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METALLURGY DIVISION

QUARTERLY PROGRESS REPORT

FOR PERIOD ENDING OCTOBER 31, 1949

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METALLURGY DIVISION

J. H. Frye, Jr., Director

QUARTERLY PROGRESS REPORT

for Period Ending October 31, 1949

Edited by

E. C. Miller

DATE ISSUED: JAN 12 1950

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SUMMARY

During the past quarter, work has continued on the ORNL (Clinton) and Hanford slug problems; the metallurgical development of components for the Materials Testing Reactor, including the mock-up and the criticality experiment; the recently initiated thorium program; and the fundamental studies of the mechanical properties of pure metals and of relations of preferred orientation to anisotropy in the properties of uranium and other metals.

The Division's radiation effects group is being expanded to work actively on the metallurgical problems connected with the Laboratory's solid state program. This group is presently engaged in completing the Solid State Group's hot laboratory facilities and placing them in operation.

The Division is just commencing an intensive investigation of metallurgical construction materials suitable for high temperature service in the presence of liquid metals. This work will be described in subsequent reports.

ORNL (Clinton) Slug Program. Aluminum-silicon bonded slugs have been received and are now under test in the program to develop methods of minimizing slug failures in the ORNL reactor. Study of the roles of magnesium and silicon in the diffusion between uranium and aluminum continues. No significant changes in dimensions or density, such as occur in Hanford pile irradiation, have been observed in heat treatment of U-Al alloys containing 5 to 40% U in the absence of irradiation.

Hanford Slug Problem. The thermal cycling experiments conducted to date indicate that cycling and end loading are critical variables in producing distortion in Hanford slugs. No significant distortion occurs when slugs are held at elevated temperatures without cycling. Increase in end loading appears to increase distortion. The tests are being extended to include alpha-rolled slugs.

Materials Testing Reactor Fuel Elements. As a result of the investigation of the causes of blistering in aluminum-clad U-Al alloy plates, modifications have been made in melting, rolling, cladding and brazing procedures which give greatly improved yields of blister-free fuel plates and assemblies.

Twenty fuel assemblies, containing enriched uranium, are being made by the Division for use in the criticality experiments to be performed in the MTR mock-up at ORNL.

Beryllium. The techniques of laboratory melting and casting have been substantially improved. Alloys containing small additions of Ti and Zr have been cast and are now under study.

Additional extrusion experience in the Adrian, Michigan plant confirms the feasibility of consistently producing sound 3¾ in. round-cornered square extrusions for the MTR, providing sound 8-inch billets are available. Recent billets containing small additions of aluminum, received from the Beryllium Corporation, were sound and extruded satisfactorily. These aluminum-containing extrusions are now under-going mechanical and corrosion tests.

Further possible modifications of extrusion technique are being investigated on the smaller press at ORNL.

Studies have been made to compare grain structure, mechanical properties, and corrosion behavior of beryllium metal extruded at different temperatures and annealed at different temperatures, and various types of hot-pressed beryllium.

Thorium. Zirconia shows promise as a crucible material for melting thorium. Cooling curves for the solidification of thorium indicate the probability of a slight thermal arrest at about 1625° C. Thermal analysis has confirmed the previously reported depression of the liquidus resulting from the addition of small amounts of beryllium or silicon. Preliminary tests have been made toward cladding aluminum and zirconium to thorium.

Mechanical Properties of Pure Metals. A research program has been formulated for the study of stress-strain-time-temperature functions of pure metals, determined from tensile and creep test data, to advance the understanding of plastic deformation. Special tensile and creep testing equipment is being developed for this and other applications.

Preferred Orientation and Anisotropy in Properties of Metals. A method has been devised for the determination of preferred orientation in uranium by a reflection method using a spherical uranium surface exposed to the incident beam.

URANIUM SLUG PROBLEMS

ORNL (Clinton) SLUG PROBLEM AND THE U-Al ALLOY SYSTEM

Slug Testing and Diffusion Studies. During the past quarter, a standard slug, which during the helium leak test prior to heat treatment had shown a leak in the aluminum can at the end weld, ruptured after heating for 309 days at 250° C. This substantiated prior evidence that slugs can fail in the ORNL reactor, after extended periods of time, by oxidation resulting from leaks in the aluminum can. It is felt that the use of Al-Si bonded slugs, as recommended in previous reports, will eliminate this type of failure and the failure resulting from possible can penetration caused by Al-U diffusion.

Fifty Clinton size slugs, canned by Hanford with Al-Si bonding between the uranium core and aluminum can, have been received. Three slugs are being heated in laboratory furnaces in the absence of radiation, at each of the following temperatures: 250, 350, 450, and 550° C. After 10 days treatment, none of the slugs showed external evidence of blistering or diffusion. Fifteen of the slugs will be placed in a channel in the ORNL reactor for irradiation tests. The air passages in the channel will be partially blocked off so that the surface temperature of the slugs will be 300° C instead of the usual maximum of 250° C.

X-ray diffraction examination of the diffusion layers in the uranium-aluminum alloy couples has been completed. The aluminum alloys had been prepared to study the effect of alloy additions on diffusion rate in the U-Al system. Successive layers were ground off the diffusion couples, and X-ray diffraction patterns were determined after the removal of each layer. The results are presented in Table I. No large differences were noted in the relative amounts of the phases present, except for the unidentified phases concentrated mainly at the interface between the aluminum and the uranium-aluminum compound.

It is possible that the alloy additions produce a ternary compound with U and Al. The presence or absence of unidentified lines in the various samples does not seem to affect relative diffusion rates since the ratio of UAl_2 to UAl_3 appears to be constant within the experimental error. The significance of the relative thicknesses of the phases shown in Table I is not apparent at present. In general, however, the Al-Mg alloys show relatively high diffusion rates.

TABLE I

Diffusion of Uranium and Aluminum or Aluminum Alloys
(Depth and Approximate Composition of Layers Formed at 450° C)

COMPOSITION OF Al (Percent Added Element)	TIME (hours)	THICKNESS OF COMPOUND LAYER (0.001 in.)	RELATIVE THICKNESS OF PHASES IN LAYER[a] (percent)			
			UAl_5	UAl_3	UAl_2	Other[b]
0.0	97	9	-	90	10	0.1
2.1 Mg	23	8	0.1	85	15	0.1
2.1 Mg[c]	23	> 90	0.2	> 76	-	0.5
7.6 Si	97	11	-	86	10	4
11.5 Si	97	20	-	86	10	4

[a] Relative thicknesses were obtained from x-ray data.

[b] The phase 'other' was present at the interface between the aluminum and diffusion layer. It represents unidentified lines which are not necessarily always the same.

[c] Work on sample was not completed; hence, no data on UAl_3 and UAl_2.

A possible explanation of the roles of Mg and Si in the diffusion between U and Al may be deduced from preliminary X-ray diffraction examination of the surfaces of the can and uranium slug of an Al-Si bonded Clinton slug. A phase was obtained which had the same lines as UAl_3, but the 2θ values were 0.8° larger than the standard UAl_3 lines. This suggests the possibility that Si can replace Al in UAl_3, with a consequent change in a_0 and possibly increased resistance to diffusion. In the case of Mg additions to Al, any Si present may be tied up as Mg_2Si and therefore inoperative in reducing the diffusion rate.

Thermal Studies of Uranium-Aluminum Alloys. Several months ago, a program was initiated to determine whether the 1 to 3% decrease in density of the 5% and 15% U-Al alloys irradiated in the Hanford pile could be duplicated by heating in the absence of radiation. Alloys containing 5% to 40% U were heated at temperatures from 150 to 450° C for time periods up to 36 days. The specimens were tested for density and hardness before and after heat treatment. No significant changes in dimensions or density were observed. Only the usual hardness changes associated with the annealing of these alloys were noted.

Before heat treatment all samples contained UAl_3. The ratio of UAl_3 to UAl_5 increased with decreasing uranium concentration, starting at 20% U. (This is in conflict with the present U-Al equilibrium diagram which predicts that no UAl_3 should exist below 60% U.) The heat treatment outlined above appeared to transform some of the UAl_3 to UAl_5, but in no case did complete transformation occur. The 5% U-Al alloy showed the least conversion of UAl_3 to UAl_5. When the alloys were slowly cooled, at a rate of 175° C/hr, from 950 to 650° C, UAl_3 did not appear in the 15, 20, and 25% U alloys, but some was retained in the 5 and 10% U alloys. A quench from 950° C caused retention of some UAl_3 in all alloys, particularly in the 20 and 25% U samples.

No satisfactory explanation has yet been developed for the phenomenon described above. Since an understanding of the U-Al system is fundamental to many reactor problems, such as the fuel elements in the Materials Testing Reactor, further work on this system will be carried out if time permits.

HANFORD SLUG PROBLEM

Cycling Tests in the Thermal Gradient Equipment. The following phases of the problem were investigated during the past quarter:

(1) Effect of constant thermal gradient after initial cycling. It was felt that some of the distortion occurring in slugs in the reactor may result from local overheating in areas where the bond between the uranium core and aluminum can is broken, thereby reducing heat transfer.

Four gamma-extruded slugs were cycled 10 times in one case and 20 times in another case, and then held at maximum temperature for periods of 300 and 500 hours. It was felt that the initial cycling might cause bond breaks which would result in local deformation during the period of holding at maximum temperature. After the 300 and 500 hour holding periods neither of the slug lines showed significant deformation. It would appear from this that cycling is very important in producing distortion.

The detailed data for the testing of all slugs during the past quarter are included in Table II. The slug lines discussed above, 19 and 20, were prepared in the manner described in ORNL 407. The aluminum can on each slug was machined off to a depth of 0.010 in. The tests on these lines were stopped when the aluminum cans ruptured at the ends of the uranium cores.

(2) Effect of increased rate of cycling. Since it is desirable to test the slugs as rapidly as possible, it was felt that a more rapid rate of cycling should be tried. Three slug lines, 16, 17, and 18, were prepared, using four alpha-rolled, triple-dipped slugs in each line. A 15 minute cycle was used with a heating period of 3½ minutes, a holding period at maximum temperature of 7½ minutes, and a cooling period of 4 minutes. The maximum slug center temperature varied from 450 to 530° C, with end loads from 2100 to 4200 pounds. The aluminum cans were again reduced by 0.010 in. in wall thickness with the contour turning tool.

Slug line 16 failed after only five cycles, by corrosion of the uranium where thermocouples had been placed to measure the thermal gradient. (Results appeared in ORNL 407). All slugs in slug line 17 showed an increase in diameter of 0.002 in., and three showed a decrease in length of 0.4% after 82 cycles. (It should be noted that length and diameter measurements are made on the aluminum cans and not on the uranium core. Diameter measurements are fairly accurate if separation does not occur between the can and the uranium core, but length measurements are only qualitative.) At the end of 180 cycles, the test was stopped by failure of the can on slug 55. The slugs again showed a decrease in length of 0.4% and one showed an increase in diameter of 0.001 in. About the same sort of behavior was shown by slug line 18. Photographs of slugs 58 and 59 from slug line 18 are shown in Figs. 1 and 2. The wrinkling

TABLE II

Hanford Slug Cycling Tests

	ROD NUMBER	SLUG NUMBER	NUMBER OF CYCLES	LENGTH OF CYCLES	END LOAD (lbs)	CENTER TEMPERATURE (°C)	TEMPERATURE GRADIENT (Calc °C)	CHANGE IN LENGTH (Percent)	CHANGE IN DIAMETER	POWER INPUT/FT OF U (KW)	COOLING H_2O VEL (ft/sec)	
Gamma Extruded Slugs (8 inch)	19	83 - 40	32	--	2490	480	340	None	None	109.0	39.4	Cycled 10 times--even increase 3¾ min, held 12½ min and even decrease 3¾ min--then held at full power except for necessary shut downs - was at full power 498 hrs, pulled end off No. 40 slug
	20	81 - 82	0 - 45	--	4200	450	325	81-0.4 82-0.4	81+0.003 82+0.002	97.5	39.0	Cycled 20 times--even increase 3¾ min, held 12½ min and even decrease 3¾ min--then held at full power for 229 hrs, except for necessary shut downs
	20**	81 - 82	45 - 51	--	4000	410	295	None	81+0.001 82-0.000	94.0	40.6	At full power 295½ hrs total--pulled end off of slug No. 82
	21	80 - 84	0 - 45	2 hr.	1000	520	385	None	None	111.5	38.5	Cycle--even increase 1 hr, at peak 30 min, cool 30 min--indication of slight bump at end of U in slug No. 80 from profile
	21**	80 - 84	45 - 56	2 hr.	750	495	365	*	*	107.0	39.3	End pulled off No. 80 slug--some U-Al alloy apparent on end of U--U about 3/8 inch longer than original
	23	39 - 85	41	2 hr.	1200	525	385	85-0.4	39+0.004 85+0.003	118.5	38.8	Cycle--even increase 1 hr, at peak 30 min, cool 30 min--broke at end of U in No. 39 slug and arced
	25	89 - 86	42	2 hr.	1500	410	290	None	None	105.0	41.0	Cycle--even increase 1 hr, at peak 30 min, cool 30 min--pulled end off No. 89 slug and arced (Al jacket full thickness)

* No. 80 slug grew about 3/8 in. in center of end. Examination indicated that the uranium had arced to the aluminum end caps and formed voids in the end of the uranium.

** Measurements of diameter and length changes after initial inspection relate to changes occurring after this inspection and are not total changes.

TABLE II (Cont'd)

	ROD NUMBER	SLUG NUMBER	NUMBER OF CYCLES	LENGTH OF CYCLES	END LOAD (lbs)	CENTER TEMPERATURE (°C)	TEMPERATURE GRADIENT (Calc °C)	CHANGE IN LENGTH (Percent)	CHANGE IN DIAMETER	POWER INPUT/FT OF U (KW)	COOLING H_2O VEL (ft/sec)	
Triple Dipped Alpha Rolled Slugs (4 inch)	16	48-49-52-45	5	1/3 hr	2200	450	320	--	--	96	39.7	Special thermocouples were inserted in the end slug to measure gradient oxidation which caused line to fail
	17	53-57-55-51	0-82	1/3 hr	4200	530	390	#57-55-51-0.4 #53 no change	all slugs +0.002	114.6	39.8	Cycle even increase 3¾ min, at peak 12½, cool 3¾ min—broke No. 41, reinstalling rod in unit replaced with No. 56, slight indication of swelling noticeable at center
	17**	53-57 55-56	82-180	1/3 hr	3600	490	370	#53-57-56-0.4 #55-3.2	#56+0.001 #55+0.085	111.4	44.8	No. 55 slug ruptured—U wrinkled in several places - measurements very doubtful
	18	54-58-55-50	0-151	1/3 hr	2100	500	370	None	#58+0.001 59+0.001 50+0.001	96.7	39.6	Cycle even increase 3¾ min, at peak 12½, cool 3¾—contour did not show any change
	18**	54-58-59-50	151-650	1/3 hr	2200	485	360	None	#50+0.001	96.4	39.3	Nos. 58 and 59 showed considerable water corrosion—No. 58 swelled in several places and burst in 3 places; No. 59 split approximately 2½ inches—no evidence of swelling
	22	61-62 63-60	28	2 hr	1000	535	400	None	None	112.0	39.8	Cycle even increase 40 min., at peak 40 min., cool 40 min.—broke and arced between 61 and 62 slugs, broke in joint
	24	72-79-71-66	0-60	2 hr	1300	420	310	#66-0.8	#72-.001 79+.002 66+.006	87.4	41.7	Cycle even increase 1 hr. at peak 30 min, cool 30 min.—small amount of swelling noticeable from profile, at joints
	24**	72-79 71-66	60-67	2 hr	1500	440		None	#66+.001			Resistance increase considerable in No. 72 slug—it was decided to check to determine the cause (all Al jackets full thickness)

* No. 80 grew about 3/8 in. in center of end. Examination indicated that the uranium had arced to the aluminum end caps and formed voids in the end of the uranium.

** Measurements of diameter and length changes after initial inspection relate to changes occurring after this inspection and are not total changes.

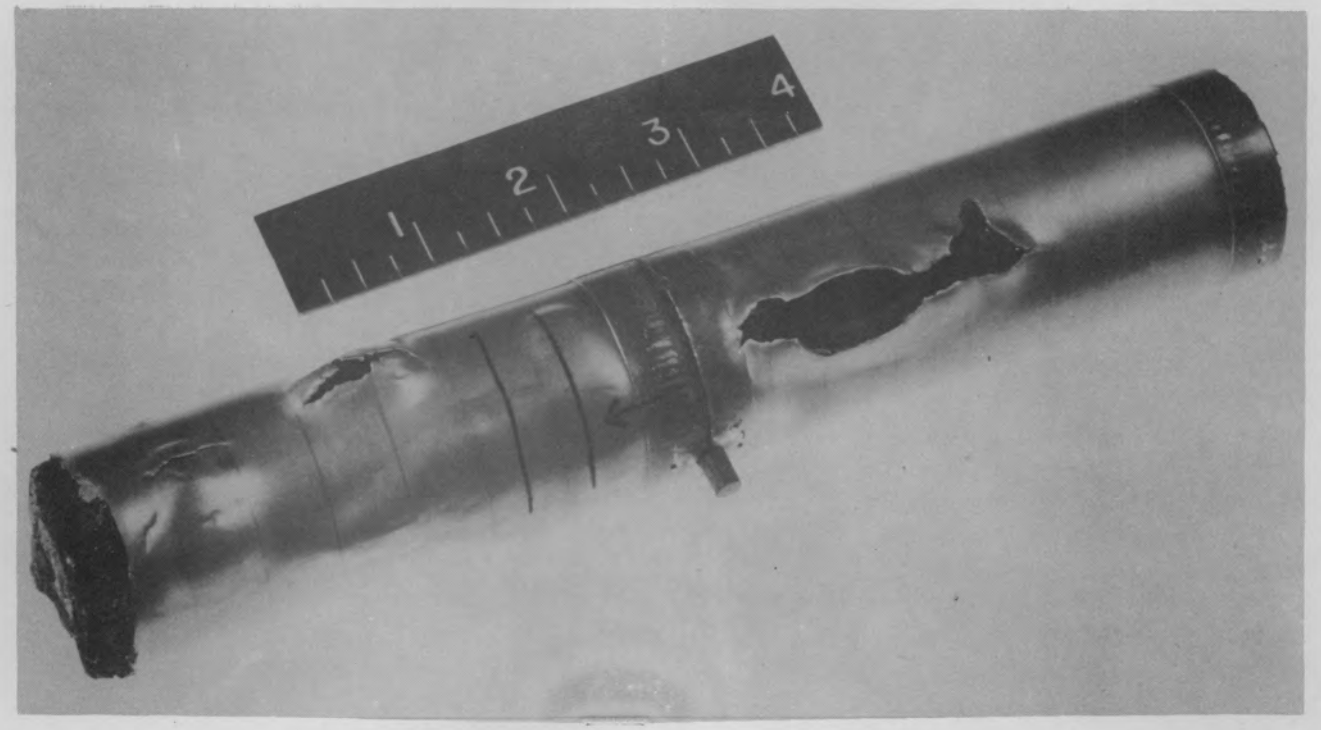


Fig. 1-Hanford 4" alpha rolled, triple dip slugs after 649 cycles. Y-460

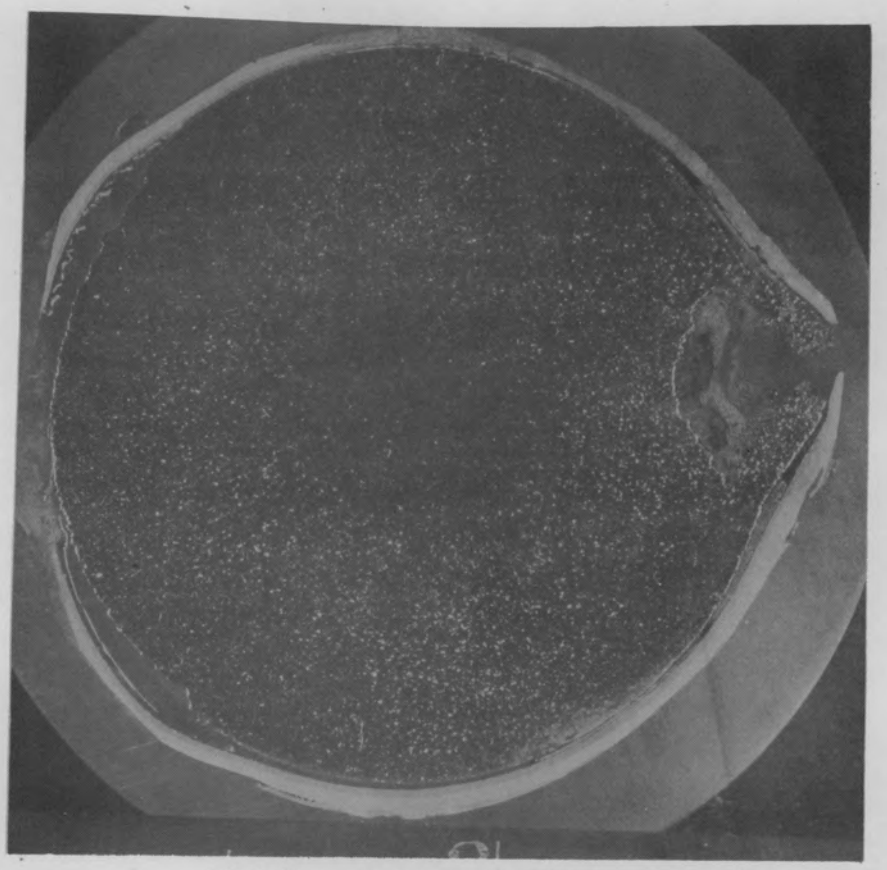


Fig. 2- Section of Slug 58 showing rupture in the can and severe water corrosion of the uranium core. Unetched, 3X. Y-570

