface, 0.1 mil thick, was identified as TiN. The heavier film underneath (~1 mil thick) and penetrating into the base metal is believed to be TiC. This was Remington Arms graphite melted grade Ti.

Beryllium (Extruded) (Mag. 500X) shows a scattered non-continuous film formation and large voids. Attack was severe.

Vanadium (Mag. 500X) shows a weight loss and a continuous non-uniform, unidentified film. Intercrystalline attack, commencing at specimen surface and running to a depth of approximately 6 mils, was noted.

1040 Steel (Mag. 100X) showed decarburization throughout the entire sample. Apparently all the carbon has been removed from sample.

Pure Metals in 1000°C (1832°C) Bismuth

Figure 12 shows micrographs of the various metals tested in 1000°C (1832°C) bismuth in the unetched conditions.

Tantalum (Mag. 1000X) shows continuous but unidentified uniform corrosion product build-up on the surface. Depth of the product was approximately 0.4 mil. The product is composed of a heavier film on the outside specimen surface, approximately 0.3 mils thick, and a diffusion layer penetrating approximately 0.1 mil into the specimen.

Beryllium (Mag. 500X) shows a moderate to heavy attack of the straight solution type.

Chromium (Mag. 500X) shows a severe attack with pure bismuth adhering to the corroded surface.

Molybdenum (Mag. 1000X) shows a continuous heavy film formation which has been identified as alpha iron. This presumably results from mass transfer of iron from the container wall. A thin unidentified layer exists between the molybdenum specimen and the iron coating.

Armco Iron (Mag. 1000X) shows good resistance to attack with no apparent weight change or damage to metal specimens for short-time exposure, but this may be due to the fact that Armco iron was the container.

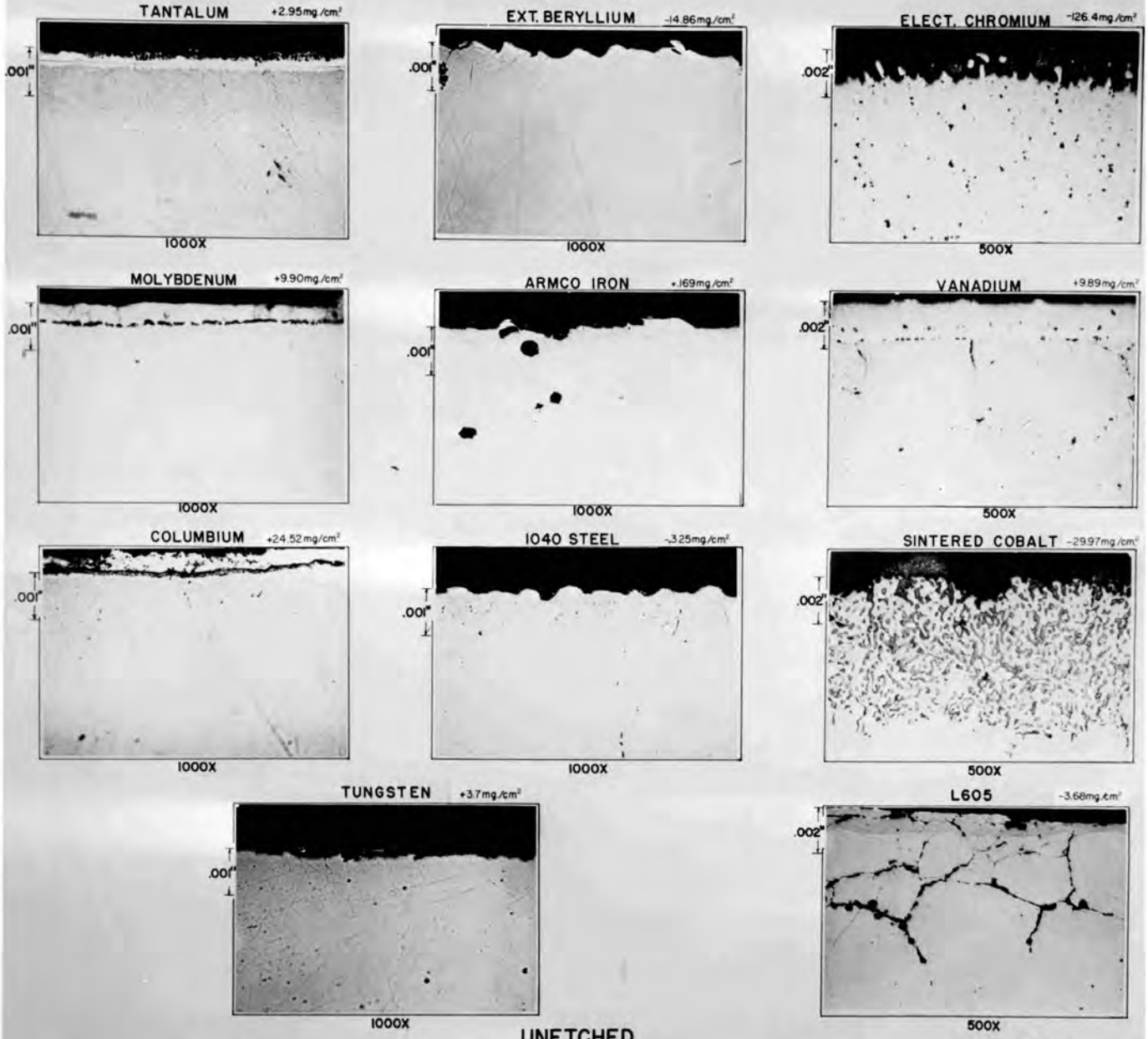
CORROSION SAMPLES TESTED IN MOLTEN BISMUTH

