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ORNL 407
PROGRESS REPORT

AEC RESEARCH AND DEVELOPMENT REPORT

METALLURGY DIVISION

QUARTERLY PROGRESS REPORT
FOR
PERIOD ENDING JULY 31, 1949

J.H. FRYE, JR., Director

EDITED BY
E.C. MILLER

LABORATORY RECORDS
1954

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ORNL 407

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Issued:

Contract No. W-7405, eng 26

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J. H. Frye, Jr., Director

Edited by:

E. C. Miller

Date Issued: OCT 14 1949

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SUMMARY

This report covers the activities of the Metallurgy Division since ORNL 69 was issued in June, 1948. During this period the program of expanding the Division's personnel and facilities has been largely completed. The new Quonset building is now occupied, and most of its equipment has been installed and placed in operation. These are described in an article which will appear in "Metal Progress" in August, 1949. The personnel complement of the Division has been increased to forty-four, forty represent new additions to the Division since ORNL 69 was issued. Metallurgical personnel and responsibilities have recently been transferred to this Division from the Engineering Materials Section of the Technical Division.

During recent months, as a result of the availability of necessary personnel and facilities, a considerable effort has been applied to older problems of immediate concern to the Laboratory and to the Commission. These include: the Hanford and ORNL (Clinton) slug problems; development work for the Materials Testing Reactor, including beryllium fabrication and the development of uranium-aluminum alloy fuel elements; metallurgical fabrication of isotopically enriched uranium for use in physical research; and a cooperative university program of graduate thesis research.

A substantial effort has also been given to the planning and initial steps in long-range investigations of additional metallurgical problems appropriate to atomic energy research. These are: a study of the fundamental physical metallurgy of thorium and its alloys, together with their fabrication; an expanded investigation of the effects of irradiation on the behavior and properties of metals and their alloys; a fundamental study of preferred orientation in uranium and other metals; active participation in the materials development phases of the Laboratory's research program.

ORNL (Clinton) Slug Problem. Laboratory studies of this problem show that interdiffusion of uranium and aluminum may cause penetration of the aluminum can at the maximum reactor slug temperature, 250° C. Of the remedial methods considered, the one which has shown most promise is a modification of the Hanford technique of metallurgically bonding the can to the slug with an aluminum-silicon brazing alloy.

Hanford Slug Problem. Thermal gradient equipment has been set up and is now being tested in an effort to reproduce by electrical resistance heating the temperature gradient and the distortion which occur in Hanford slugs under

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reactor conditions. It is not yet certain that the conditions are analogous, but if the work establishes such a correlation, the equipment will serve as a valuable tool to develop dimensionally stable states of uranium or its alloys for use as reactor slugs.

Beryllium. Experimental work, using a 2750 ton extrusion press at Adrian, Michigan, and performed under the technical direction of this Division, has established the feasibility of extruding the approximately 3-5/8 in. square beryllium sections required for reflector units in the Materials Testing Reactor. Additional investigations are in progress to establish optimum extrusion conditions. It now appears that the major factor limiting production of these large sections by extrusion will be the availability of crack-free eight inch diameter cast billets. Of the 33 billets extruded to date at Adrian, at least 29 contained casting defects, and in 26 cases the cracks were revealed by radiographic examination prior to extrusion.

The properties of large size hot pressed beryllium (QM metal) sections are being investigated.

Fundamental investigations are being made of the effects of various fluxes, slags, and alloy additions on the purification and properties of beryllium. As a preliminary to this work, techniques have been developed for the preparation of Be_3P_2 for use in a study of the beryllium-phosphorus system.

Laboratory casting of beryllium at ORNL has been facilitated by casting under a 3.7 psi helium atmosphere instead of in vacuum.

Molten glass has been shown to be an effective lubricant in small scale laboratory extrusion of beryllium.

Materials Testing Reactor. A correlation has been observed between ambient atmospheric moisture and the tendency of blisters to form during annealing after rolling of aluminum-clad uranium-aluminum alloy plates for fuel elements. As a result of this correlation, air-drying equipment is being installed on the rolling mill furnaces. Additional information has been obtained on the blistering which occurs during brazing and which results from the use of brazing flux and various cleaning methods, but the problem has not been entirely solved. Contributions have been made to the metallurgical design of various other parts of the Materials Testing Reactor.

Thorium. A brief description is given in this report of the proposed program for study of the physical metallurgy and fabrication of thorium and its alloys. This will constitute one of the major long-range efforts of the

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division. Initial experimental work has been directed toward the development of (1) suitable melting procedures, and (2) coatings to protect the metal from corrosion and thereby prevent coolant contamination in reactors.