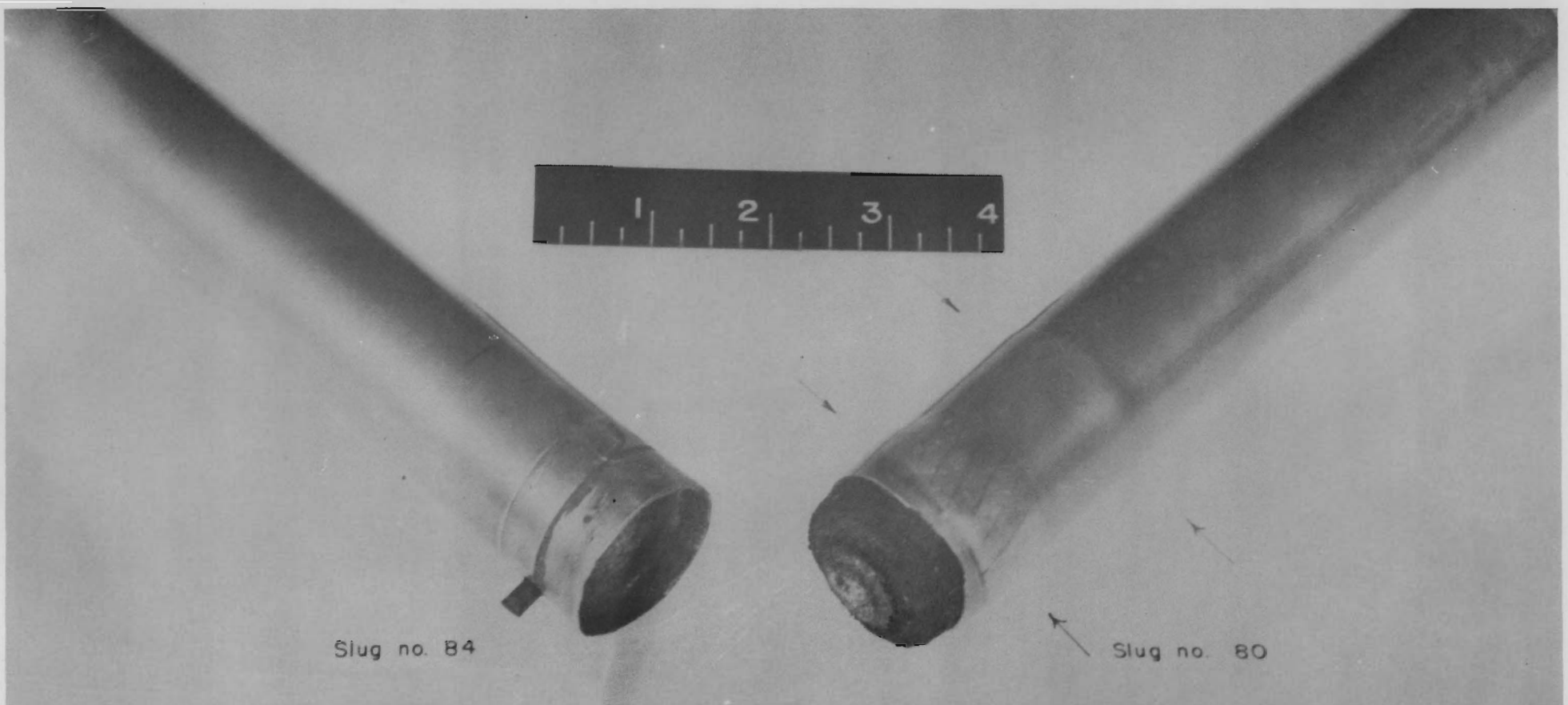
and eventual rupture of the cans which occurred on these slugs is typical of the behavior of the alpha-rolled, triple-dipped slugs in these fast cycling tests. It would appear that the cans wrinkle from differential expansion and contraction of the uranium core and aluminum can. According to Mr. E. A. Eschbach of the Pile Technology Group at Hanford, this same sort of can wrinkling occurs on alpha-rolled, triple-dipped slugs in the Hanford Piles.

(3) Effect of reduction of end loading on slug distortion. Up to the present time all slugs have been cycled in the thermal gradient equipment with end loads from 2000 to 5000 pounds. Since the end loading in the Hanford Piles is about 300 pounds maximum, it was decided to more nearly duplicate this loading in the present tests. Two slug lines, 21 and 23, were prepared with 8 in. gamma extruded slugs. The can thicknesses were reduced 0.010 in. and the lines were cycled under 750 and 1200 pounds end loads, respectively. Both lines failed by can rupture after relatively short periods of time, without showing the significant pimpling characteristic of gamma extruded material. A photograph of slug line 21 is shown in Fig. 3. The rupture of the aluminum can at the end of the underlying uranium core is typical of the failures which occur at these low end loads. Slug line 25 was tested as above, but the cans were left at full thickness, resulting in a somewhat lower maximum center temperature. This line failed in the same manner as 21 and 23.

Two slug lines, 22 and 24, were fabricated from alpha-rolled, triple-dipped slugs and were tested at low end loads as above. No. 22 had reduced can thickness while the cans of 24 were left at full thickness. Line 22 failed at the brazed joint without exhibiting any significant deformation. Line 24 showed some increases in diameter and decreases in length before there was a considerable increase in electrical resistance of one of the slugs. Testing of these slugs will be continued after a frost test is made to determine whether a bond break occurred in the slug showing the high resistance.

Results and Future Work. On the basis of the evidence obtained to date, it appears that cycling and end loading are critical variables in producing distortion in Hanford slugs. No significant distortion is produced when slugs are held at elevated temperatures without cycling. Distortion appears to increase with an increase in end loading on the slugs.

The failure of the slug lines by rupture of the aluminum cans continues to be a major problem. It is planned to devise techniques which will allow uranium bars with no joints and with suitable protection against water corrosion, to be tested. Several preliminary water corrosion tests were made on



Slug line 21

56 2 hr. cycles. 60 min. heating, 30 min. at peak and 30 min. cooling.
Average temperature 522° C with 1032 lbs. end load.
(Y-666)

FIG. 3

[redacted]

uranium bars electroplated with Ag, Cu or Cd. The coatings failed after 5 to 8 hours when heated in water at 80 to 90° C, with metal surface temperatures from 90 to 110° C. Consideration is being given to the use of a shrunk-on aluminum sleeve and to the use of cooling media other than water.

Reduction of Grain Size by Water Quenching Following Gamma Extrusion. Attempts were made to produce grain refinement in gamma extruded uranium by quenching the extruded bar as it left the die. At the same time, a trial was made of the efficiency of using molten glass as a protective heating medium and as a lubricant in extrusion. Seven 3 in. billets were extruded. All billets were heated in a glass composed of sodium oxide, sodium silicate, and borax. This glass worked quite well as a protective medium for heating uranium to temperatures as high as 1000° C. The glass, which adhered to the billets upon removal from the bath and insertion into the extrusion press, served as a lubricant during extrusion, and in some cases produced a uniform coating which protected the extruded rod against oxidation during air cooling to room temperature.

The pertinent extrusion data are listed in Table III. In one case, where the largest reduction ratio was used, a very good surface was produced on the extrusion. In all other cases, the initial part of the extrusion rod had good surfaces, but rattlesnaking was very pronounced on the trailing end. The quenching of the last three rods was accomplished by attaching a pipe to the to the die container and spraying water on the rod as it left the die and entered the pipe. Very effective cooling was accomplished; however, examination of microsections of the quenched rods showed about the same grain size as for the air cooled rods.

TABLE III

Extrusion of 3 in. Uranium Billets

EXTRUSION NO.	EXT. TEMP. °C	CONTAINER TEMP. °C	DIE TEMP. °C	DIE SHAPE	EXTRUSION RATIO	MAX. PRESSURE BILLET (psi)	RUNNING PRESSURE BILLET (psi)	DIE CONDITION (start)	DIE CONDITION (end)	METHOD OF COOLING EXTRUSION	REMARKS
2	900	350	450	0.9in. round	12:1	—	—	Fresh Polish	Scored, Seized	Air Cool	60 in. good and coated with glass, balance rattlesnaked
3	950	350	450	"	"	94,000	51,200	"	Good	" "	68 in. good, balance rattlesnaked
4	900	350	550	"	"	128,000	70,400	Clean	Badly Seized	" "	40 in. good, balance rattlesnaked
5	850	360	550	0.75 in. round	17.4:1	76,800	76,800	Fresh Polish	Good	" "	Good over entire length
6	850	375	500	0.9in. round	12:1	89,600	89,600	Clean, Cracked	Good	Quenched	60 in. good, balance rattlesnaked
7	850	375	—	"	12:1	89,600	64,000	Clean, Cracked	—	Quenched	36 in. good, balance rattlesnaked
8	900	375	450	1½ in. round	4.3:1	44,800	44,800	Fresh Polish	—	Quenched	20 in. good, some tears on balance but no rattlesnaking

REACTOR COMPONENT DEVELOPMENT

FUEL ELEMENTS FOR THE MATERIALS TESTING REACTOR (MTR)

Fuel Plates. (1) Blistering and Reduction Studies. Previous investigations (ORNL 407) indicated that the use of a dried air atmosphere in the furnace used to preheat the uranium-aluminum alloy for rolling and cladding decreased blister frequency. Yields obtained were about 76%. This occurred under conditions where the absolute humidity of the external atmosphere was about 8×10^{-4} lb/cu ft. A similar test using two ingots (N8R, N9R) melted with the regular practice gave yields of 2½% blister free plates when the external humidity was about 12.5×10^{-4} lb/cu ft, even though the atmosphere of the preheating furnace was dried. This indicated that drying the preheating furnace atmosphere would not alone guarantee freedom from blistering.

Table IV lists a series of tests conducted to study the effects of melting variables. Subsequent to reduction and remelting all were rolled and clad using standard procedures.

Analysis of the cryolite used as a flux in the reduction step showed that it contained 0.3% H_2O. In one experimental heat (N7) the cryolite was melted to drive off the water, then the aluminum was added and finally the U_3O_8. When the U_3O_8 was added, a violent reaction took place, spattering the hood with slag and metal. In another heat (N13) U_3O_8 and cryolite were charged into the crucible and heated to 600° C, and then the aluminum was added; the mixture was then melted at 115° C. The slag was separated and the metal pigged. The pig was remelted in air and the ingot rolled and clad, resulting in a yield of only 22%. In still another heat (N15), cryolite and U_3O_8 were melted and the aluminum was added. No violent reaction occurred. The resultant pig was remelted in vacuum and cast in air. A yield of 78% clad plates resulted.

Two pigs (N11, N12) which had been reduced with the standard procedure, were remelted in vacuum at different pressures. Yields were comparable, about 80%. When scrap from these two runs was remelted in air, the yield of blister-free plates was only 19%. One heat (N16) was melted in air using uranium metal and aluminum metal as the charge. The yield obtained was 56%.

These experiments indicated the possible desirability of melting the cryolite in U_3O_8 to drive off water before adding aluminum in the reduction heat, and of

TABLE IV

Effect of Melting Variables on Yield of Blister-Free Plates

HEAT	REDUCTION	REMELT	HUMIDITY x 10^{-4} lb/ft^3 Cast	HUMIDITY x 10^{-4} lb/ft^3 Roll	NON-BLISTERED PLATE YIELD (Percent)	REMARKS
N7	E	--	---	---	---	Violent Explosion
N8	S	A	12.6	12.1	5	Regular Practice
N9	S	A	12.3	12.9	0	Regular Practice
N11	S	V	---	11.4	80	28 μ pressure
N12	S	V	---	11.4	78	210 μ pressure
N13	D	A	12.1	14.5	22	-----
N14	--	A	11.4	9.9	19	Scrap from N11, N12
N15	C	V A	13.1	12.4	78	Ingot cooled in air
N16	---	A	11.8	12.4	56	U metal + Al metal
N17	S	V A	6.9	8.0	67	Dried Air bled into system after melting. Ingot poured in air and cooled in desiccator
N18	S	V A	6.2	8.0	45	
N19	C	V A	8.6	7.5	100	
N20	C	V A	8.9	7.5	90	
N21	C	V A	5.0	5.3	100	
N22	C	A	5.0	5.3	65	Ingot cooled in desiccator

Legend

S - Standard Reduction Procedure, melt Al, add cryolite - U_3O_8 mixture
C - Modified Reduction Procedure, melt cryolite - U_3O_8 mixture add Al
D - Heat cryolite, U_3O_8 to 600° C, add Al, melt
E - Melt cryolite, add Al, add U_3O_8
A - Standard remelt - melt and cast in air
V - Vacuum cast
VA - Melt in vacuum (~100 μ), cast in air

vacuum remelting and casting. However, vacuum remelting and casting represent a time consuming operation and make it difficult to sample for chemical analysis as well as to stir the melt to avoid segregation. In heats N17 through N21, the product of the reduction heat was melted in vacuum, poured in air, and allowed to cool in a desiccator. The results, presented in Table IV show that the highest yield resulted from heats N19 to N21 in which the aluminum was added to a molten cryolite and a U_3O_8 mixture, and the reduced material was vacuum and remelted and then air cast.

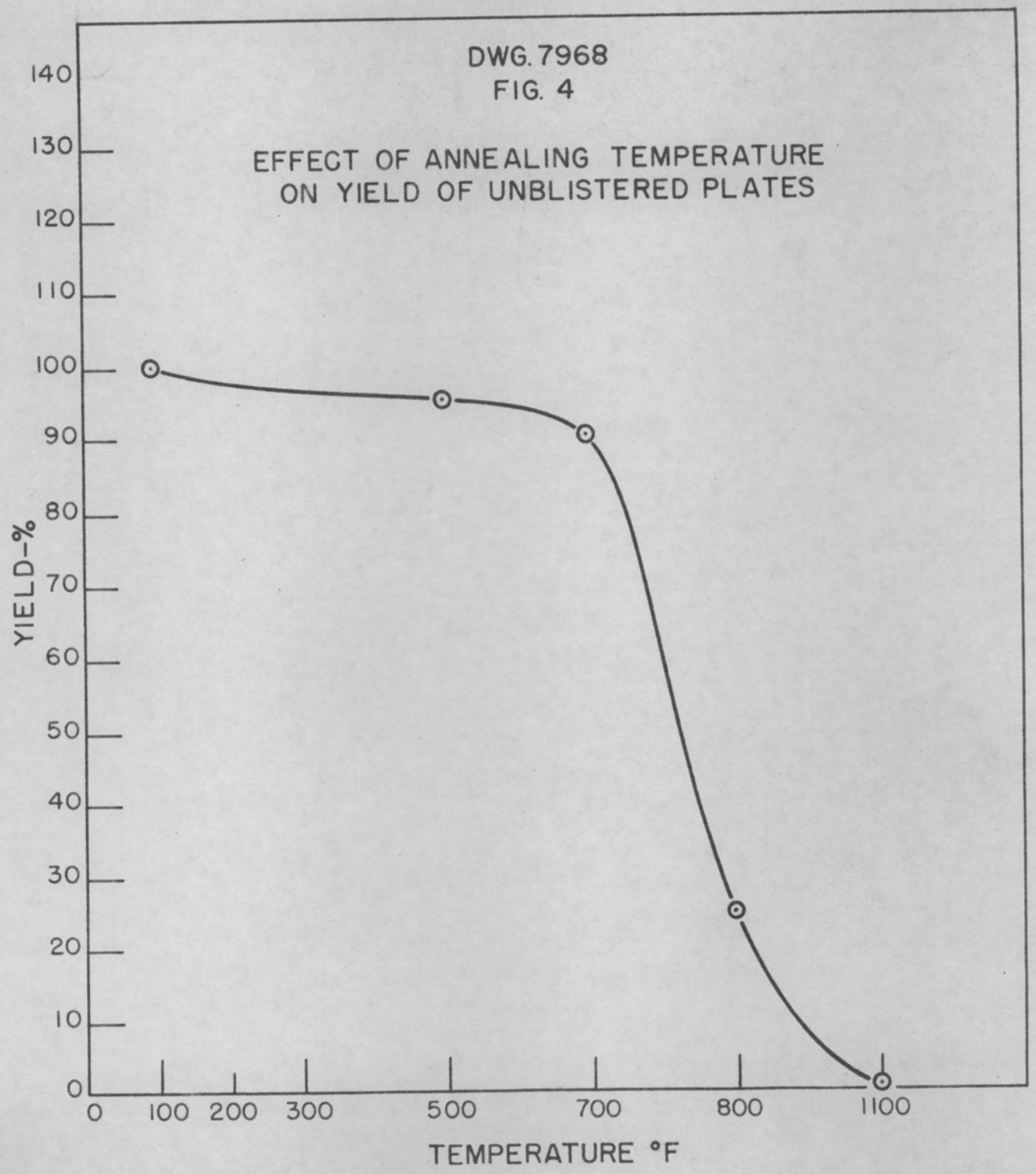
To obtain data on the effect of annealing temperature on blistering, one heat (N10) was melted in air and preheated for rolling in an ordinary (not dried) air atmosphere. Plates rolled from this ingot were annealed successively for three hours at 500, 700, 900 and 1100° F. After each anneal, plates were cooled to room temperature and number of blisters and blistered plates recorded. The results are shown in Fig. 4.

(2) Inspection. After cladding, cores are located using the fluoroscopic screen of the X-ray machine and a steel gage machined as shown in Fig. 5. The narrower sections at the ends (2.5 in.) are lined up with the core edges. A line is then scribed on either side at the wide part of the gage (3 in.) and the plate is sheared along these scribed lines. The scribing and shearing operations are subject to error so that after shearing the plate is not always well-centered. After shearing, the plates are machined to 2.839 in. ± 0.002 in. Roughly 0.080 in. is machined from each edge. If there is at least 0.0160 in. aluminum on each edge after shearing, the chances of machining into the core are very low. Accordingly a go-no-go gage similar to the marking gage was made up for checking core location and for shearing. The wide dimension (3 in.) is lined up with the sheared edges and then viewed on the fluoroscope. If any part of the core is outside the narrow dimension of the gauge (2.680 in.) it has less than 0.160 in. aluminum on the edge at that point and is rejected.

(3) Prerolling, Cladding. Since the U-Al alloy core is harder than the aluminum frame, lateral asymmetry of position might promote curving during rolling. To study the effect of core centering in the frame, ten cores from one ingot (N20R) were centered within 1/32 in. and ten were put off center by 1/2 in.