1000°C FOR FOUR HOURS

EXPOSED SURFACE AT TOP OF PHOTOMICROGRAPH

UNCLASSIFI

Y-1437



UNETCHED

FIG. 12

R.J. Gray

Vanadium (Mag. 500X) shows formation of a continuous heavy film (1.8 mils) identified by X-ray diffraction as α iron. It also appears that some selective corrosion occurred at the grain boundary.

Columbium (Niobium) (Mag. 1000X) shows a continuous unidentified film formation (approximately 0.1 mil thick) with pure bismuth metal adhering to the specimen surface.

1040 Steel (Mag. 1000X) selective corrosion (decarburization) occurred near the surface of the sample, extending to a depth of approximately 1 mil.

Sintered Cobalt (Mag. 500X) drastic attack, penetrating deeply into specimen. Apparently attack proceeds down through the sintered powdered particles and leaves a matrix of unattacked metal behind. Note the pure bismuth present in the net-work.

Tungsten (Wolfram) (Mag. 1000X) showed a heavy uniform straight solution type of attack.

Alloy 605 (Mag. 500X) showed severe intercrystalline type attack to a depth of approximately 6 mils. A continuous film was formed on the specimen surface, approximately 1 mil in thickness, identified as gamma iron.

In addition to the static tests reported above, work is proceeding on a number of other phases of the program which will be reported in greater detail at a later date.

The static corrosion sorting tests are being continued, with some modifications, to include other structural materials—ceramics, metal-ceramics, high temperature alloys including stainless steels; other liquid coolants—lead, sodium, sodium hydroxide; liquid fuels—lead-uranium and bismuth-uranium; and at different times and temperatures.

Tests are being run in the static corrosion equipment to investigate the effects of such factors as crystal structure, carbide phases, and relative concentration and metallographic form of soluble alloy components, stress-corrosion, addition agents in the liquid coolant, and gettering agents for purification of the coolants.

Several harps—thermal convection loops—are being made to study long time corrosion effects in systems involving flow of the liquid and a temperature differential.