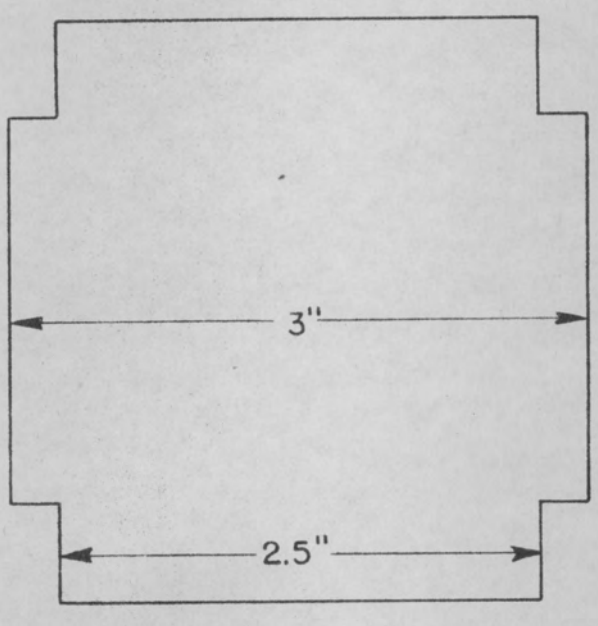
Examination of the off-center plates after prerolling showed large voids at the corners of the cores nearest the frame edge. Yields of blister-free plates from these rollings were (a) centered cores, 90%; and (b) off-center

[redacted]

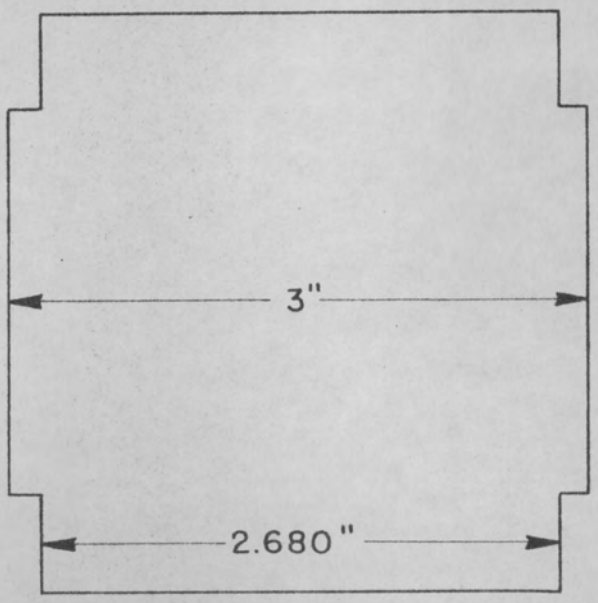

DWG. 7968
FIG. 4
EFFECT OF ANNEALING TEMPERATURE
ON YIELD OF UNBLISTERED PLATES
YIELD-%
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
0
100
200
300
500
700
800
1100
TEMPERATURE °F

[redacted]

DWG. 7969
FIG. 5



GAGE FOR MARKING CLAD PLATES FOR SHEARING



GO-NO GO GAGE FOR INSPECTING SHEARED PLATES

cores, 60%. All of the plates were radiographed. Their curvature varied from zero to 0.025 in. in 12 in. of length, but there was no significant difference between the two classes.

To study the effect of scratch brushing core and frame prior to cladding, half the cores from each of two heats (N21, N22) were scratched and the other half were not. After cladding, all were given a 3 hour test anneal at 1000° F. None of the plates from ingot N21 (vacuum melted) and none from N22 (air melted) which had *not* been scratched, were blistered. Destructive testing of samples from each type showed no difference in bond strength. Samples have been submitted for metallographic examination.

(4) Shearing Clad Plates to Size. The product of the rolling and cladding operation is a plate 4 in. wide, containing a core 2.5 in. wide. Present practice is to shear to 3 in. width and machine to 2.839 ± 0.002 in. Considerable saving could be effected by eliminating the machining operation. Experiments were conducted using the ¼ in. power shear, fitted with a guide on the front of the table. It was found that cold rolled aluminum could be sheared perpendicular to the rolling direction with a variation from straightness of only 0.003 in. per 24 in. length. However, when clad plates were sheared the edges curved. This was more serious with cold rolled plates than with annealed plates. Variation averaged 0.010 in. with cold rolled plates and 0.006 in. with annealed plates.

Critical Experiment Requirements. Twenty fuel assemblies containing enriched uranium will be manufactured for a critical experiment to be conducted in the MTR mock-up. Specifications for the active plate thickness have been changed from 0.064 in. ± 0.001 in. to 0.060 in. ± 0.001 in. Plans are complete for storing, guarding and handling the active material when it becomes available.

Brazing of Fuel Assemblies. (1) Flux Inclusions. Flux inclusions in aluminum brazing present a considerable problem if the aluminum is to be put in contact with aqueous systems. Corrosion of the joined surfaces and of the filler metal is accelerated by the presence of halide fluxes as inclusions within the brazed joints. However, corrosion tests conducted in the Technical Division have shown that MTR brazed fuel assembly sections can withstand simulated reactor cooling water for periods much greater than the expected time in the reactor, and that no excessive corrosion can be traced to flux inclusions.

Since the fluxes used in brazing fuel assemblies contain small amounts of lithium compounds, consideration must be given to flux inclusions because of of the high neutron cross section of lithium. Analyses of representative brazed assemblies show the lithium to be below the accepted tolerance limit, but these samples are only spot checks and it is desirable to keep flux inclusions at a minimum thus lowering probable lithium entrapment. Flux formulation studies gave encouraging results. Joints of simple design several inches in length were produced by furnace brazing using fluxes containing no lithium compounds, but full size assembly joints employing these fluxes showed insufficient fluxing action and incomplete flow.

Tests were made to evaluate some of the factors influencing flux inclusions, by visual examination of the cross sections of assembly joints. Atmospheric exposure of inclusions results in moisture pick-up, due to the hygroscopic nature of the brazing fluxes. Examination at 20X after exposure disclosed tiny droplets of moisture at flux concentration points. The surfaces were prepared for exposure by sawing and machining, which produced fresh, smooth, readily examined surfaces.

(a) Flux Composition

If the physical properties, density, viscosity and surface tension, of one flux mixture differ, the tendency to remain entrapped within a given molten metal may vary. The two fluxes previously found to produce satisfactory assembly brazing, Eutectic #190 and Alcoa #33, were employed in brazing one assembly. One side of the assembly was brazed with the Eutectic flux, the other with the Alcoa flux. Other conditions were held constant, except possible non-uniform temperature distribution. The results showed inclusions in both sets of specimens, with some slight advantage to the Alcoa flux. This test will be repeated several times, reversing the respective furnace positions of the two fluxes. Table V shows the number of inclusions to be large in both instances.

(b) Moisture Content

High temperature properties may be affected by the presence of combined moisture, as in dip brazing, where aluminum sheets are dipped into the molten flux bath until there is no further reaction with the aluminum. Tests are planned to determine the effect of moisture and combined moisture on the quality of furnace brazed joints.

TABLE V

Comparison of Commercial Brazing Fluxes

PIECE	LOCATION OF CUT (FROM A END)	ALCOA #33		EUTECTIC #190	
		INCLUSIONS	VOIDS	INCLUSIONS	VOIDS
1	2-1/2 in.	12	4	21	4
2	7 in.	26	0	57	1
3	12 in.	28	0	47	1
4	14-1/4 in.	33	2	34	4
5	16 in.	29	1	43	7
6	21 in.	39	3	41	3
7	26-5/8 in.	12	2	22	3
	Total	179	12	265	23

Assembly No. 169 - Sectioned for Flux Inclusions

Braze metal - 11-1/2% Si

Drying temperature - 300° F

Preheating temperature - 850° F 50 minutes, Hevi Duty Furnace

Brazing temperature - 1100° F 45 minutes, Lindberg Furnace

Brazing jig: Alsimag No. 202, four component type

Examination at 20X after 48 hours exposure

(c) Cleaning Practice

As reported in ORNL 407, cleaning practice may cause blister formation during fuel assembly brazing. The usual cleaning methods for the preparation of aluminum for brazing have been found unsuited to our purpose because of this. However, investigations may disclose acceptable modifications, if advantages resulting from etch type cleaning methods make them desirable. One advantage may be in the matter of flux inclusions. To determine the difference between joints obtained when etching methods are used as opposed to those obtained when vapor degreasing is employed, an assembly was prepared, one side of which was cleaned according to one technique, and the other side according to the other technique. The active plates, mounted for cleaning in the usual manner, were degreased in the vapor degreaser. After drying, they were dipped only partially into the aqueous etching type baths of hot caustic and nitric acid. Similarly, one side plate was degreased while the other plate was both degreased and etched. After fluxing, drying, and brazing, the assembly was examined. Both sides were well brazed. The assembly was sectioned and examined after exposure for flux inclusions. The side which had been subjected to the etch type cleaning showed fewer inclusions than the side which was degreased only.

(d) Position of Flux

An assembly was produced, one side plate of which had been fluxed prior to the insertion of the active plates. This resulted in greater spillage of flux on the surface of the drying jig and consequently on surfaces of the assembly not requiring flux. After brazing, the assembly was examined for braze quality. The bonding down both sides was good. The assembly was sectioned and examined for flux inclusions. More inclusions were noted on the side treated with the additional flux.

(e) Position of Filler Metal

Many commercial examples of aluminum brazing employ braze clad sheets of aluminum or aluminum alloy. The combs employed in the fabrication of fuel assemblies for the MTR are fabricated from such material. It was early considered advisable that side plates be fabricated from similar material, but commercial limitations precluded this use of the material. The groove design requires more filler metal than is available on commercial sheet. Efforts to have a thicker cladding produced for our use were unsuccessful. Attempts made to roll such a material at this site resulted in the separation of the braze alloy cladding material.

Some advantage in metal flow may result from the use of clad material augmented by the addition of suitable auxiliary filler metal. An assembly was prepared using braze clad material for side plates. To

recheck the inadequacy of the available cladding, only one side was supplemented with additional filler metal. It was given the usually used strips of 11½% silicon brazing alloy, placed on alternate active plates. The total thickness of the side plates used was 0.168 in. containing approximately 0.008 in. of brazing alloy on one surface. Machining the grooves removed about one-half of this. The resulting assembly showed incomplete brazing on the side provided only with the clad side plates. Adequate brazing was evident along the other side plate. Sections were prepared and exposed for flux inclusion examination. This disclosed inclusions of similar size and frequency to those usually obtained from assemblies with 2S aluminum side plates, active plates, and strips of aluminum silicon brazing alloy.

(f) Time and Temperature of Brazing

The time and temperature chosen for the brazing operation were selected after consideration of assemblies successfully brazed after varying these conditions separately. It was considered desirable to employ as low a temperature as possible to avoid the danger of diffusion of the uranium-aluminum alloy core into the cladding. It was also considered advisable to keep time at a minimum to avoid high temperature attack of the molten fluxes on the aluminum surfaces. No study has been made of joint quality from the flux inclusion standpoint under various conditions of time and temperature. It is planned to investigate these variables further.

Fuel Assembly Uniformity. Improvements in assembly uniformity are desirable, especially with respect to the dimension designated as vertical center height. The assemblies fabricated for the reactor mock-up showed considerable variation in this dimension. Close inspection of the assembly components disclosed wide variations in comb dimension. The position of the top plate with respect to the bottom plate can be influenced by the comb dimension. If the comb accomplishes its purpose it will braze to both plates and set the vertical center height dimension. Close inspection was made of a series of combs and side plates. Side plates were selected in pairs according to the measured distance between their top and bottom grooves. Combs were chosen in pairs to match these dimensions, allowing 0.004 in. to 0.006 in. for the two joints to top and bottom plate.

These groups of parts were then used in the fabrication of a series of assemblies. The following results were obtained from a dimensional examina-

tion of these assemblies:

ASSEMBLY	ACTIVE PLATE THICKNESS (inches)	VERTICAL CENTER HEIGHT (inches)		COMB LENGTH (inches)	SIDE PLATES AVERAGE DISTANCE TOP TO BOTTOM GROOVE (inches)
		Maximum	Minimum		
171	0.064	3.078	3.065	2.946	3.014
174	0.064	3.080	3.065	2.946	3.014
177	0.060	3.074	3.065	2.941	3.013

One of these assemblies, 177, used active plates 0.060 in. in thickness. A change has been made in the plate design thickness to permit the use of more cooling water. The purpose of 0.060 in. plates in assembly 177 was to study the changes in brazing, particularly with respect to side plate and comb fit. No change was noted in the side plate joining. However, some active plates were not joined to the comb on both surfaces. Only about half the surfaces of the comb were brazed to the active plates. Since the comb spacing measures 0.067 in. for 0.064 in. active plates it will be necessary to produce combs with spacing of 0.063 in. for active plates 0.060 in. in thickness.

Dimensional Changes after Thermal Cycling. An assembly was selected and re-inspected before submission to the Technical Division for thermal cycling. This cycling was accomplished by alternating the flow of steam through the inside of the assembly and cold water to the exterior. The time of the first set of cycling tests consisted of 130 seconds of steam followed by 120 seconds cold water. After 274, 1424, and 1924 cycles, the assembly was completely re-inspected. Dimensional changes observed were within the accuracy of the original measurements, particularly considering the difficulty of relocating points for measurement. After two thousand more cycles were run, each consisting of ten minutes of steam followed by two minutes of water, the assembly showed no observable dimensional change beyond the range of accuracy of the measurements. This work has been reported in ORNL 473.

The removal of brazing flux is considerably more difficult if post-brazing etching types of cleaners are avoided. One of the best methods for flux removal is quenching after brazing. When parts permit immediate quenching no further cleaning is necessary. It was considered desirable to determine how severe a quench could be employed without distortion of the assembly structure. A fitting was provided on one end of an assembly to permit vertical quenching after suitable heating. Beginning at a temperature of 300° F, the assembly was successively quenched into cold water (75° F) after several 50° F incre-

ments. After each cycle the assembly was inspected for dimensional change. Table VI shows the changes after quenches from 400°, 450°, 500°, and 550° F. Changes observed after quenching from 300° and 350° C were less than the limits of accuracy of measurement.

Since quenching is most effective for flux removal when carried out from temperatures of 500° F and higher, it cannot be recommended because of the relatively large dimensional changes taking place when the operation is carried out on assemblies at those temperatures.

Modifications in Fabrication of Fuel Elements. Short active plate design has included the slotting of fuel plates to provide accurate positioning of braze metal clad combs. Since this detail of design was added, additional positioning elements have been fitted to the drying jig. These provide positioning of the combs with respect to the plate center-line. It has been determined that combs thus positioned do not require the slots in the active plates. Adequate positioning and joining has been achieved on the last five fuel assemblies, none of which made use of slotted active plates.

(1) Shearing. An assembly was prepared from sheared plates containing natural uranium-aluminum alloy cores. This assembly, 177, passed dimensional inspection showing measurements similar to assemblies made from machined plates under similar brazing conditions. The use of sheared plates will require a double acting die to flatten and hold the plates prior to shearing. Since only one assembly has been fabricated from sheared plates it will be necessary to produce a series of such assemblies to recheck the above observations.

(2) Plate Forming Die. Because of the repeated repositioning problems experienced whenever the die parts had to be removed from the hydraulic press, it was decided to set up permanently for plate forming. After inspection of the die components, it was found necessary to regrind the surfaces of the female section. In addition, a bolster plate of 3 in. boiler plate was provided to fit between the die female section and the press table. This was ground flat on both contacting surfaces. Shims were cut from brass shim stock to take up the gaps showing up because of irregularities of the press table.

After assembly of the die and bolster plate, it was possible to produce plates uniformly within ± 0.003 in. in sagitta, as measured from the surface plate.

TABLE VI

Dimensional Changes in Fuel Assemblies Resulting from Thermal Cycling

	DISTANCE FROM *A* END (inches)	QUENCHED FROM 400° F (inches)	TOTAL CHANGES 450° F (inches)	500° F (inches)	550° F (inches)
	2½	--	--	+ 0.007	+ 0.013
Uppitta	7	- 0.005	- 0.009	- 0.016	- 0.020
	12	- 0.006	- 0.008	- 0.011	- 0.013
	16	- 0.005	- 0.007	- 0.009	- 0.011
	21	- 0.007	- 0.010	- 0.010	- 0.013
	26-1/8	- 0.005	- 0.010	- 0.011	- 0.015
Sagitta	2½	--	- 0.005	- 0.009	- 0.014
	7	--	- 0.006	- 0.013	- 0.023
	12	- 0.004	- 0.008	- 0.011	- 0.015
	16	- 0.004	- 0.006	- 0.008	- 0.009
	21	- 0.003	- 0.006	- 0.008	- 0.007
	26-1/8	- 0.010	- 0.006	- 0.024	- 0.030
Vertical Center Height	2½	+ 0.003	+ 0.010	--	+ 0.020
	7	--	--	--	+ 0.003
	12	--	+ 0.003	--	+ 0.006
	16	--	+ 0.002	--	--
	21	- 0.004	--	--	- 0.005
	26-1/8	+ 0.006	+ 0.011	--	+ 0.018

Note - Quench Vertical in 75° F water - "B" end first

BERYLLIUM

Laboratory Melting and Casting. The casting of 4¼ diameter ingots of beryllium in the 12 in. diameter induction vacuum furnace has been continued, a total of 30 castings having been made. Previous conclusions about the desirability of casting the billets in an inert atmosphere rather than in a vacuum, have been substantiated. The use of argon or helium at a pressure of 11 psi during casting has eliminated the blowing of the liquid metal from the mold. Apparently this blowing from the mold is the result of vaporization of volatile condensate on the mold walls. Such condensate has been observed to oxidize with explosive violence when a furnace in which a melt has been frozen in the crucible rather than allowed to pour, is opened to the atmosphere.

It has been found that the quality of the virgin pebble beryllium used in melting has a marked effect on melting loss and ingot quality. Table VII lists results on two lots of beryllium pebble, PB-96 and P-96. Melts made from PB-96 showed dull surfaces on the ingots and poor metal recovery, considerable amounts of volatile material were evolved during melting, and difficulty was encountered in pouring, due to sticking of the stopper rods. On the other hand, lot P-96 produced clean surfaces on the ingots, good metal recovery, and no difficulty in pouring. Analysis of these lots, as supplied by the Brush Beryllium Co., was as follows:

	P-96 (per cent)	PB-96 (per cent)
Assay	99.50	98.29
Fe	.06	.05
Al	.108	.16
Si	.029	.028
	(ppm)	(ppm)
Mg	about 7500	about 2300
Cu	52	72
Ni	51	53
Mn	140	90
Ag	-1	1.4
Cd	-1	-1
Zn	160	35
B	6	.3
B*	2.9	

* Done by N.B.C. wet method

TABLE VII

Laboratory Beryllium Melts

NO. A	WEIGHT (kg)	MELTING TIME (min)	HOT TOP MATERIAL	REMELT (percent)	CASTING TEMPERATURE (°C)	CASTING PRESSURE	MAXIMUM PRESSURE (microns)	GRADE Be	CRUCIBLE NUMBER	METAL RECOVERY (percent)	COMPOSITION
105	5395	145	Al_2O_3	68	1400	100μ	500	PB-96	161	--	--
106	4400	118	"	93	"	150μ	300	"	161	--	--
107	4400	132	"	32	"	11.0 psi Argon	100	"	161	84.1	--
109	5300	135	"	70	"	"	50	"	161	88.8	--
110	5280	168	"	0	1415	"	140	"	161	--	--
111	3450	178	--	100	1400	"	140	"	161	--	--
113	3420	118	K-20	100	"	100μ	240	"	161	--	--
114	3380	103	"	100	"	11.0 psi Argon	300	"	161	--	--
116	5220	127	Al_2O_3	58	"	"	400	"	161	84.2	--
124	5275	136	"	0	1440	"	320	"	161	86.0	--
125	5295	117	"	64	1400	11.0 psi He	300	"	161	82.6	--
175	5250	130	"	0	"	"	600	"	201	74.0	--
179	5250	116	"	0	"	"	230	"	192	74.5	--
192	5300	122	"	0	"	"	300	"	191	69.9	--
194	5300	93	"	0	"	"	210	P-96	191	93.0	--
196	4270	97	Castolite	0	"	"	190	"	191	92.5	--
197	4380	115	"	41	"	"	200	"	191	93.5	--
134	5321	109	Al_2O_3	0	1400	11.0 psi Helium	300	PB-96	161	--	0.5% Ti
135	5301	60	"	0	1425	"	460	"	161	--	0.5% Ti
139	5213	196	"	0	--	"	1400	"	202	--	0.25% Ti
140	5200	102	"	0	1400	"	1000	"	202	--	0.25% Ti
141	5200	139	"	0	"	"	400	"	202	--	0.25% Zr
145	5200	159	"	0	"	"	500	"	201	--	0.5% Zr
146	5200	138	"	0	"	"	600	"	201	--	0.25% Ti
148	5200	142	"	0	"	"	700	"	202	--	0.75% Ti
158	5200	127	"	0	"	"	900	"	201	--	1% Zr
160	5200	96	"	0	"	"	400	"	201	--	0.1% Zr
161	5200	129	"	0	"	"	240	"	161	--	1% Ti
164	5200	196	"	0	"	"	280	"	201	--	0.1% Ti

Attempts were made to refine the grain size of beryllium ingots by additions of Ti and Zr. Alloys containing 0.1% to 1% Ti and 0.1% to 1% Zr were prepared according to standard Be melting practice, and cast into cold graphite molds of 4½ in. diameter. Photographs of transverse sections cut from the center of 4½ in. diameter billets are shown in Fig. 6. The results are not definite, but it appears that 1% Ti may have been effective as a grain refiner. More work will be necessary before conclusive evidence can be shown. Data for these alloys are listed in Table VII.

Extrusion. During this period two beryllium extrusion runs were made at the Gerity Michigan Company Plant in Adrian, Michigan, and one was made at ORNL.

(1) Extrusion at Adrian. Twelve billets were extruded on August 10 and 11, 1949. Seven were reported as being sound after having been radiographed in six positions instead of the usual three, and five billets contained internal cracks. Two of the sound billets contained small additions of aluminum, 0.5% in one case, and 1% in the other. All billets were heated to 1800° F and extruded with a ram speed of less than one-half inch per second. The general method of extrusion was that described in ORNL 407.

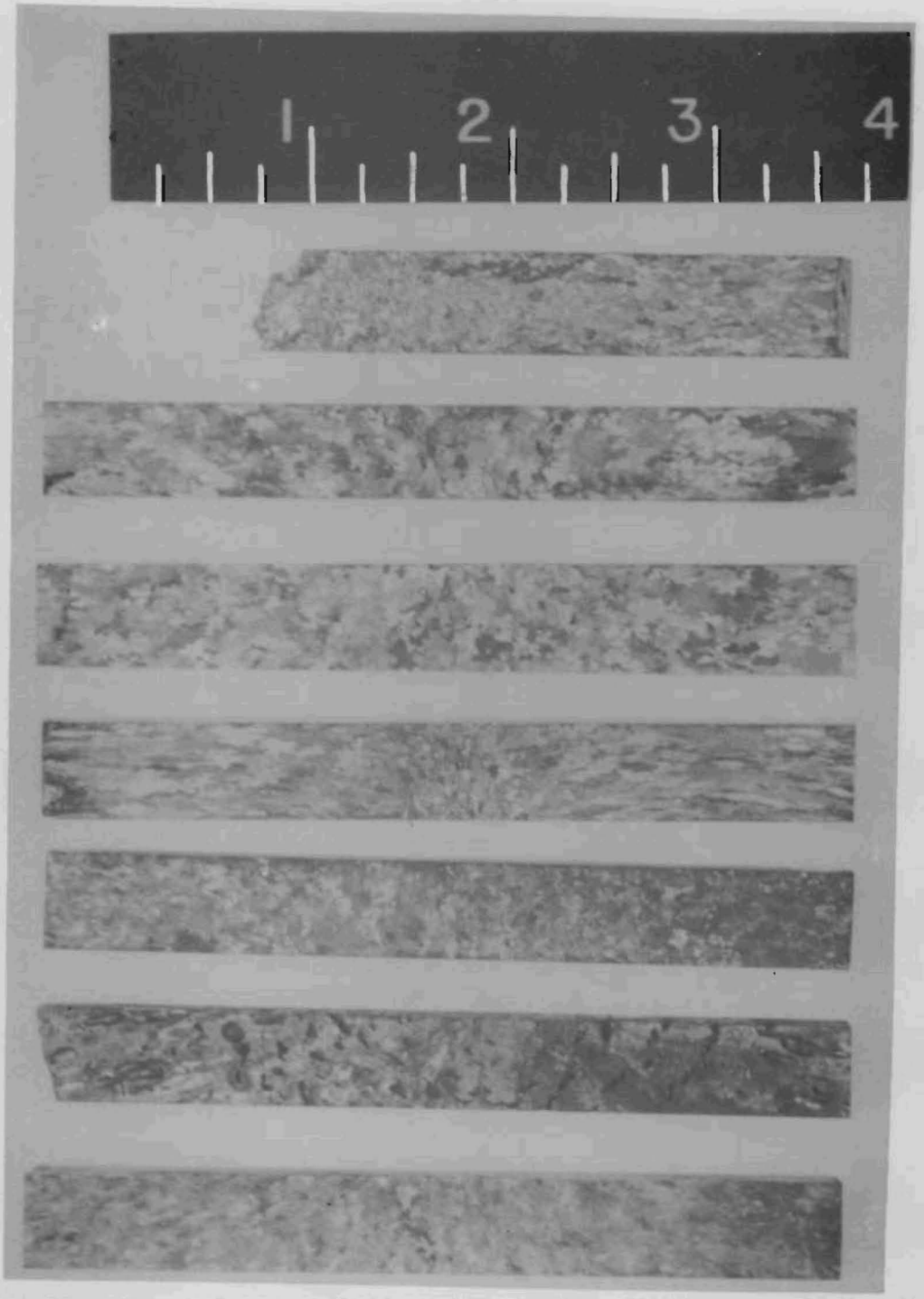
All extruded bars were cut into six inch lengths to permit investigation of internal cracks. After being chemically polished and etched, all the bars which did not contain aluminum additions were found to contain internal cracks. The sixth section, 36 inches from the leading end, was the first section that was consistently sound. The bars that came from the reportedly good billets contained fewer and smaller cracks than those from unsound billets, but all showed cracks. No cracks were found, by either fracture or chemical etching, in any section from the extrusions containing aluminum.

During the second extrusion on August 31, billets containing 0.1%, 0.25%, and 0.50% aluminum respectively, and one plain billet, were extruded. The extrusion conditions were identical with those of the previous run.

The bars were sectioned at the 6, 12, and 56 inch marks and the sections examined. None of the bars contained internal cracks. One did contain fine lines caused by contamination by a non-metallic impurity (beryllium carbide), but there were no cracks.

Corrosion and physical tests are being run on the entire series of aluminum-containing bars. These tests include both material annealed at 1800° F and as-extruded material.

Fig. 6



EFFECT OF Be GRAIN SIZE WITH Zr AND Ti ADDITIONS
(Y-691)

The Instrument Department at ORNL designed and built an instrument for recording instantaneous values of speed and pressure for any position of the ram during the working strokes. The instrument was installed at Adrian and worked very well during these extrusions. The records showed that the operator was holding both the pressure and speed much more uniform than was thought probable. Since the work reported in ORNL 407, twenty-five billets have been extruded into 3¾ round-corner squares using the technique mentioned previously. Of these twenty-five, only one contained a serious defect that could be traced to the extrusion. This indicates that a technique has been developed that is suitable for extruding the three-inch squares necessary for the MTR.

The status of the problem of casting 8-inch billets is not definite. Aluminum, even in low concentrations, seems to help eliminate cracks. However, since the last straight beryllium billet extruded was also good, it is possible that the improvement was due to some change in melting or casting technique. Since the cracks occupy such a small percentage of the bar volume it seems possible that even a small amount of a low melting phase such as beryllium-aluminum alloy could help alleviate them.

(2) Extrusion at ORNL. Since the large 8 in. diameter billets are both expensive and difficult to make, some work was scheduled at ORNL, using 3 in. billets and the same ratio of reduction as employed at Adrian. This work is summarized in Table VIII.

The extrusions made at the higher temperatures were carried out to see if a comparison could be made between the results obtained on the two presses, and indicated that such a comparison is reasonable. The major difference in technique between the two operations is that in Oak Ridge a one-piece die is used exclusively, eliminating the need for a cone on every billet. The radius on the corners of the die was 3/16 in. instead of the 3/8 in. used on the large press dies. Since the supply of cast billets was limited, some billets were made by machining a previously extruded bar.

The results show that copper cans lower the required pressure. At the lower temperatures the copper seemed to deform more easily than the beryllium, since in many cases the bar was cracked, but the jacket had not ruptured. Beryllium has a zone of maximum plasticity at about 850° F, and, to take full advantage of both this zone and the plastic properties of the copper jacket, extrusions at still lower temperatures will be attempted.

The corners of the bars were well filled under all conditions. This indicates that the radius of the corners of the large squares can be safely reduced to lessen machining losses.

TABLE VIII

Beryllium Extrusions at ORNL

EXTRUSION	TYPE BERYLLIUM	JACKET	TEMPERATURE (°F)	RUNNING PRESSURE (psi)	COMMENTS
1	Extruded	Steel Copper plated	1800	1300	Good bar
2	Cast	"	1800	1000	" "
3	Extruded	"	1600	1200	" "
4	Cast	"	1600	1300	(Speed very slow) cracked last ¼
5	Extruded	"	1400	1300	Cracked entire length
6	Cast	"	1400	1150	" " "
7	Extruded	copper	1400	1100	Good bar
8	Cast	"	1400	1050	Cracked entire length
9	Extruded	"	1200	1700	" " "
10	Extruded	"	1100	1400	Good bar
11	Extruded	"	1000	1400	Cracked entire length
12	Extruded	"	1000	1600	Cracked entire length

Testing. During the past quarter, comparative grain size, mechanical strength and corrsoion data were obtained on the metal types actually considered for use in construction of the Materials Testing Reactor reflector. Interest has centered on Adrian extruded (4.1 to 1 reduction) and QM and QRM grades of sintered powder metal.

Review of beryllium corrosion work to date indicates that hot-pressed metal is at least as corrosion resistant in simulated reactor cooling water as is extruded metal, and emphasizes the importance of completing corrosion tests to determine the relative resistance of sintered metals grades QM, QRM, and QMV in simulated reactor cooling water and in autoclave testing. Plans for corrosion equipment suitable for testing each beryllium piece prior to final acceptance for reflector use, are being considered.

(1) Grain Structure Comparison. Typical grain structures of sintered and extruded metal were examined metallographically to correlate mechanical testing and machining results. Transverse and logitudinal specimens were prepared from the center and edge of an Adrian-extruded billet (extrusion temperature, 1700° F). Grain refinement resulting from one-hour anneals at 1700, 1800, and 1900° F, was also observed. A refined, equi-axed structure resulted in each case. The grains from the 1900° C anneal were slightly larger than those obtained at the two lower temperatures. Polarized light photomicrographs (100X) of the as-extruded and the recrystallized grain textures are shown in Fig. 7. Included are macrophotographs of the hot-pressed QM grain structure.

(2) Compressive Tests. Room temperature compressive tests were made to obtain information on the effect of extrusion temperature on strength, the degree of anistropy found in extruded metal, and the relative mechanical strength of hot-pressed and extruded metal. Compressive testing was selected in preference to tensile testing because of the large number of samples involved and the greater conveniences of fabrication.

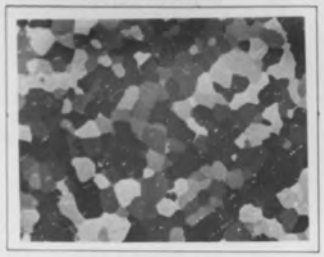
Specimens were 0.500 in. diameter by one inch long cylinders, with ends machined parallel to within at least 0.001 in. Care was exercised in selecting each longitudinal and transverse sample so that specimens representative of metal from the center and edge of the billet could be prepared. A 120 ton Baldwin-Southwark testing machine was used for loading. Deformation or change in length measurements were made by a 0.001 in. dial indicator mounted between the platens of the subpress. A constant strain rate of 0.05 in./min. was used in all tests.

NOT CLASSIFIED

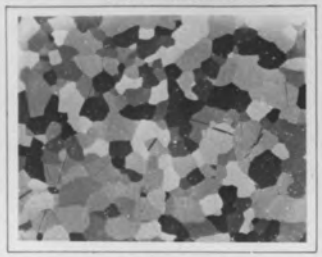
EXTRUDED BERYLLIUM

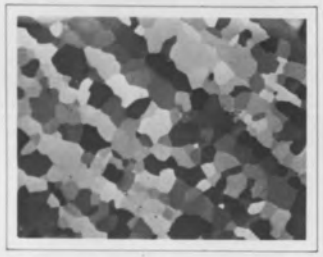
SURFACE(A) — LONGITUDINAL — TRANSVERSE

CENTER(B) — LONGITUDINAL — TRANSVERSE



POLARIZED LIGHT 100X — AS EXTRUDED AT 1700°F — POLARIZED LIGHT 100X





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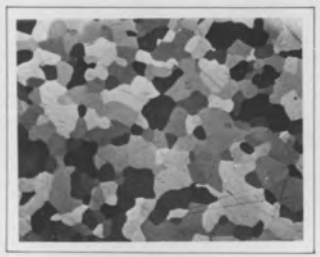
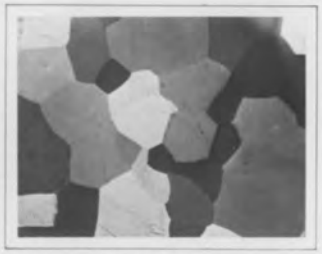
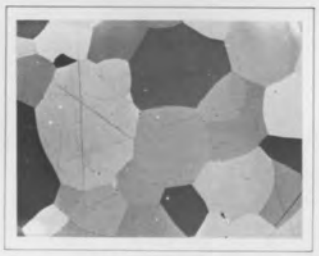
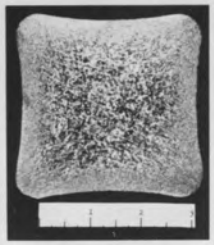




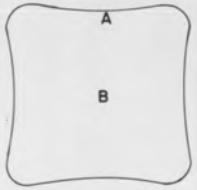
POLARIZED LIGHT 100X — ANNEALED AT 1900°F — POLARIZED LIGHT 100X



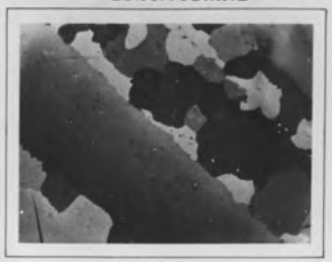
TRANSVERSE



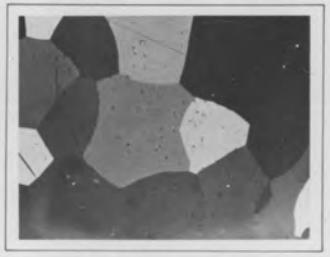
AS EXTRUDED AT 1700°F



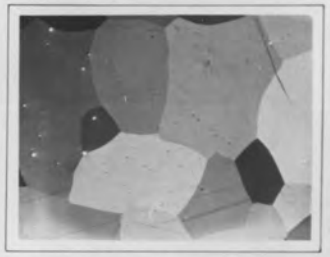
5% SULFURIC ACID 1X

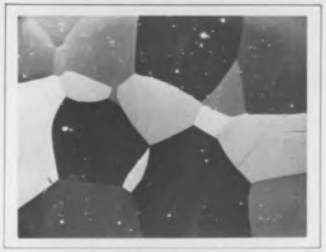


POLARIZED LIGHT 100X — AS EXTRUDED AT 1700°F — POLARIZED LIGHT 100X

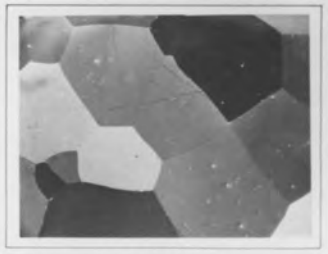


POLARIZED LIGHT 100X — ANNEALED AT 1700°F — POLARIZED LIGHT 100X





POLARIZED LIGHT 100X — ANNEALED AT 1900°F — POLARIZED LIGHT 100X



HOT PRESSED QM BERYLLIUM

5% SULFURIC ACID 1X

POLARIZED LIGHT 100X

Fig. 7

GRAIN STRUCTURE OF EXTRUDED, ANNEALED, AND HOT-PRESSED BERYLLIUM

Metallography by R. J. Gray

Y-665

Compression test results on vacuum cast metal extruded at 1400, 1600, 1700 and 1800° F with a reduction in area of 4.1 to 1 are given in Table IX. Also included are results on QM and QRM grades of sintered metal, and 6.5 to 1 reduction metal extruded at 1870° F. All specimens were run in duplicate.

Although the data reveal no clear-cut correlation, the compressive strength appears to increase in the transverse direction and decrease in the longitudinal direction with an increase in extrusion temperature within the range tested. As expected from examination of the grain structures and from work previously reported by MIT, the extruded sections tested show a high degree of anistropy. Differences of properties were also evident between center and edge samples. These differences were not observed in the sintered metal.

Extruded metal is substantially weaker in compression in the transverse direction, but generally stronger in the longitudinal direction than in sintered metal.

Stress-strain curves are plotted in Fig. 8. For convenience in plotting, the curves have been grouped into four categories with respect to location and direction of extruded samples: transverse center, transverse edge, longitudinal center, and longitudinal edge.

The curve dip or bow found only in extruded metal compressed in the longitudinal direction may be due to the formation of twins. An analogous twinning phenomenon is found in the compression magnesium metal.

A similar set of tests is planned on metal in the annealed condition, and on extruded metal to which small additions (less than 1% by weight) of aluminum have been made.

(3) Impact Tests. Because of its brittle nature beryllium does not lend itself to testing on the conventional impact machine, but tests were made on a more sensitive machine normally used in testing plastics. Data were erratic and did not lend themselves to interpretation.

(4) Corrosion. At the request of the Laboratory, Battelle Memorial Institute conducted some distilled-water corrosion tests on beryllium metal at 600° F (1500 psi) and 525° F (800 psi). Samples were selected from vacuum cast metal extruded at 1400, 1600, 1700 and 1800° F, and from sintered powder metal, grades QM and QRM.

Results showed that all samples, regardless of condition (as-machined, annealed, or chemically polished for removal of cold worked metal), were resistant to 525° F water. The hot-pressed beryllium was superior to the extruded metal in its resistance of 600° F water.