Powder metallurgy equipment is being set up for use in preparation of materials to be used in the study of diffusion and alloy systems, as well as for the fabrication of fuel elements and other components of the Aircraft Reactor Experiment (ARE). Fabrication studies are in process in efforts to:

Obtain a metallurgical bond in bi-metallic tubing

Form bi-metallic tubing by shrouding

Weld bi-metallic materials into reactor system components

Coat special materials by electroplating

Vapor-deposit corrosion resistant materials on refractory metals or alloys.

RADIATION EFFECTS

FUNDAMENTAL STUDIES

It has been observed that alloys of the copper-aluminum system exhibit a decrease in electrical resistance when exposed to reactor radiation.[1] In addition, the alloys show an increase in hardness. In view of the fact that all alloys exposed show a complete solid solubility, it is difficult to understand this behavior in terms of the usual metallurgical mechanisms. It was conceivable that the "thermal spike" theory could explain this anomalous behavior; *i.e.*, the high temperatures momentarily developed might enable the original solid solution to become depleted in aluminum due to the fact that the aluminum atoms should diffuse away from the high temperature regions more rapidly than the copper atoms. The removal of aluminum atoms from the matrix solid solution would be accompanied by a decrease in electrical resistance. Should the size and distribution of the displaced atoms also be of the right order, an increase in hardness might be expected.

On the basis of the above reasoning, it would be expected that upon subsequent heat treatment, the aluminum in the aluminum-enriched region would diffuse back to obtain the original solid solution and thus restore the original resistance.

Annealing experiments were performed on the irradiated copper-aluminum alloy containing 9.5 atomic per cent aluminum, observing the resistance behavior as a function of time and temperature. As the specimens were heated the resistance returned to its pre-irradiation value. By plotting $1/t_{0.5}$ versus $1/T$, where $t_{0.5}$ is the time required for 50% of the effect to anneal out and T is the absolute temperature of the heating, a value of 44,800 cal/mol was obtained for the activation energy of the reaction. This value is within 10% of the activation energy for the diffusion of aluminum in copper. It is planned to continue this study in a more carefully controlled experiment and also to check the depletion of the solid solution by means of precise lattice constant measurements. Hall effect measurements are also planned.

Similar work is being done on copper-zinc, copper-silicon, and pure copper, observing the changes in hardness and resistance. Alloys of copper-tin

(1) Snell, A. H., *Physics Division Quarterly Progress Report for Period Ending September 25, 1949*, ORNL 480 (Nov. 8, 1949).

and copper-manganese of various compositions have been fabricated into resistance and hardness specimens for irradiation.

Experiments are being continued on copper-beryllium specimens of two weight per cent beryllium to determine the effect of irradiation on the rate of precipitation of the gamma phase.

Alloys of copper-zinc, copper-gallium, copper-germanium, and copper-arsenic have been fabricated for irradiation and determination of changes in elastic modulus, electrical resistivity, and hardness as functions of composition and irradiation. In addition, specimens will be machined from these alloys for stored energy measurements.

IN-PILE CREEP

A creep test apparatus capable of operation under irradiation in a stringer in the ORNL reactor is being developed. A preliminary design has been chosen, and a partially completed model is being tested to minimize temperature differentials and achieve good temperature control in the furnace. A specimen nine inches long and one-eighth inch in diameter, is enclosed in a constant temperature furnace through about two-thirds of its length. The specimen is to be stressed by means of cables extending outside the reactor where a dead weight furnishes constant load. The central four inch section of the specimen is the gauge length over which the strains are measured. Longitudinal strain is transmitted to a microformer by extensometer elements welded to the ends of the gauge length. A duplicate microformer, whose core may be moved a known distance by an electromagnet controlled from outside the pile, will be included in the apparatus alongside the strain measuring microformer, so that any effects of temperature and radiation may be compensated. An entire duplicate creep apparatus may be placed in series with the load supplying cables outside the pile, so that the difference in creep rates, in-pile and out-of-pile, may be observed.