TABLE IX

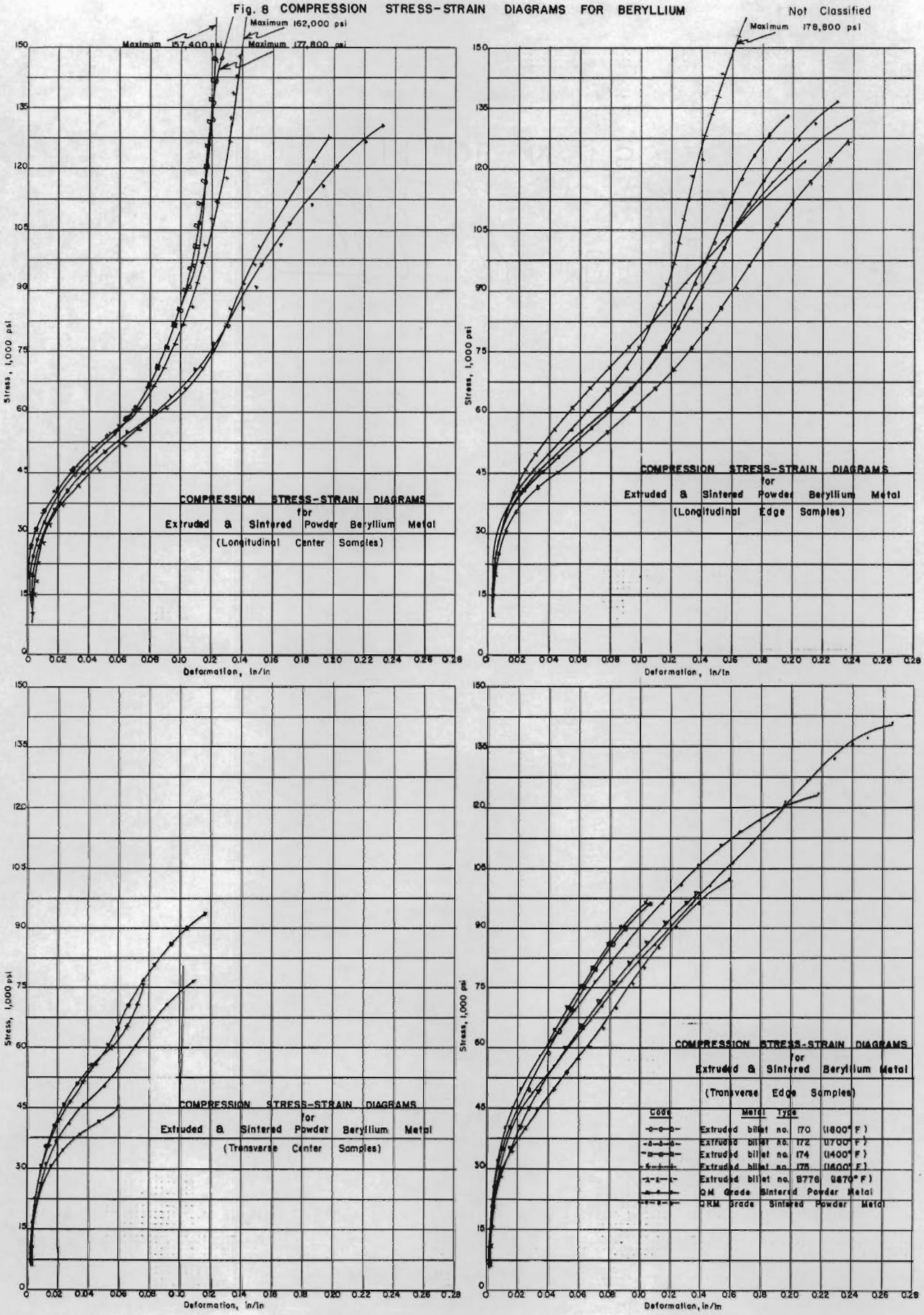
Beryllium Compression Testing Data

METAL	BILLET NUMBER	EXTRUSION TEMPERATURE	CONDITION AND REDUCTION RATIO	SPECIMEN LOCATION	LONGITUDINAL DIRECTION		TRANSVERSE DIRECTION	
					Ultimate Strength (psi)	Change in Length (percent)	Ultimate Strength (psi)	Change in Length (percent)
Vacuum Cast Extruded	174	1400	As Extruded (4.1 to 1)	Edge #1	----	----	55,000	3.6
				Edge #2	----	----	48,400	3.3
				Center #1	157,400	12.8	42,400	3.2
				Center #2	150,300	14.1	44,200	4.8
Vacuum Cast Extruded	175	1600	As Extruded (4.1 to 1)	Edge #1	178,800	19.5	86,100	7.2
				Edge #2	152,300	16.8	96,800	10.4
				Center #1	162,000	14.8	72,600	6.8
				Center #2	167,000	16.9	75,800	7.7
Vacuum Cast Extruded	172	1700	As Extruded (4.1 to 1)	Edge #1	141,100	22.8	98,400	13.0
				Edge #2	139,500	23.8	92,800	12.2
				Center #1	136,400	23.6	93,800	11.9
				Center #2	136,400	24.1	99,800	11.8
Vacuum Cast Extruded	170	1800	As Extruded (4.1 to 1)	Edge #1	133,000	19.7	96,800	9.0
				Edge #2	139,000	23.8	91,700	9.3
				Center #1	169,600	15.6	76,500	11.0
				Center #2	177,800	14.0	67,900	7.9
Vacuum Cast Extruded	B776	1870	As Extruded (6.5 to 1)	Edge #1	130,400	23.6	89,200	12.4
				Edge #2	133,900	25.3	104,500	15.8
				Center #1	131,000	19.5	---	--
				Center #2	137,100	19.1	---	--
Sintered Powder	QM 4381	--	As Sintered	#1	134,300	24.0	142,000	26.4
				#2	136,400	23.0	138,400	25.3
Sintered Powder	QRM 4405	--	As Sintered	#1	125,800	20.9	125,800	21.6
				#2	123,800	20.6	125,200	21.2

DWG. No. 7981

Not Classified

Fig. 8 COMPRESSION STRESS-STRAIN DIAGRAMS FOR BERYLLIUM



Deep Hole Drilling. Activity on the problem of drilling long, narrow holes in beryllium metal has increased as a result of the successful extrusion work at Adrian. Stock of the desired length and cross section is now available for fabrication into full-sized reflector units. Success in drilling water-cooling channels will permit greater flexibility of design and reduce cost of fabrication of the MTR reflector.

Deep-hole drilling tool manufacturers were contacted regarding an abnormal number of tool failures which occurred at the silver-soldered bit-shaft joint. It was hoped that the correspondence might lead to a new tool design more suitable for drilling beryllium than the presently used gun-barrel drilling tool. Arrangements were made with the High Standard Manufacturing Company of Hamden, Connecticut, and the Conner Tool and Cutter Company of Detroit, Michigan, to test drill some beryllium metal in their shops. All this work was observed by Mr. P. Kofmehl of the Research Shops to insure proper safety precautions in the drilling of the beryllium.

(1) Work at Conner (August 19 and 20, 1949). A series of 3/16 in. diameter holes was drilled to determine optimum conditions for deep hole drilling of extruded metal. All tests were conducted on a standard deep hole machine using the Conner-fabricated tungsten carbide (Carboloy 883) tipped tools. Speeds and feeds were varied from 800 to 1600 rpm and between 0.24 and 1.40 in. drill depth per minute, respectively. Rotation of both drill and work and of work alone were tried.

Optimum conditions for drilling with this tool were:

Work only turning
Spindle speed - 1400 rpm
Feed - 0.006 in. per spindle revolution or 0.840 in. depth per minute
Coolant - 50% paraffin and 50% sulphur-base oil
Coolant Pressure - 500 psi

Evaluation of the tests revealed that prolonged tool life, greater speeds, and heavier feeds are possible with the Conner carbide tipped tool. The amount of hole run-out or variance from the true center, was greater than that found in similar tests conducted with high speed steel tools at Oak Ridge. It is felt, however, that additional development work will result in a greater accuracy in the use of the tungsten carbide tool.

(2) Work at High Standard (August 24 and 25, 1949). A similar set of tests was conducted to determine the applicability of hardened high speed steel tools for drilling beryllium. In all test cases, the drill remained stationary

while the work revolved. Best conditions determined for use of the HSS tool are:

Spindle speed - 100 rpm

Feed - 0.00028 in. per spindle revolution or 0.280 in. depth per minute

Coolant - "Cutmax"

Coolant Pressure - 700 psi

High speed steel tools require more resharpening than do the carbide tools. One important design feature noted is that the HSS tool bit is considerably longer (2½ in. as against 5/8 in.) than the carbide bit, and this is believed to be conducive to greater accuracy in drilling.

(3) Work at ORNL. Lack of adequate deep hole drilling equipment at the Laboratory necessitated outfitting a conventional lathe with an external prime mover to power the drill, a high pressure coolant pump, and special fixtures for holding and rotating the long stock. Drill feeding is accomplished through use of the lathe carriage on which a constant speed motor and drill holding fixtures are mounted.

This improvised set-up is satisfactory except that the present lathe bed is too short and requires drill changes every 8 in. as the hole depth increases. Alterations on the lathe to increase its length to 96 in. are underway.

The majority of tool failures are a result of motor slow-down under load, which in turn greatly increases the rate of feed. This had been remedied by use of a constant speed motor.

High speed tool design with proper clearance and cutting angles are shown in Fig. 9. Conner has encountered considerable trouble in attempting to fabricate a 2½ in. length carbide tipped tool for beryllium work. Figure 10 shows how the deep hole drilling tool works in the material.

Data for drilling deep holes in beryllium with both tool and work rotating are as follows:

Spindle speed - 146 rpm (counter-clockwise)

Drill speed - 1340 rpm (clockwise)

True cutting speed of drill with respect to work - 1486 rpm

Coolant - sulphurized oil with 1-2% by volume "Oil Dag"

Coolant pressure - 22 psi

Commerical "Oil Dag" (colloidal suspension of graphite in oil) was added to sulphurized oil to enhance the lubricating property of the coolant. Figure 11 is a photograph of the new design A-Type reflector unit, fabricated from a single extrusion instead of the former three extruded pieces. Adequate water

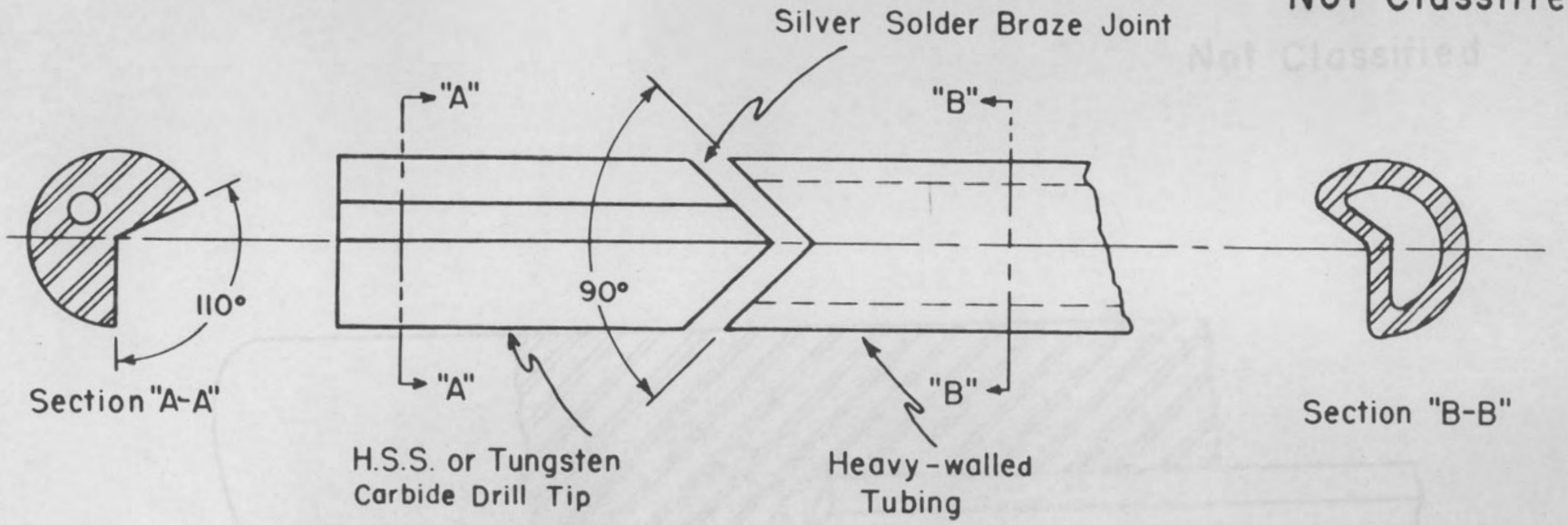
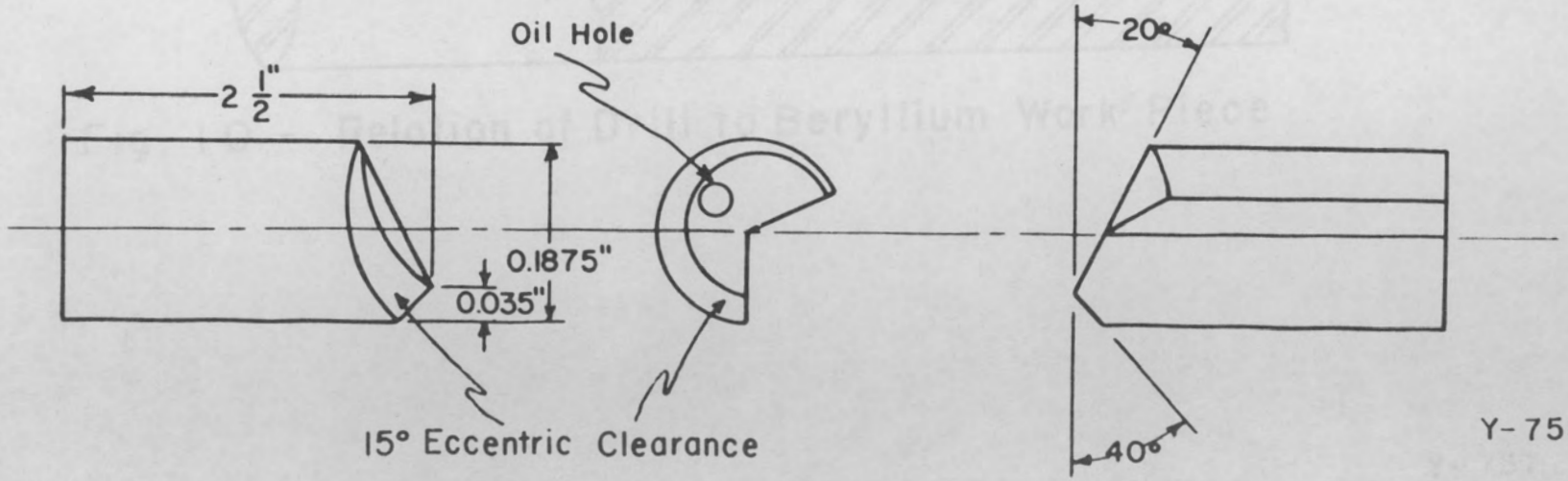


Fig. 9 - Deep Hole Drilling Tool Design



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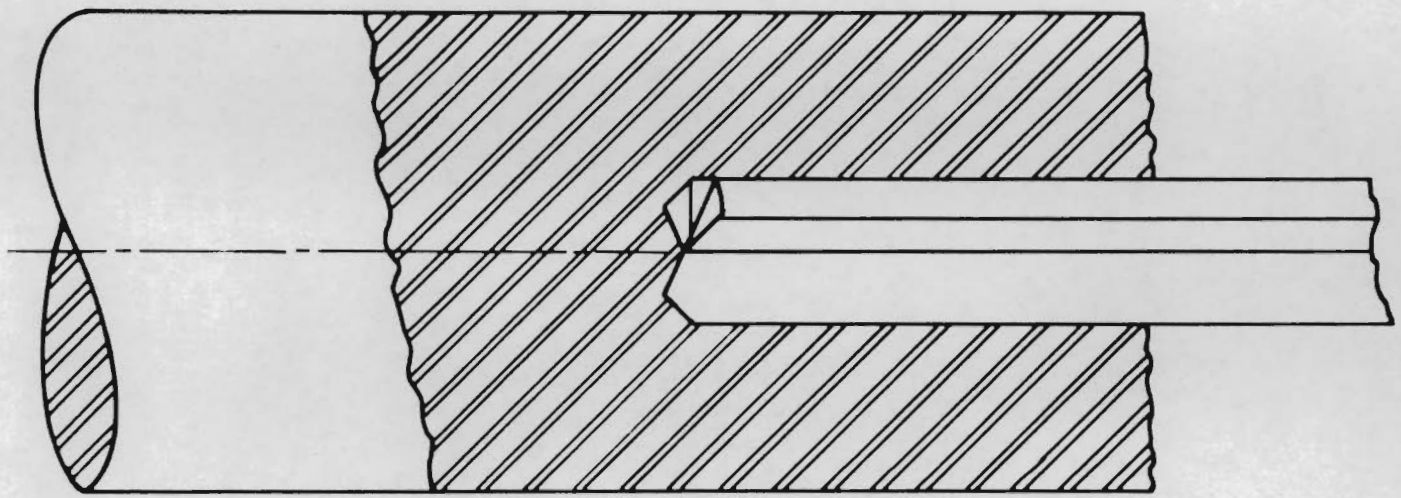


Fig. 10 - Relation of Drill to Beryllium Work Piece

Y-757

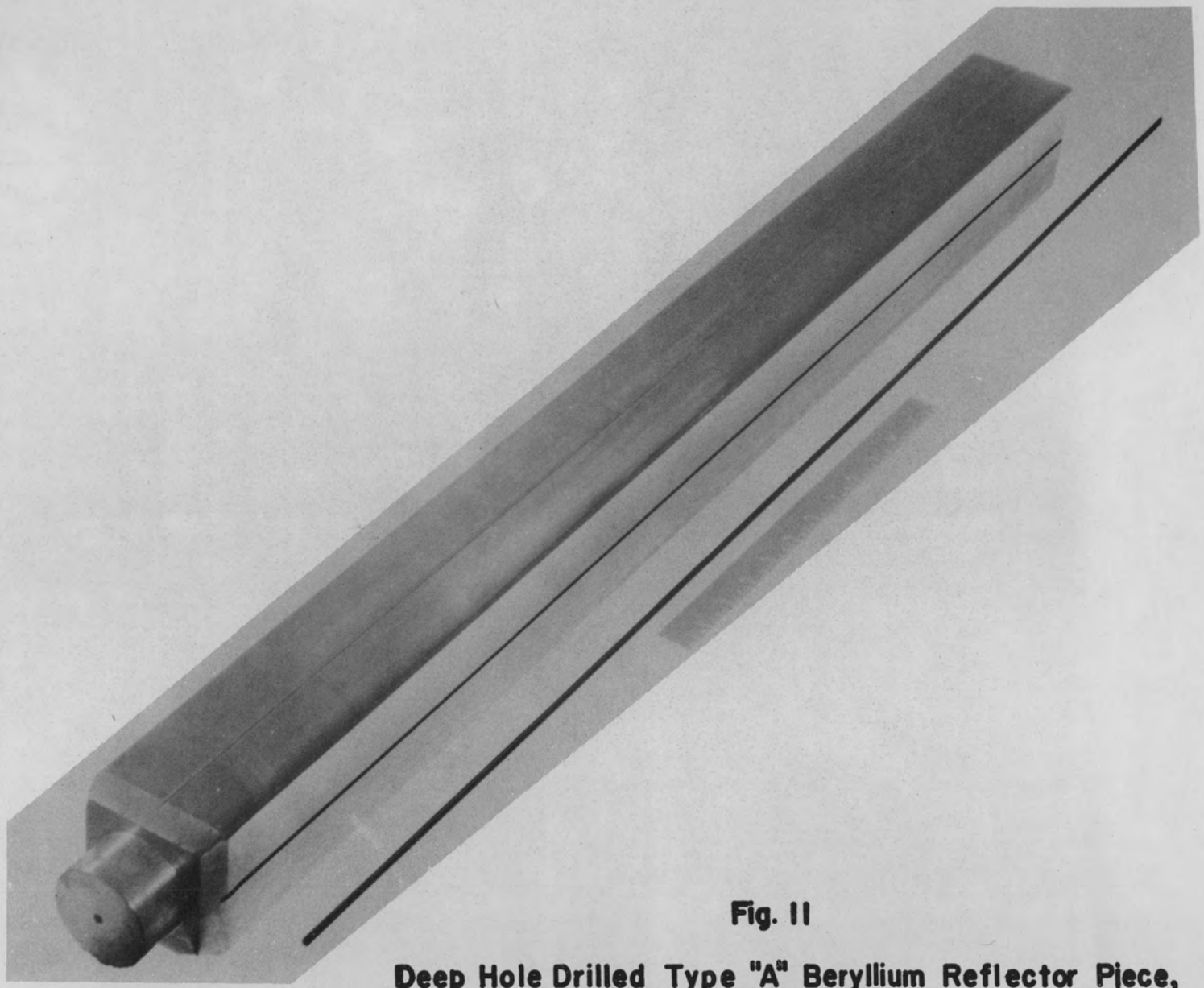


Fig. 11

Deep Hole Drilled Type "A" Beryllium Reflector Piece, With Drill Rod

DWG. NO. 7982

SECRET

cooling is provided by the 3/16 in. diameter channel running the entire length.

Beryllium-Phosphorus System. During the past quarter work has continued on the effect of phosphorus additions to beryllium. Phosphorus was introduced as the phosphide of beryllium, Be_3P_2. Alloys were prepared such that the phosphorus content should be approximately 2%, 10%, and 1.6%. Chemical analysis, however, failed to find more than a veryslight trace , even in the intended 10% alloy. X-ray spectrometry gives the peaks of Be_3P_2, but in no case are they extremely strong.

Metallographic examination so far has revealed nothing ususual about the microstructure of the as-cast material. Hardness tests are in the same range as those for beryllium, and characteristic cracking aboutthe indentation ispresent. Work is continuing with heat treating and tensile tests.

A vacuum fusion unit for the analysis of oxygen and other gases is being assembled in cooperation with the Chemistry Division. With the aid of this unit, it may be possible to determine whether or not phosphorus has any effect on the oxygen content of beryllium.

NEUTRON WINDOW FOR MATERIALS TESTING REACTOR

A survey was made of both open and project literature to determine available information on metallic bismuth. Bismuth is used in industry principally as low melting alloys, but even in this field technical information is meager. The only use of the metal itself appears to be in the form of filaments and wire. No information was found on higher temperature physical properties of the metal.

Casting appears to be the most feasible method of fabricating a bismuth window. As mentioned in ORNL 407, the expansion of the metal during freezing would be a major problem.

Three binary systems were found which formed high melting compounds on the high bismuth end of the phase diagrams. These were alloys of bismuth with calcium, magnesium, and potassium. None of these alloys is used commerically and no information was found on their properties. Since they form compounds with much higher melting points than metallic bismuth it seems likely that they would have better creep resistant qualities.

If lead is used for fabrication of the window, the major question to be answered is the creep that will be encountered. At the conditions to which it is anticipated that the window will be subjected (30 psi and 150° C) no information is availabe on the creep of lead or lead alloys. A setup has been assembled for determining this value.

Two drying ovens have multipane glass windows built into their doors. The lead specimens are suspended from the oven roof and in turn hold the weights by means of eye hooks screwed into enlarged sections of the specimens. Any deformation is determined by measuring the change in distance between two lines engraved on the lead specimens. The measuring is done with a cathetometer reading to 0.001 mm.

One test that has been running for 500 hours contains three samples of an alloy containing approximately 0.1% calcium, and one of high purity lead. The load and temperature are those expected in operation. The other test contains two specimens of high purity lead loaded to 30 psi, and three to 60 psi, and one specimen of the calcium alloy loaded to 60 psi. Data are not yet available.

CADMIUM SECTION OF MTR SHIM ROD

To obtain an aluminum clad sandwich containing cadmium, a 0.020 in. sheet of cadmium was covered with a layer of 0.010 in. zinc foil and an outer cover of aluminum. Hot rolling at 250° C was tried with reductions of 50% and greater. The zinc layer was intended to promote bonding of the aluminum to the cadmium, but destructive tests showed that the bond was not satisfactory. Further tests are planned.

ANTIMONY AND ANTIMONY-LEAD FOILS

At the request of H. W. Newson (consultant to the Physics Division) the problem of producing 0.005 in. foils of antimony or antimony-lead alloy was studied, but only a limited amount of work was done. Methods considered were:

Casting. A 0.032 in. thick section of Sb was cast satisfactorily, but it was found that metal flow into thinner molds was poor.

Rolling. Alloys of lead and antimony containing 11.3 (eutectic composition), 20, 30, 40, 50, 60, 70, 80, and 90% antimony were cast. Fractures of these showed the grain size to be progressively smaller as the lead content increased. All except the eutectic composition were very brittle. The 11.3% alloy was cold rolled from ¼ in. to 0.005 in. without difficulty.

Hot-Pressing. A brief trial was made of hot pressing Sb at 500° C, but the results were not satisfactory.

Sputtering and Electroplating. After several discussions with members of the Chemistry Division experienced in such techniques, particularly Mr. T. E. Willmarth and Dr. A. R. Brosi, it was observed that difficulty would be experienced if thicknesses of the order of several thousandths of an inch were de-

sired. This represents much greater masses of material than are usually prepared by this method. A literature survey into the electrodeposition of Sb resulted in the selection of several possible bath compositions. As an initial cathode it was believed that a sputtered film of antimony might be used, prepared upon some particularly non-adhering base material such as a plastic. A piece of lucite 2 in. square and 3/16 in. thick was submitted to Mr. Willmarth for antimony sputtering.

The resulting film was not particularly good for electro-deposition. Variations in thickness could be noted on visual examination. Several pin hole spots were found to permit the passage of light. Since this preparation was not successful, investigation will be made into the plating of antimony on thin films of other metals. It will probably be necessary, however, to provide for suitable removal of such base material. An additional problem presenting itself with this technique is the presence of hydrogen in the plated film. Dr. Newson advises that very little hydrogen contamination can be tolerated in the product requested.

THORIUM PROGRAM

USE OF ZrO_2 AS A CRUCIBLE MATERIAL

One of the initial problems in the preparation of thorium alloys is the selection of a crucible material. BeO has been used but has the disadvantage of being reduced by thorium at temperatures somewhat over the melting point of thorium. Stabilized ZrO_2 crucibles, which appeared to have been slip cast, were tested. At a temperature of 1800° C no appreciable attack occurred after a one hour holding period. However, at 1900° C ZrO_2 was attached by molten Th, and at 2000° C the crucible was almost completely reduced. This behavior is approximately the same as that obtained with BeO, with ZrO_2 probably slightly superior to BeO. A film of reacted material at the interface between a Th ingot and ZrO_2 crucible was analyzed after the Th had been repeatedly remelted and heated to 1800° C. Spectroscopic analysis showed a large amount of Th and a moderate amount of Zr. X-ray diffraction results indicated the thorium to be present as ThO_2 and the Zr in the metallic form. Analysis of the Th ingot did not show any detectable amount of Zr present.

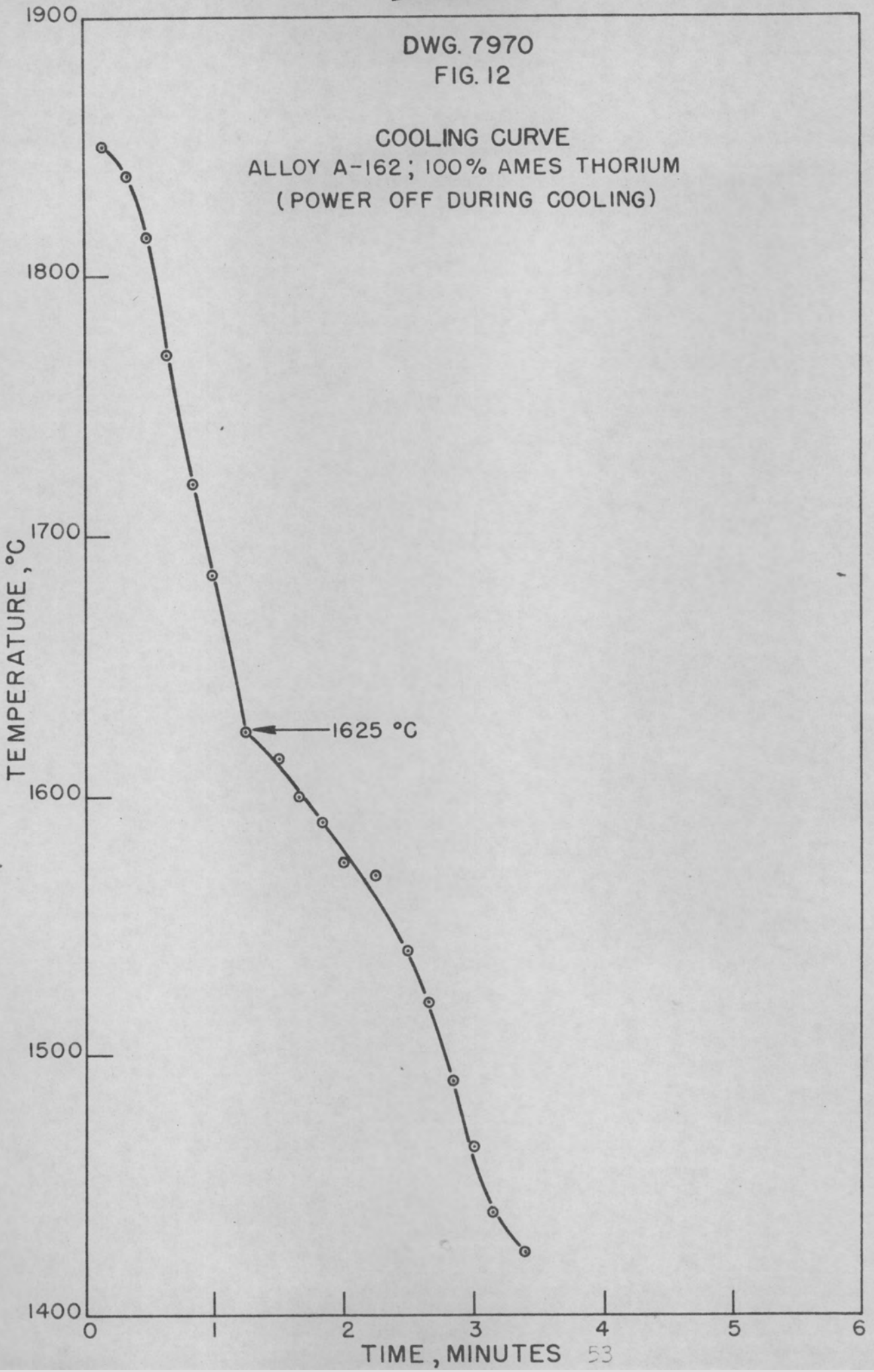
The same sort of reaction was observed when BeO crucibles were used. A large amount of powdered material was collected from the surface of an ingot melted in a BeO crucible and was found to be practically all ThO_2. No Be was detected but this was probably because of the high vapor pressure of Be at these temperatures and its ease of solution in Th.

THERMAL ANALYSIS AND ALLOYS WITH Be AND Si

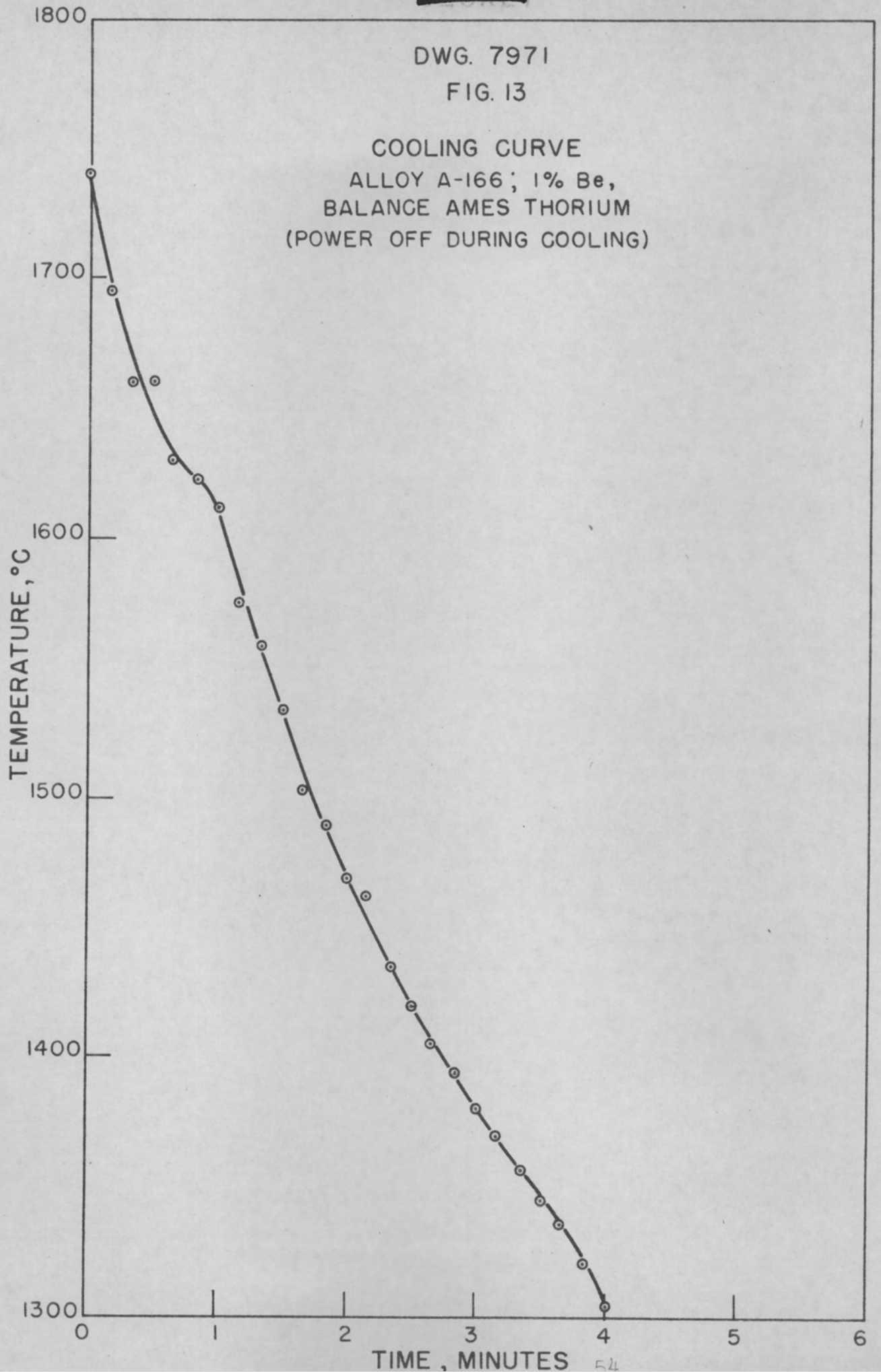
It has been reported that no thermal arrest can be observed when cooling a liquid melt of thorium, due to its very low latent heat of fusion. Preliminary experiments to check this were made by optical pyrometer readings on a thorium melt in a 3 in. diameter vacuum induction furnace.

A number of curves were obtained in which a definite change of slope was observed at about 1625° C. Representative curves are shown in Figs. 12 and 13. The cooling rate was too rapid and the heat evolved too small to obtain a good thermal arrest when the power was completely cut off during cooling. The curve in Fig. 14 shows the results obtained when the melt was cooled with a small amount of power input to the furnace. A definite change in slope, perhaps indicating the freezing point of Th, was observed at 1626° C. However, later attempts to duplicate these results gave curves which were somewhat erratic.

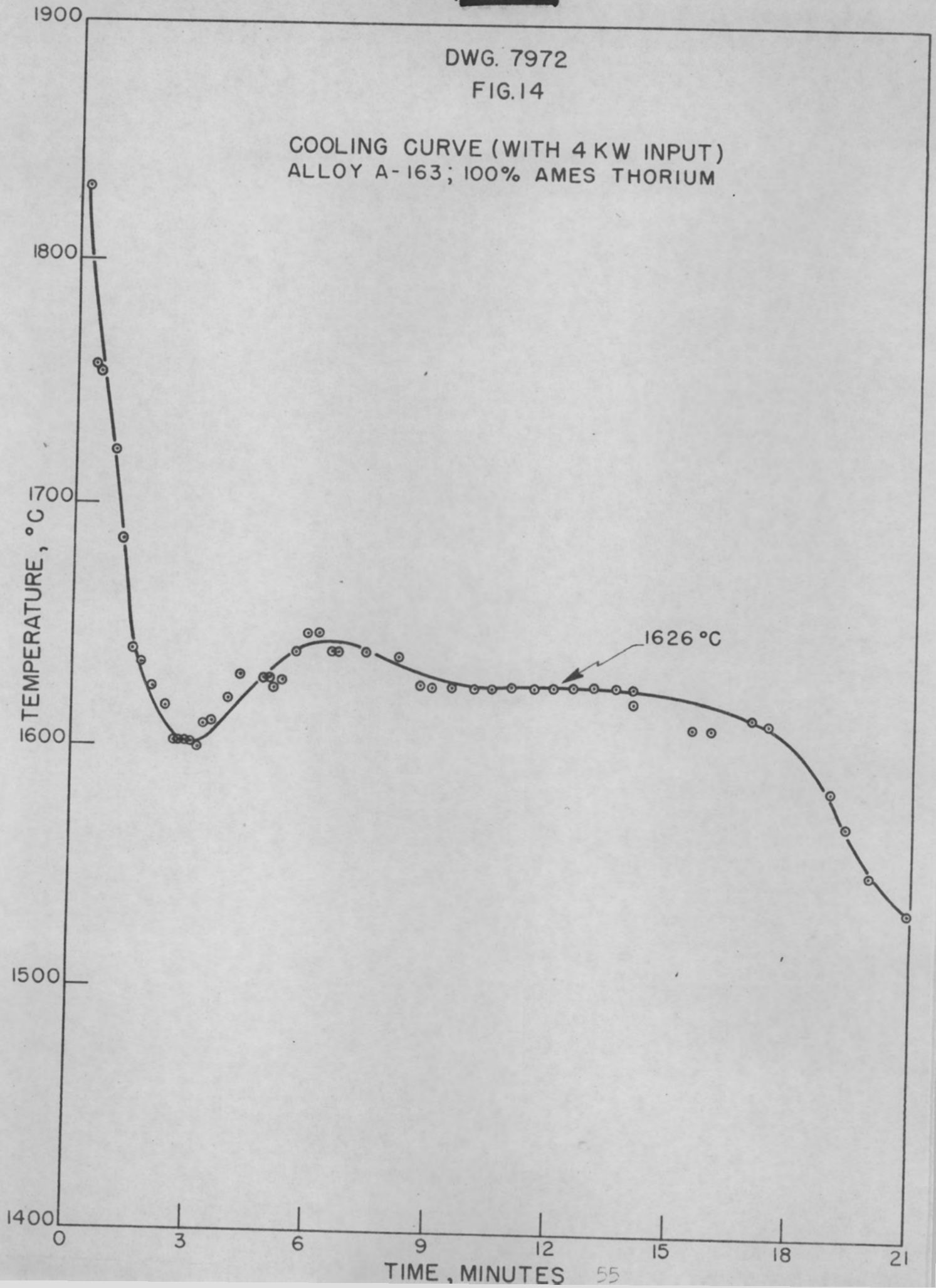
[redacted]


DWG. 7970
FIG. 12
COOLING CURVE
ALLOY A-162; 100% AMES THORIUM
(POWER OFF DURING COOLING)
1625 °C
TEMPERATURE, °C
1900
1800
1700
1600
1500
1400
0
1
2
3
4
5
6
TIME, MINUTES

[redacted]


DWG. 7971
FIG. 13
COOLING CURVE
ALLOY A-166; 1% Be,
BALANCE AMES THORIUM
(POWER OFF DURING COOLING)
TEMPERATURE, °C
1800
1700
1600
1500
1400
1300
0
1
2
3
4
5
6
TIME, MINUTES

[redacted]


DWG. 7972
FIG. 14
COOLING CURVE (WITH 4 KW INPUT)
ALLOY A-163; 100% AMES THORIUM
1626 °C
TEMPERATURE, °C
1900
1800
1700
1600
1500
1400
0
3
6
9
12
15
18
21
TIME, MINUTES

Because of the inherent inaccuracy of optical pyrometer readings when used for determination of this sort, all subsequent thermal analysis was performed with Pt + Pt-Rh thermocouples.

The first alloy systems investigated were the Th-Be and Th-Si systems. It has been reported that Be and Si produce marked reductions in the melting point of Th and that there is a good possibility that a ternary eutectic exists. The eutectic composition in the Th-Be system has been reported at 1.75% Be. Alloys of 1% Be and 2% Be were prepared and thermal analysis was carried out with Pt + Pt-Rh couples in BeO protection tubes. The heating curve, cooling curve, and inverse rate cooling curve for the 2% Be alloy are shown in Figs. 15-17. The inverse rate cooling curve for the 1% Be alloy is shown in Fig. 18. It would appear that the liquidus temperatures of the 1% and 2% Be alloys are 1175° C and 1178° C, respectively. A complete tabulation of data for the Th alloys prepared to date appears in Table X.