IRRADIATION IN A LIQUID METAL ENVIRONMENT

The Metallurgy Division is cooperating in an experiment to determine the induced radioactivity due to corrosion products and radiation effects in a

circulating lithium loop to be installed in the ORNL reactor. The effects of operating conditions on the radiation stability of the tube material will also be investigated.

Another experiment in conjunction with the North American Aviation Laboratories is using a deuteron beam from the Berkeley cyclotron to bombard materials in a high temperature, liquid metal environment.

SERVICE WORKS

PREPARATION OF CP-3 FUEL RODS

The Division has been asked to fabricate fuel rods for the CP-3 reactor at Argonne National Laboratory. This work is divided into two parts: the melting and casting of extrusion billets, and the extrusion of finished rods from the billets.

The melting and casting department has prepared eight ingots of pure aluminum and ten ingots of U-Al alloy containing 2% natural uranium. These alloys were prepared to develop techniques in melting, casting, and accountability procedures, as well as to supply billets for experimental extrusions.

The aluminum was melted in a graphite crucible using a high frequency induction furnace, and the uranium was added to the molten aluminum. The molten metal was stirred with a graphite rod to insure complete solution of the uranium, and was cast at 725°C into a preheated graphite mold. The alloy, when solidified without a hot top, developed an excessively large shrinkage cavity. A moveable heating coil was placed around the top of the mold to eliminate this pipe by controlling the rate of progressive solidification. Control analyses were made from dip samples taken immediately before casting. One ingot was sectioned longitudinally and analytical samples were taken from various points to check the homogeneity of the ingot. The analytical results were not consistent, but there was no evidence of segregation.

Preliminary extrusions have been made to determine the optimum extrusion conditions: temperatures of billet, die and container; die design; and extrusion rate. Data obtained during these tests are given in Table X. These data indicate the optimum extrusion conditions to be: (1) die—flat face, 1/32 in. radius on fillet, 1/8 in. bearing with a 30 minute relief on the throat, used at a temperature of 200°C. (It is believed that blueing the die prior to use improves the surface of the extrusion.); (2) billet—a temperature of about 280°C appears to give the most uniform extrusion with a good surface, (3) container temperature—although a temperature of 200°C has been adopted, no study has been made of this variable; (4) extrusion rate—a rate of approximately 40 ft per minute produces the best extrusion.