HEAT TREATMENT OF THORIUM

Several samples of thorium were heat treated to determine the effect of this treatment on the carbides and oxides. Three different types of thorium were used, two grades of Ames produced metal and one grade of Westinghouse metal. The heat treatments are as follows: 1 hr at 1425° C and water quenched, then reannealed at 1000° C and 800° C and water quenched; 1 hr at 1425° C, then furnace cooled to 1000° C and 800° C followed by water quenching; and then long time anneals (24-50 hrs) at 1000° C and 800° C followed by water quenching. There were some changes in hardness values, but this has not yet been correlated with the microstructures.

A sample of thorium produced by the iodide decomposition process had been received. This has been polished and examined and will be used as a reference material in studying the impurities in the other grades of thorium.

ALUMINUM CLADDING OF THORIUM

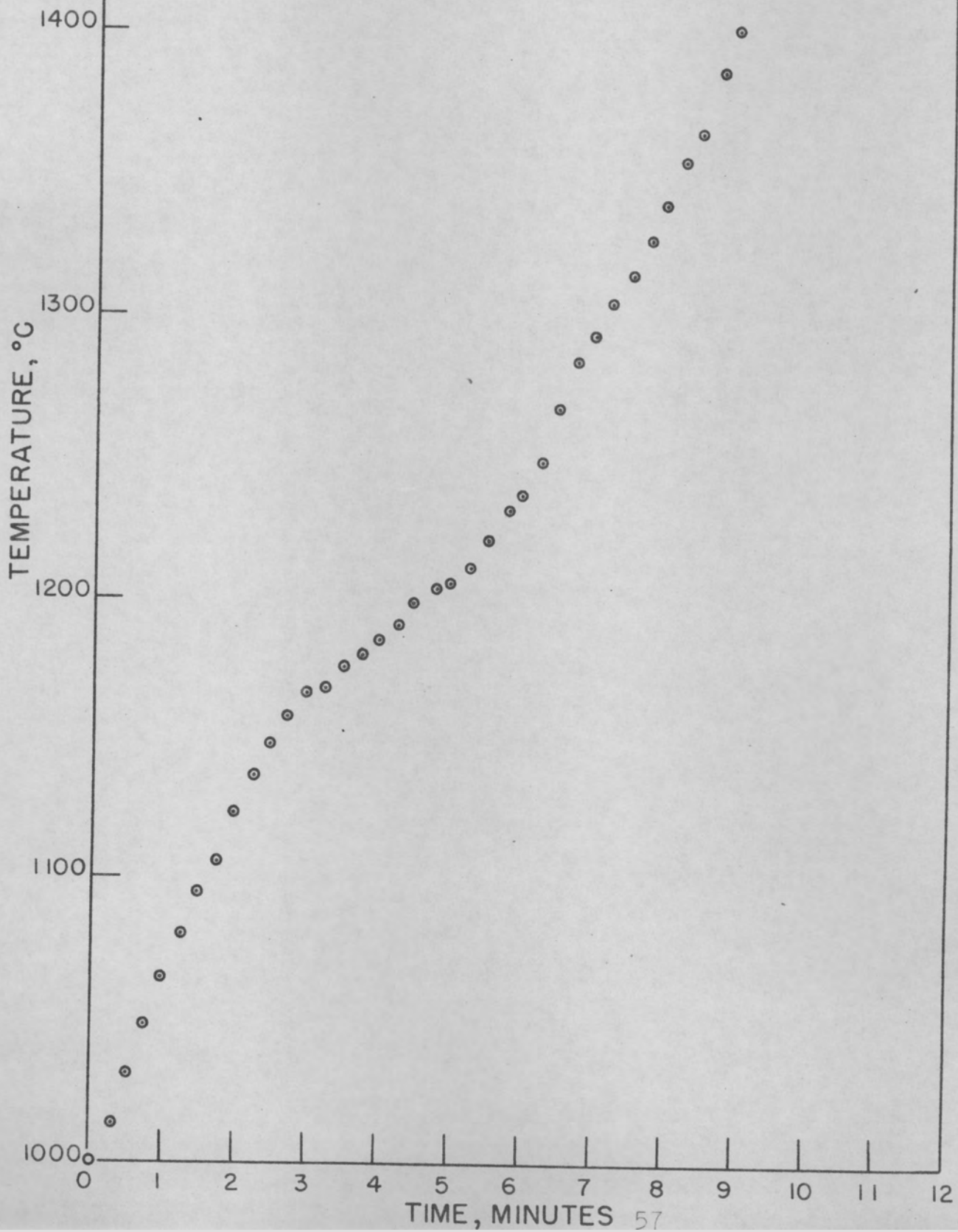
To study the effect of reduction rate in rolling on the preferential elongation of aluminum at the core ends, seven sandwiches were made up as follows:

Core - Ames thorium - 1 in. x 2½ in. x 0.290 in.
Frame - 2S aluminum - 3 in. x 6 in. x 0.270 in.
Cover - 2S aluminum - 3 in. x 13 in. x 0.120 in.

[redacted]

DWG. 7973
FIG. 15

HEATING CURVE
ALLOY A-180; 2% Be,
BALANCE AMES THORIUM



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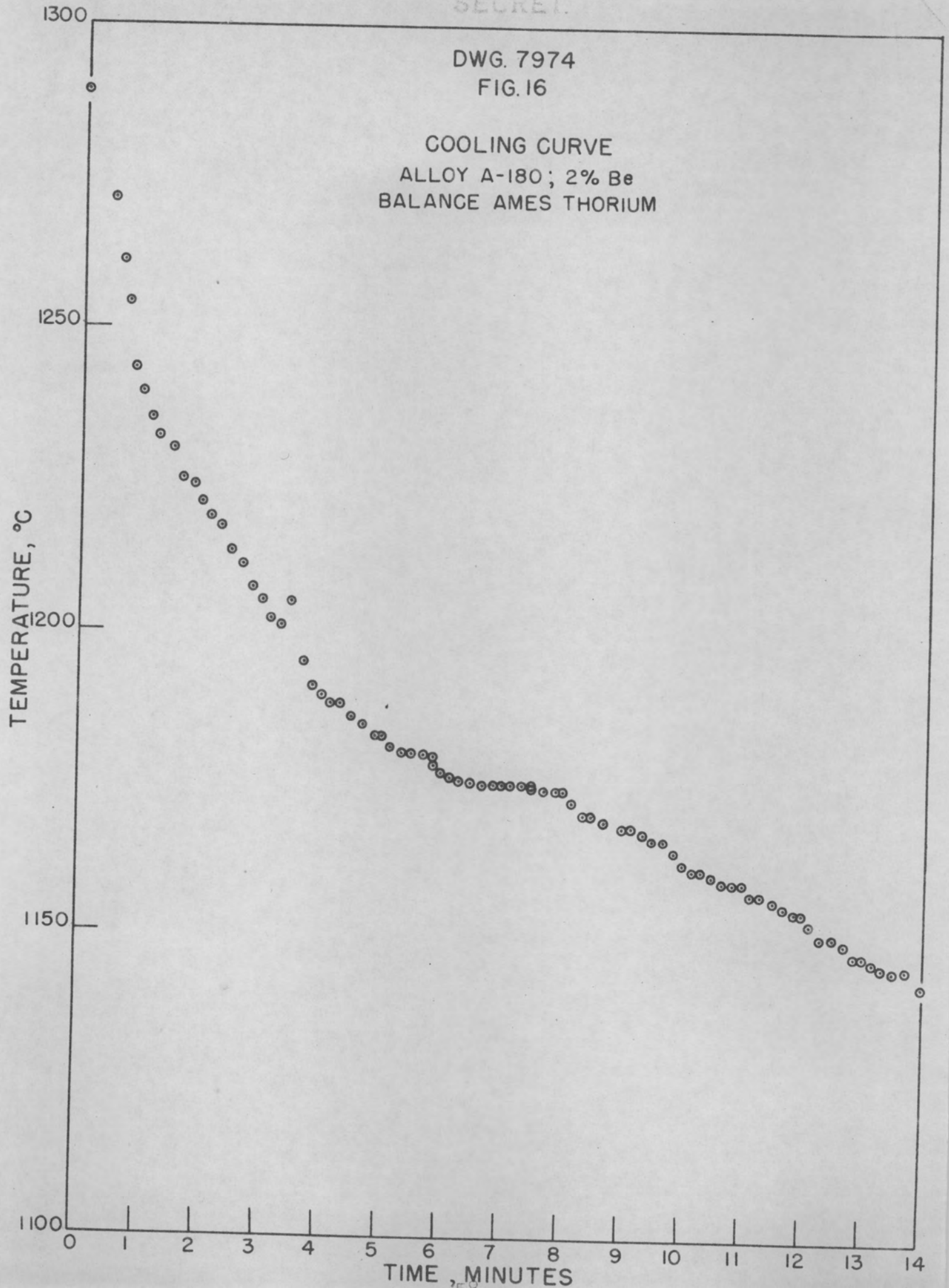
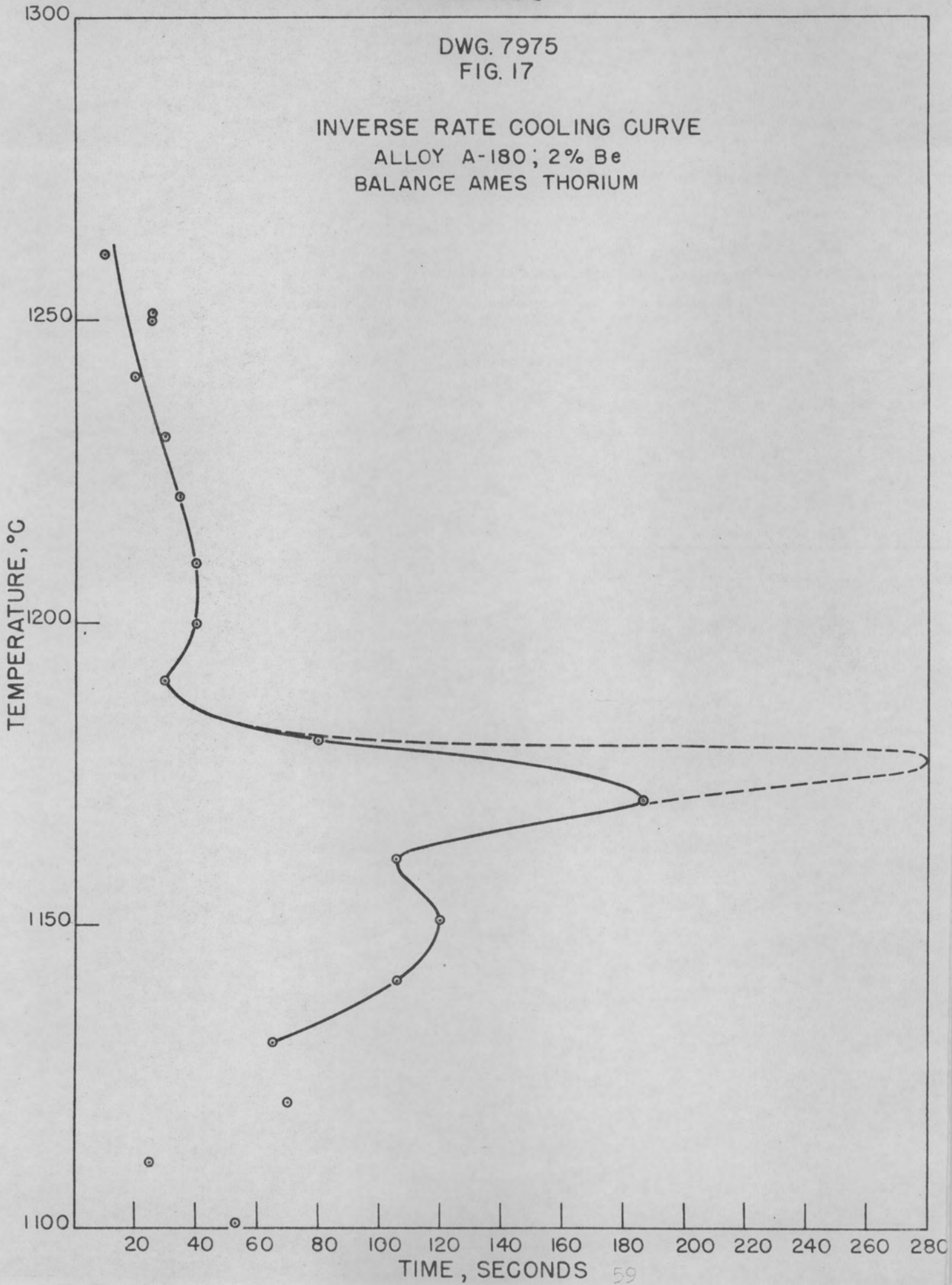

DWG. 7974
FIG. 16
COOLING CURVE
ALLOY A-180; 2% Be
BALANCE AMES THORIUM
TEMPERATURE, °C
1300
1250
1200
1150
1100
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
TIME, MINUTES

[redacted]


DWG. 7975
FIG. 17
INVERSE RATE COOLING CURVE
ALLOY A-180; 2% Be
BALANCE AMES THORIUM
TEMPERATURE, °C
1300
1250
1200
1150
1100
20
40
60
80
100
120
140
160
180
200
220
240
260
280
TIME, SECONDS

[redacted]

DWG. 7976

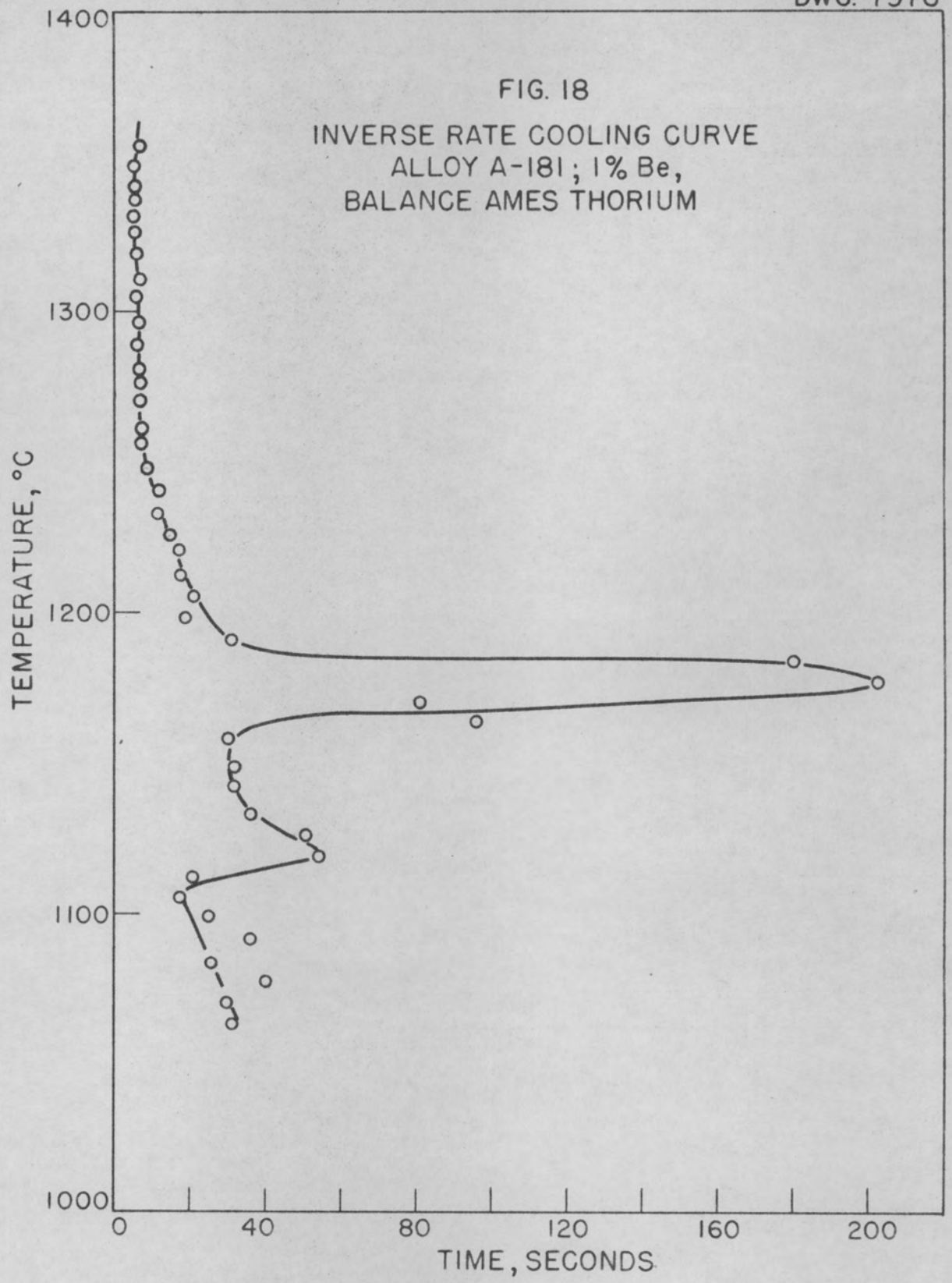


TABLE X

Record of Thorium Melts

MELT NO.	WEIGHT CHARGED (grams)	WEIGHT RECOVERY (grams)	MELTING LOSS (grams)	COMPOSITION	REMELT	TEMPERATURE (°C)		TIME (min.)		TYPE OF CRUCIBLE	CONDITION OF CRUCIBLE (initial)	CONDITION OF CRUCIBLE (final)	INSULTATING MATERIAL	MAXIMUM PRESSURE	AVERAGE PRESSURE	HEATER
						MAXIMUM	APPROXIMATE MELTING TEMPERATURE	TOTAL	at MAXIMUM TEMPERATURE							
A 112	168	168	--	Th	--	2100	--	25	5	ZrO_2	New	Erode	SiO_2	--	--	Graphite
115	117	60	57	Th	112	1800	--	25	13	"	"	Good	MgO	>500	500	"
117	194	84	110	Th	115	1810	--	40	24	"	"	Good	BeO	>500	500	"
123	234	234	--	Th	36% 117	1850	--	75	37	"	reused	Good	BeO	500	500	"
126	56	--	--	Th	--	1750	--	44	--	Al_2O_3	New	Melted	BeO	>500	500	"
127	258	251	7	Th	74% 123	1845	1815	42	26	ZrO_2	New	Good	BeO	>500	400	"
128	293	283	10	Th-1% Be	85% 127	1800	--	60	30	"	reused	Good	BeO	>500	200	"
149	134	--	--	Th	--	1880	--	20	--	"	"	"	"	"	500	"
155	288	212	76	Th	47% 149	1800	--	23	9	"	"	"	"	"	"	"
156	7	7	0	Th west.	--	1700	1700	20	2	"	New	"	"	helium	helium	"
162	146	146	0	Th	--	1900	--	80	--	"	"	"	"	500	200	"
163	146	144	2	Th	162	1922	--	48	41	"	reused	"	"	170	80	"
165	221	221	0	Th 1% Be	65% 163	1950	--	85	75	"	"	"	"	>500	150	"
166	221	200	21	Th 1% Be	165	1850	--	97	80	"	"	"	"	500	150	"
168	--	--	--	1% Si Th	--	1800	1575	50	40	"	"	"	"	>500	500	"
169	--	--	--	2% Be 1% Si	168	1425	1425	60	--	"	"	"	"	>500	500	"
170	--	--	--	2% Be 1% Si	169	1525	1285	23	--	"	"	"	"	"	"	"
177	290	290	0	1.75% Be	--	1700	1660	--	--	"	New	"	"	200	20	None
178	408	408	0	2% Be	177	1600	1300	--	--	BeO	"	"	"	100	20	"
180	658	655	3	2% Be	178	1400	1180	--	--	BeO	"	"	"	--	--	"
181	264	244	20	1% Be	76% 166	1400	1285	45	--	ZrO_2	"	"	"	150	40	"
182	212	209	3	3% Be	155	1340	1295	52	35	"	"	"	"	50	15	"
183	245	229	16	4% Be	--	1450	1350	22	--	"	repaired	"	"	25	25	"
184	297	247	50	6% Be	--	1800	1700	20	--	"	New	"	"	50	50	"
185	229	135	94	10% Be	--	1800	--	40	--	"	"	"	"	80	50	"
186	286	238	48	0.5% Be	--	1510	1435	--	5	"	"	"	"	50	40	"
188	275	273	2	0.25% Be	--	1800	1740	30	--	"	repaired	"	"	80	50	"
189	279	237	42	1.5% Be	--	--	--	30	--	"	"	"	"	25	20	"
190	137	--	--	1% Be	181	1340	1320	75	--	"	New	"	"	90	20	"
193	516	504	12	Th	--	1780	--	40	10	"	"	"	"	150	40	"
195	247	--	--	Th	--	1725	--	72	15	"	"	"	"	60	20	"

These were hot rolled on the 20 in. x 30 in. mill at 600° C to 0.200 in. thickness as follows:

2 cores - 10% reduction per pass
1 core - 20% reduction per pass
1 core - 30% reduction per pass
1 core - 40% reduction per pass
2 cores - 50% reduction per pass

After rolling, samples were cut through the end of each core and polished to determine the core shape. All showed about the same contour. Typical sections are shown in Fig. 19. These are the two samples rolled with 10% reduction per pass.

ZIRCONIUM CLADDING OF THORIUM

In a preliminary cladding test, four thorium-zirconium sandwiches were prepared as follows:

Core	Ames thorium	0.5 in. x 0.3 in. x 0.248 in.
Frame	Bureau of Mines Zirconium	4 in. x 1.5 in. x 0.244 in.
Cover	Bureau of Mines Zirconium	4 in. x 1.5 in. x 0.120 in.

Each sandwich contained three cores. Edges were sealed by Heli-Arc welding—two by merely fusing the cover and frame metal, two by using a filler rod of Bureau of Mines Zirconium. All were heated in air at 1500° F for one hour and rolled at the rate of 10% reduction per pass for total reductions of 50%, 60%, 70% and 80%. During rolling, cracks developed in the welds of the two sandwiches on which a welding rod had been used. The sandwiches will be subjected to metallographic and destructive testing to determine strength and quality of the bond.

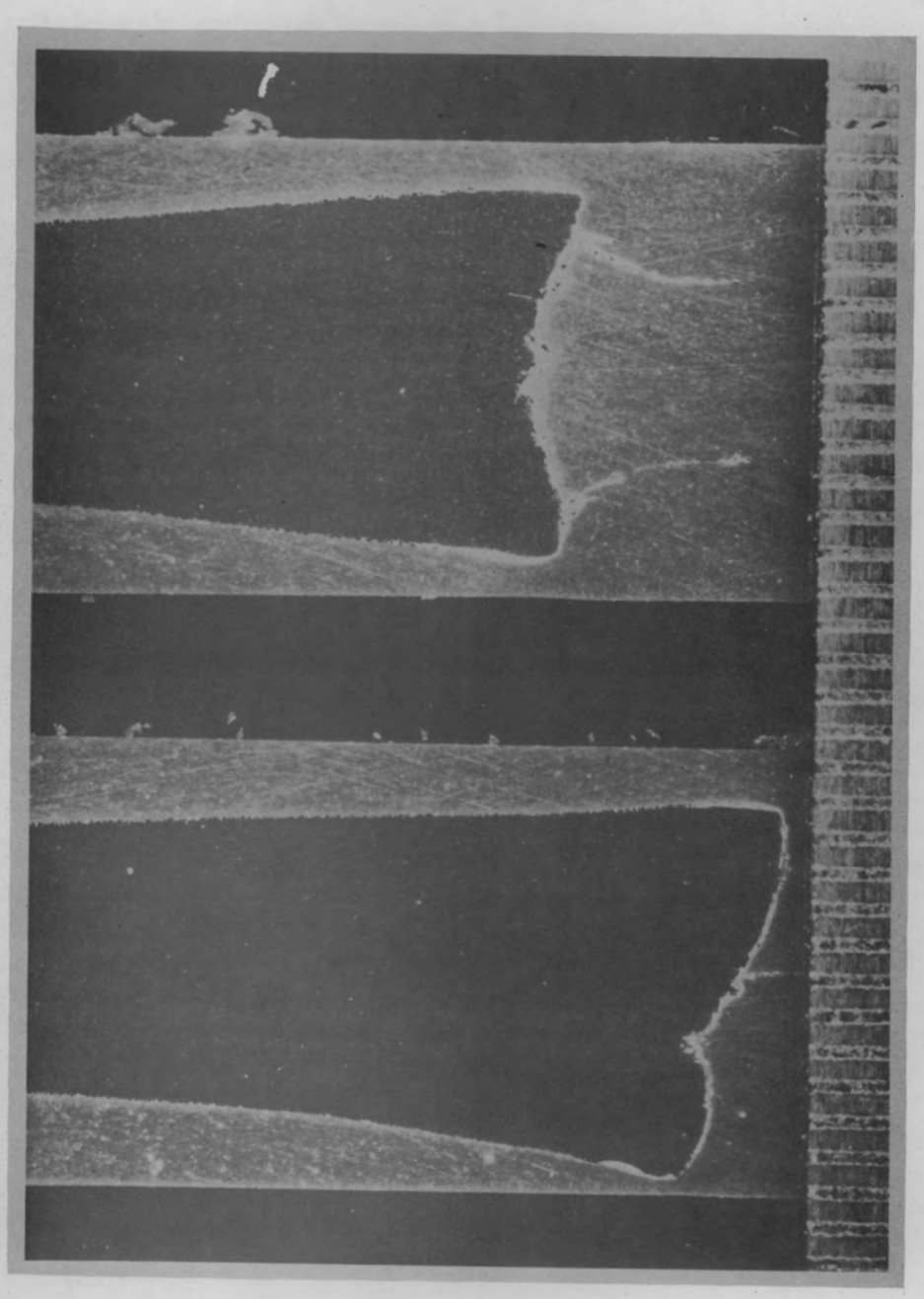


Fig. 19 - Longitudinal Vertical Cross-Section showing Ends of Aluminum-Clad Thorium 15 X Unetched.

Y-643

MECHANICAL PROPERTIES OF PURE METALS

A program for investigation of the stress-strain-time-temperature functions of metals will be presented to the Laboratory for consideration as a major research project. The program has been formulated in a paper consisting of two parts: the first outlines a working hypothesis of plastic deformation; the second specifies the kind of experiments that the hypothesis indicates are useful or necessary to advance formal understanding of deformation by rational treatment of data obtainable from tensile and creep testing.

Several exploratory tests both by creep and by tensile testing machines are now in progress. Kochendorfer's experimental results, (Zur Dynamic der Plastischen Verformung Kochendorfer, Zeits. F. Krist., 1937, Vol. 97, p. 263) obtained on naphtalene, have been checked by creep tests on copper and Orowan's conclusion (The Creep of Metals. E. Orowan, Feb. 1947) that Kochendorfer's results are applicable to metals, has been verified. Orowan states, "There exists no simple relation between strain, stress, time and temperature." Any program for the study of deformation must begin by taking this basic fact into consideration.

Tensile Testing Laboratory. Work is continuing on obtaining true stress-strain diagrams by photographic means. The DeVry camera mentioned in ORNL 407 has been received, but the shutter has not functioned properly, thus delaying its use. Tests have shown that Eastman spectrographic plates (649-GH emulsion) give a very satisfactory definition of the edges of the test pieces, but also that their use requires much higher light intensity. Films of this material are on order for use in special cases. Methods of measuring the films and recording the data have been worked out.

In the immediate future it is planned to obtain strain-stress-rate curves at room temperature on OFHC copper and on lead of 99.99% purity. A spring support for the grips of the tensile testing machine is being made. It is hoped that this will make the machine adaptable to very slow rates of testing, of the order of two hours total testing time.

Creep Laboratory. Any creep testing on project metals at elevated temperatures must be carried out either in vacuum or in purified rare gases, to prevent undue oxidation. The furnaces and extensometers of our commercial creep machines are not satisfactory for all the work planned. Redesign is in progress, and one method selected involves the use of a vacuum furnace with re-

sistance windings in conjunction with the testing apparatus. The extensometer will include a simple moveable platinum wire attached to the test specimen and provided with a reference point in such manner that the extension can be measured with a cathetometer through a window in the furnace wall. A more eleborate extensometer, employing push rods, SR-4 strain gauges, and recording equipment, is being studied.

Dr. E. Orowan and Dr. E. C. Andrade have shown that some fundamental concepts of creep can advantageously be studied on data obtained with constant stress creep machines. Such machines are not commercially available and the only available designs are those in the papers of E. C. Andrade. ["On the Viscous Flow in Metals and Allied Phenomena". Andrade. Proc. Roy. Soc. (A) 1911, vol. 84, p. 1. "The Resistivity of Polycrystalline Wires in Relation to Plastic Deformation and the Mechanism of Plastic Flow". Andrade and Chalmers. Proc. Roy. Soc. (A) 1932, vol. 138, p. 348. "A New Device for Maintaining Constant Stress in a Rod Undergoing Plastic Extension." Andrade. Proc. Phys. Soc. 1948, vol. 60, p. 304.] The designs of Andrade utilized submersible parabolic-shaped weights and are not suited to our work. Consequently, in cooperation with Mr. Warren Brand of the Instrument Section, ORNL, a device for converting a conventional creep machine into a constant load creep machine has been designed on a different principle. The weight will be moved along the lever arm in response to motion of the extensometer. Thus, as the specimen elongates and the cross-sectional area is decreased the applied load will be decreased by decreasing the lever arm ratio.

The other pieces of equipment for creep testing were devised in cooperation with the Instrument Section which now is working out their design. These are (1) a microformer extensometer that can follow the fast extension in the transient stage of creep and record this on a Brown Strip Chart Recorder as a function of time, and (2) a device for applying load at a constant rate to the creep specimen. The latter will be accomplished by dropping lead shot at a constant rate into a bucket at the end of the lever arm.

PREFERRED ORIENTATION AND ANISOTROPY IN PROPERTIES OF METALS

The problem here is to correlate quantitatively the anisotropy in mechanical properties with the type and degree of preferred orientation in uranium and other metals. Before proceeding with the determination of anisotropy in mechanical properties, it has been deemed desirable to study the influence of some of the testing variables upon the tensile stress-strain curve of uranium. The effect of strain rate as investigated with the result that no significant or consistent difference was found in the tensile stress-strain curves determined on 0.357 in. diameter specimens (1.4 in. gauge length) machined from the same 1½ in. diameter α-rolled bar and tested at constant strain rates of 0.02, 0.10, and 0.20 in./min.

It has been reported previously that the tensile stress-strain curve of annealed uranium appeared to be affected by the rate of cooling from the annealing temperature. The comparison was made between 0.357 in. diameter specimens (1.4 in. gauge length) machined from 1¼ in. diameter α-rolled bar that had been heated 8 hours at 600° C and cooled to room temperature in still air, or furnace cooled to room temperature at a rate less than 100° C/hr. This effect, if real, presumably is a result of residual microscopic stresses induced by the anisotropy of thermal expansion and retained at higher levels in the material cooled at the faster rate, assuming of course that neither rate of cooling is sufficiently rapid to induce any residual macroscopic stresses.

The experiment has been repeated employing for each cooling rate two specimens from each of two different bars. It was found that the specimens from bars cooled at different rates exhibited no consistent difference in their tensile stress-strain curves as shown in Table XI. Apparently, the difference in residual stresses resulting from difference in cooling rate is not of a type or magnitude to influence the stress-strain curve. Actual determination of the residual stresses awaits the acquisition of proper X-ray diffraction equipment.

The high X-ray absorption coefficient necessitates the use of reflection methods for determination of preferred orientation in uranium. Reflection methods employing photographic recording have been developed for this purpose (A. Smigelskas, ANL-4212 Report for July, August, and September 1948, Metallurgy Division), but these do not give the precise numerical data on density of poles of the reflecting planes for various orientations desired for a correlation of

TABLE XI

Effect of Cooling Rate from Annealing Temperature Upon Stress-Strain Relationship for α-Rolled Uranium.

BAR	SPECIMEN	COOLING RATE*	STRESS AT 2 PERCENT STRAIN
1	1	Air Cooled	51000 psi
	2	" "	55000
	3	Furnace cooled	51000
	4	" "	52000
2	5	Air Cooled	52000
	6	" "	55800
	7	Furnace cooled	55000
	8	" "	55500

*After 8 hr. anneal at 600° C.

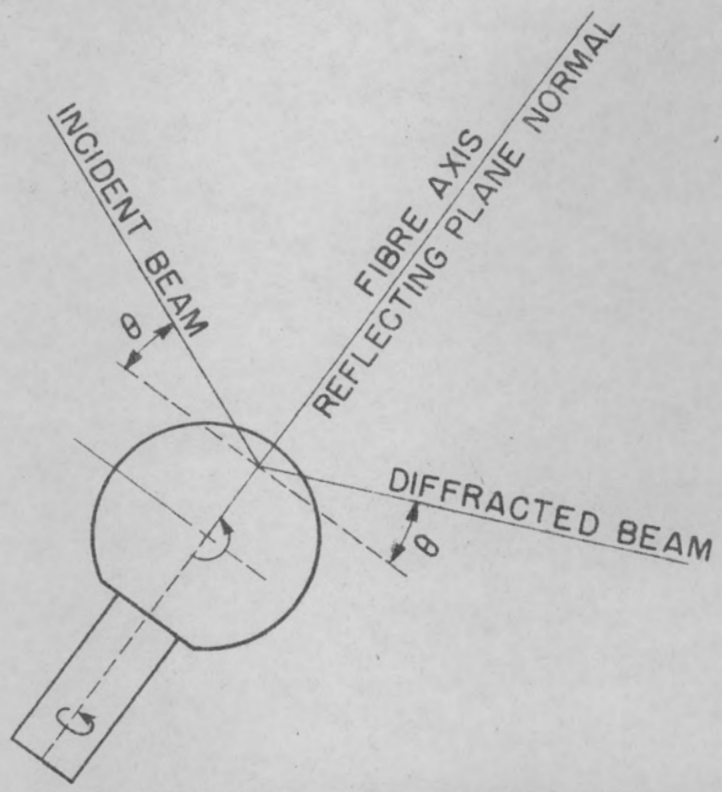
anisotropy of properties with type and degree of preferred orientation. To obtain such numerical data, reflection methods using Geiger-counter spectrometric recording have been devised. (D. Harker - HW-10476, July 15, 1948. M. Field and M. E. Merchant - Jnl. Applied Physics 20, (Aug. 1949, 741). In these methods, flat or cylindrical surfaces of the specimens to be examined are exposed at various angles of inclination to the incident X-ray beam--a technique by which investigation of the entire 90° range of azimuthal and radial angles required to develop the complete pole figure, is difficult if not impossible. Furthermore, to obtain accurate reflection intensities to relate to density of poles of the reflecting planes, requires correction for variations in focusing, in area of surface irradiated, and in relative distances traveled by incident and diffracted beam as the surface is tilted toward or away from the incident beam.

To eliminate these difficulties, a method has been devised whereby a spherical surface is exposed to the incident beam. With such an arrangement, illustrated in Fig. 20, the required range of azimuthal and radial angles can be investigated with one specimen with no change in focusing or diffracting volume; consequently, the relative intensities measured should be related directly to the relative densities of poles of the reflecting plane. From preliminary investigation this method appears to be quite promising. It is readily applicable to shapes from which a sphere can be easily machined. There is a possibility that even for thin material, a spherical specimen can be machined from a compact made of many sections of identical orientation.

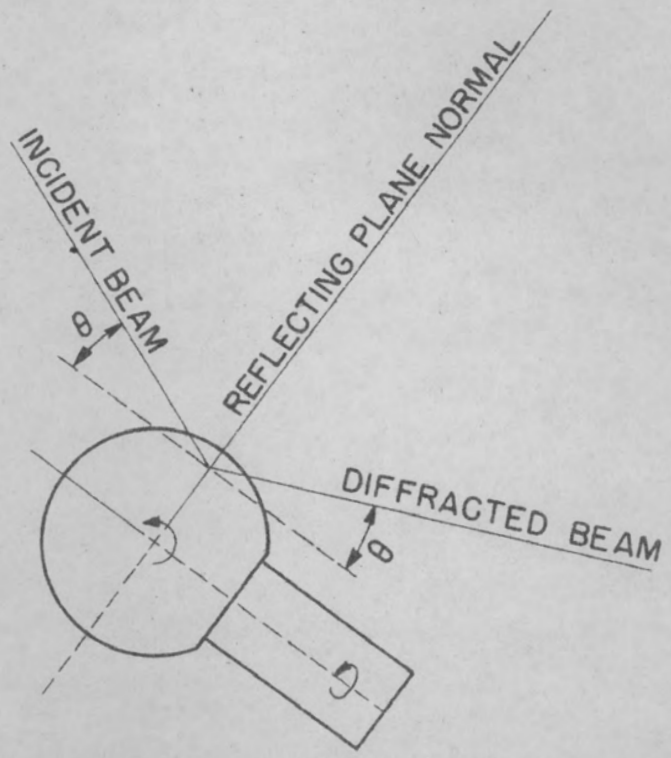
[redacted]

DWG. 7977
FIG. 20

ARRANGEMENT FOR DETERMINATION OF PREFERRED ORIENTATION IN URANIUM USING GEIGER-COUNTER SPECTROMETER



FIBRE AXIS 0° TO NORMAL TO REFLECTING PLANE



FIBRE AXIS 90° TO NORMAL TO REFLECTING PLANE

PERSONNEL

Table XII lists the members of the Metallurgy Division whose work is presented in this report. It includes the organization as of October 31, 1949.

TABLE XII

Metallurgy Division Organization

J. H. Frye, Jr. - *Director*		Roberta Hudson, *Secretary*
L. E. Banker - *Administrative Assistant*		Ruby Bullard, *Secretary* Helen Carr, *Secretary* Doris Jacox, *Stenographer* Dolores Poe, *Librarian*
A. G. H. Anderson	*Fundamental Beryllium and Thorium Research.*	
W. H. Bridges	*Purification of Beryllium*	R. T. Clem W. C. Weaver
J. C. Wilson	*Pure Metals.*	
D. S. Billington	*Radiation Effects*	
G. T. Murray	*Radiation Effects*	
B. S. Borie, Jr.	*X-ray Diffraction*	
E. J. Boyle	*Clinton and Hanford Slug Problems, Thorium Alloy Development*	
J. H. Erwin	*Hanford Slug Problem*	G. D. Goldston J. D. Hutchins D. E. Rosson J. P. Turner

D. E. Hamby	*Thorium Alloy Development Melting and Casting*	E. E. Layne
W. D. Manly	*Physical Metallurgy of Thorium and its Alloys, Hanford Slug Problem*	
R. O. Williams	*ORNL Slug Problem and Thorium Alloy Development*	C. L. Smith
R. J. Gray	*Metallography*	E. R. Boyd E. P. Griggs B. C. Leslie
R. S. Crouse	*Metallography*	
L. K. Jetter	*Fundamental Physico-Metallurgical Research*	
G. H. Boss	*Fundamental Physico-Metallurgical Research*	
F. Kerze, Jr.	*Reactor Development*	
E. C. Miller	*Reactor Development; Liquid Metals*	
J. E. Cunningham	*Liquid Metals, Beryllium Fabrication*	T. W. Fulton
J. T. Howe	*Liquid Metals,*	
G. M. Adamson	*Beryllium Fabrication, Lead Creep*	
C. D. Smith	*Melting, Rolling, Cladding*	F. M. Blacksher C. F. Cutcher
H. J. Wallace	*Melting, Rolling, Cladding*	
F. W. Drosten	*Brazing, Fabrication*	G. E. Cooley J. N. Hix

ACTIVE CONSULTANTS

J. L. Gregg	*Metallurgical Coordinator of Beryllium for the Materials Testing Reactor*
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