TABLE X

Extrusion of Aluminum and Uranium—Aluminum Alloys

	BILLET				DIE								EXTRUSION							
EXTRUSION ROD	MATERIAL	SIZE	FINISH	TEMPERATURE (°C)	TYPE	FILLET	BEARING LENGTH	TYPE THROAT	SIZE	TEMPERATURE (°C)	CONDITION START	CONDITION END	CONTAINER TEMP. (°C)	STARTING PRESS	RUNNING PRESS	APPROXIMATE EXTRUSION SPEED (ft/min)	DIAMETER OF ROD MAXIMUM	DIAMETER OF ROD MINIMUM	SURFACE QUALITY	REMARKS
1	Al	3-1/8	Mach	343	Flat	1/16	1/8	Straight	.850	*	Polished		300	2200	2200 1500	6				* Insert Dies. Heated about 15 min. in Container.
2	Al	3-1/8	Mach	343	Flat	1/32	3/16	Straight	.850	*	Polished	Good	300	1500	900 900	14				
3	Al	3-1/8	Cast	343	Flat	1/32	3/16	Straight	.850	*	Good	Good	300	1100	700	32			Fair	
4	Al	3-1/8	Mach	343	Flat	1/16	1/8	Straight	.850	*	Polished	Fair	300	1000	700	59			Spiraled	
5	Al	3-1/8	Cast	343	Flat	1/16	1/8	Straight	.850	*	Polished	Fair	300	1500	900	42			Spiraled	
6	Al	3-1/8	Mach	343	Flat	1/32	3/16	Straight	.850	*	Polished	Good	300	1200	800	46	.845		Good	
7	Al	3-1/8	Cast	343	Flat	1/32	3/16	Straight	.850	*	Polished	Good	300	1800	1000	35			Fair	
9	Al	3-1/8	Mach	285	Flat	1/32	3/16	Straight	.850	*	Clean	Good	300	1000	700	42	.850	.848	Good	
10	Al	3-1/8	Cast	285	Flat	1/16	1/8	Straight	.850	*	Clean	Good	300	1400	1000 600	35			Spiraled	
11	Al	3-1/8	Mach	400	Flat	1/32	3/16	Straight	.850	*	Clean	Good	300	800	800	42	.845	.844	Good	
12	Al	3-1/8	Cast	400	Flat	1/16	1/8	Straight	.850	*	Clean	Good	300	1200	1200 600		.852	.848	Spiraled	
13	Al	3-1/8	Mach	265	Flat	1/32	3/16	Straight	.850	*	Clean	Good	300	1700	1700 1000	38	.852	.844	Good	
14	Al	3-1/8	Cast	245	Flat	1/32	3/16	Choked	.850	200	Polished	Clean	200	1500	1500 1000	43	.840	.836	Rough	
15	Al	3-1/8	Cast	245	Flat	1/32	3/16	Choked	.850	200	Loaded	Clean	200	1500	1500 1100	42	.841	.837	Good	
1	4.5% U	3-1/8	Cast	490	Cone			Straight	.9	425	Clean	Loaded	300	600	600	5			Rough	
2	4.5% U	3-1/8	Cast	400	Cone			Straight	.750	450	Clean	Loaded	300	1150	1150 800	4			Rough	
3	4.5% U	3-1/8	Cast	343	Cone			Straight	.562	300	Polished	Loaded	300	1550	1550				Rough	
4	4.5% U	3-1/8	Cast	185	Cone			Straight	.750	180	Clean	Loaded	300	1550	1400	50			Rattlesnaked	
7	2% U	3-1/8	Mach	340	Flat	1/32	3/16	Straight	.850	*	Polished	Fair	300	1400	1400 800	42	.850	.844	Good	
8	2% U	3-1/8	Mach	340	Flat	1/16	1/8	Straight	.850	*	Polished	Fair	300	1400	1400 900	35	.851	.847	Spiraled on end	
9	2% U	3-1/8	Mach	340	Flat	1/32	3/16	Straight	.850	*	Clean	Fair	300	1300	900	42	.849	.844	Fair	
10	2% U	3-1/8	Mach	340	Flat	1/16	1/8	Straight	.850	*	Clean	Fair	300	1200	800	42	.851	.844	Fair	
11	2% U	3-1/8	Cast	295	Flat	1/32	1/8	Straight	.850	300	New	Fair	300	1600	1000	36	.		Fair	
12	2% U	3-1/8	Cast	295	Flat	1/32	1/8	Straight	.850	300	Clean	Fair	300	2000	2000 1000	42			Fair	
13	2% U	3-1/8	Cast	250	Flat	1/32	1/8	Straight	.850	200	Clean	Fair	250	1600	1600 1000	42			Fair	
14	2% U	3-1/8	Cast	250	Flat	1/32	1/8	Straight	.850	200	Fair	Fair	250	1800	1800 1200	30			Fair	
15	2% U	3-1/8	Cast	250	Flat	1/32	1/8	Straight	.850	200	Fair	Fair	250	1800	1800 1300	35			Fair	
16	2% U	3-1/8	Cast	250	Flat	1/32	1/8	Straight	.850	200	Fair	Fair	250	1900	1900 1300	28			Fair	
17	2% U	3-1/8	Cast	240	Flat	1/32	1/8	Choked	.850	200	Polished	Loaded	200	1600	1600 1100	42	.840	.837	Rough	
18	2% U	3-1/8	Cast	280	Flat	1/32	1/8	Relief 30 min.	.860	200	Polished	Clean		1400	1400	35	.855		Good	
19	2% U	3-1/8	Cast	280	Flat	1/32	1/8	Relief 30 min.	.860	200	Polished	Clean		1400	1400 800	35	.855		Good	
20	2% U	3-1/8	Cast	280	Flat	1/32	1/8	Relief 30 min.	.860	200	Good	Clean		1400	1400 800	35	.855		Good	

Aluminum cast billets were used for a large portion of the investigation of extrusion variables, since aluminum was thought to have approximately the same extrusion characteristics as the 2% alloy. This was not true, however, because the 2% alloy had a greater tendency to weld to the die than did aluminum

The following outlines the various changes in extrusion procedures that were tried to develop optimum conditions:

(1) Dies—Three general types of dies were used during the investigation. Since 25° cone dies were available, they were tried while two inexpensive insert dies were being made. The insert dies were used to obtain an approximate ideal of the bearing size and other pertinent data while normal dies were being made.

Variation in die design included the following: (a) 25° cone approach to a 3/8 in. bearing in the throat; (b) flat-faced die with a 1/16 in. radius on the fillet, 1/8 in. bearing, straight throat; (c) flat-faced die with a 1/32 in. radius on fillet, 3/16 in. bearing, straight throat; (d) flat-faced die with a 1/32 in. radius on fillet, 1/8 in. bearing, throat choked 30 minutes; and (e) flat faced die with a 1/32 in. radius on fillet, 1/8 in. bearing, throat relieved 30 minutes.

(2) Billet—Since recommended temperatures for aluminum extrusion range from about 250 to 460°C, billets were extruded through two different dies at intervals of approximately 50°C in this temperature range. A temperature of 200°C was used for one extrusion to see if a bright finish could be maintained over the full length of the extrusion. This was not successful.

If the billets could be extruded without scalping, a large saving in time and material would be effected. So both scalped billets (extrusions Al-1, 2, 4, 6, 9, 11, 11, and U-Al 7, 8, 9, 10) and unscalped billets (extrusions Al-3, 5, 7, 8, 10, 12, 14, 15, and U-Al-1, 2, 3, 4, and 11 through 20) were extruded. There appeared to be little or no difference in the extrusion of the scalped and unscalped billets.

(3) Container temperature—Although no study was made of the effect of container temperature, it was generally maintained 50-100°C below the billet temperature.

(4) Extrusion rate—The study of extrusion rate was limited to changing from a very slow speed through a medium speed up to a fast speed. A slow

speed tends to produce a rough extrusion. A fast speed heats the extrusion and is hard to control. Extrusion rates of approximately 40 ft per minute appear to produce the most uniform extrusion.

Additional work is needed to study the use of coated dies and the effect of extrusion rate in more detail.

PREPARATION OF ZIRCONIUM CORROSION SAMPLES

The Division was asked to vacuum melt samples of zirconium which were prepared by calcium reduction of zirconium tetrafluoride at the Materials Laboratory of the Y-12 Area, and to fabricate samples suitable for corrosion tests. The specimens were melted in graphite crucibles, using a high frequency vacuum induction furnace and allowing the charge to solidify in the crucible.

It was necessary to break up the as-cast structure by a preliminary hot forging at 900°C before cold rolling the samples to 1/16 in. plate. In the course of this work, specimens of iodide crystal bar and U. S. Bureau of Mines zirconium were fabricated in the same manner to permit evaluation of the relative rolling quality of the Ca reduced metal. The first production of the metal was found to be high in silicon and iron. This material could not be rolled due to the presence of an extremely hard and brittle phase (DPH 1000-1200). When the metal was prepared with a lower silicon content, a hard constituent was still present in the form of dendrites. However, this material could be rolled, after preliminary hot forging with a resultant break up of the dendritic structure. Apparently the Ca reduced zirconium, when substantially free of silicon, is comparable to Bureau of Mines zirconium, produced by the magnesium reduction of zirconium tetrachloride.

HANFORD TYPE SLUGS OF U-AL ALLOY

Fifty slugs, 4 in. long by 1.359 in. diameter containing 4.25% enriched U were cast and machined for use at Hanford. Reject plates from the MTR critical experiment run were used as the raw material.

Later a request was made by Hanford for similar slugs, 8 in. long, and containing about 7.5% enriched uranium necessitating melting larger batched

than heretofore used.

Considerable difficulty developed in obtaining sound castings and uniform distribution of uranium in the melt. Consequently, some work was done at ORNL with natural uranium to relate the melting variables to slug quality. Summarized results are:

1. A low pouring temperature, 725%, produced the least number of voids.

2. Rate of solution of U in liquid aluminum is an exponential function of temperature as shown in Fig. 13.

3. Mold temperature and pouring temperature had little effect on surface quality.

4. Remelt metal was superior to virgin metal from the standpoint of porosity.

5. There is always some gravity segregation; bottom of the slug is higher than the top by about 2% of the uranium content.

6. Analysis of the sample from the original melt accurately represents both the machined slug and the scrap from machining, provided homogeneity is attained prior to casting.

MISCELLANEOUS SERVICE

Service work performed for other departments and divisions include the following:

(1) Preparation of 4 extruded rods of 7.5% U - 92.5% Al for Hanford.

(2) Preparation of various metals and alloys for the ANP Program.

(3) Preparation of copper ingots for preferred orientation studies after extrusion.

(4) Melting of scrap platinum for reclamation.

(5) Production of silicon ferrite for growing single crystals.

(6) Production of copper alloys for radiation damage studies.

(7) Production of uranium alloys for extrusion.

(8) Consultation with Shielding Group regarding fabrication of Boral.

(9) Study of methods of rolling 4 ft × 5 ft sheets of uranium for shielding.

(10) Miscellaneous radiographic services.

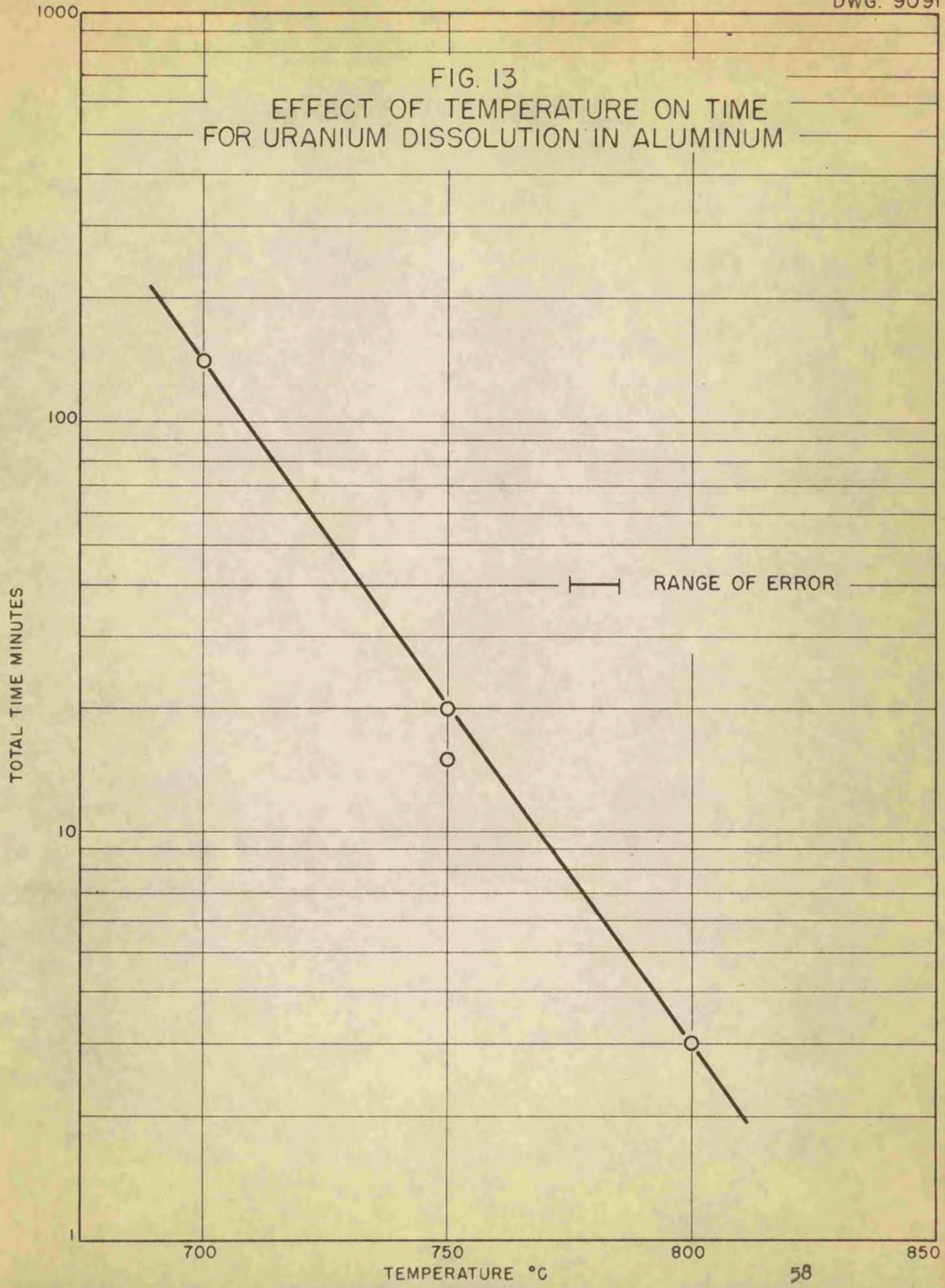


FIG. 13
EFFECT OF TEMPERATURE ON TIME
FOR URANIUM DISSOLUTION IN ALUMINUM

METALLURGY DIVISION
ORGANIZATION CHART

May 1, 1950

J. H. Frye, Jr. - Director		Roberta Hudson, Secretary
E. J. Boyle - Assistant to the Director		
L. E. Banker - Administrative Assistant		Helen Carr, Secretary Doris Jacox, Stenographer Dolores Poe, Stenographer Jane Thomas, Stenographer Mona Wiseman, Clerk-Typist
A. G. H. Andersen	Mechanical Testing, Properties of Pure Metals, and Mechanical Properties of Uranium and of High-temperature-resistant Alloys	R. T. Clem C. W. Weaver J. W. Woods
D. S. Billington	Radiation Effects and ANP	
J. T. Howe*	ANP	
G. T. Murray	Radiation Effects	F. M. Blacksher
J. C. Wilson	ANP	
B. S. Borie, Jr.	X-ray Diffraction	
R. J. Gray	Metallography	E. R. Boyd E. P. Griggs B. C. Leslie
R. S. Crouse		
L. K. Jetter	Fundamental Physico-Metallurgical Research	
G. H. Boss		J. C. Ogle
F. Kerze, Jr.	Reactor Component Development	
G. M. Adamson	Beryllium Fabrication and Procurement, Lead Creep	
C. D. Smith	Reduction, Melting, Casting, Rolling, Cladding (charge of 101-B Building)	C. F. Cutcher G. E. Cooley J. N. Hix H. J. Wallace

[redacted]

Metallurgy Division Organization Chart (cont'd)

W. D. Manly	Thorium Research and Clinton and Hanford Slug Problems	
J. H. Erwin	Hanford Slug Problem (charge of Metal Working and Heat Treating Room in 101-D)	G. D. Goldston D. E. Rosson J. P. Turner C. J. Zachary
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