Mechanical Properties of Pure Metals. This is a program of basic research in which an extended study of the plasticity of unalloyed metals is being made. It is hoped to accomplish this by obtaining a better understanding of testing methods and by establishing more exact data on the mechanical properties of pure metals. This information is required to permit a more effective interpretation of the effects of reactor irradiation on metals. Equipment has been designed and built for recording true stress-strain data and straining rates to be used in the study of these mechanical properties.

Graduate Research. The Division has initiated a program this summer to make its facilities for thesis research available to neighboring universities through the Oak Ridge Institute for Nuclear Studies. Two graduate students have been studying the effects of manganese on rates of growth and rates of nucleation of pearlite, as a fundamental problem in rate processes in the solid state. It is anticipated that this program will be expanded in the future and will be directed toward metallurgical research in the nuclear energy field.

Irradiation Studies. The Division plans a substantial increase in the work on irradiation effects on metals and alloys, performed in cooperation with the Solid States Group of the Physics Division. This will include studies of the effects of irradiation on engineering properties as well as on the fundamental reactions of the solid state. Particular attention will be given to the use of irradiation studies in interpreting the effects of cold working on properties of pure metals and solid solutions. Plans for this proposed study are in preparation and will be reported at a later date.

Service Fabrication. Ingots cast from depleted uranium, and from an alloy of aluminum with depleted uranium, have been rolled into foil for the Physics Division.

A number of small square wires have been prepared from enriched uranium for neutron exposure tests in the solid states work. The fabrication methods which were developed are described in some detail.

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URANIUM SLUG PROBLEMS

1. *ORNL (Clinton) Slugs - Diffusion Studies.* This project was initiated in September, 1948, in an effort to determine causes and prevention of slug failures in the ORNL air-cooled reactor. Some 51 slugs have ruptured since the reactor has been in operation. These ruptures, which result from oxidation of the uranium core when the aluminum can is penetrated by air, produce atmospheric contamination and loss of operating time.

Evaluation of the available data indicates that about two-thirds of the ruptures originate as mechanical imperfections or holes in the aluminum cans, primarily at the welds. Laboratory tests, consisting of heating slugs under various conditions at temperatures from 250° C to 550° C, have shown that penetration of the aluminum cans can occur by interdiffusion of aluminum and uranium. While no actual failures have resulted from this mechanism in laboratory tests carried out at temperatures lower than 400° C, diffusion has occurred at the lowest test temperature, 250° C (the maximum slug temperature in the reactor), and it appears probable that, at time intervals as long as the exposure life of slugs in the pile, failures could result from this cause. Following are data for the minimum times required for complete diffusion penetration of the 0.032 in. thick aluminum can on a standard Clinton slug:

TEMPERATURE, °C	MINIMUM TIME FOR COMPLETE PENETRATION, DAYS
250	504*
450	10
550	1.25

*based on 50% penetration in 127 days.

In seeking methods of preventing slug failures, major attention has been centered on the use of barrier layers between the uranium and aluminum. Oxide coatings on the aluminum and uranium have been tried, but the best solution to the problem appears to be the use of a slug in which the uranium is metallurgically bonded to the aluminum can with a eutectic alloy containing aluminum with 12% silicon. This is the standard technique for canning Hanford

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slugs. Laboratory tests on Hanford slugs and Clinton size slugs coated with aluminum-silicon alloy but not bonded to the can, have shown that the interdiffusion of aluminum and uranium is greatly retarded by the aluminum-silicon barrier layer. Of corresponding importance to that of the barrier effect of bonded slugs is the fact that such slugs will be mechanically sounder with less likelihood of holes in the welds. Since it is felt that most slug failures result from weld defects, this factor assumes major importance. A quantity of Clinton size bonded slugs has been ordered from Hanford. They will be subjected to laboratory heating tests, both in the absence of radiation and under conditions of actual pile exposure. It has been recommended that, after the above tests are complete and in the event the present data on the efficacy of bonded slugs are substantiated, such slugs be used, at least in part, to replace slugs discharged from the ORNL reactor. ORNL 269 covers this problem.

While major attention was fixed on the use of barrier layers to prevent the interdiffusion of aluminum and uranium, some experimental work was done on the effect of alloying the aluminum can. Aluminum alloys were prepared with additions of Cr, Se, Mg, Zr, Ni, Si, Cu, Pb, Zn, Bi, Sb, Be, Co, Ca, Mo, Sr, Cb, V, Ti, W, Th, Te, and Mn to high purity metal. Also tested were the commercial alloys 3S, 4S, 14S, 24S, 52S, 61S, and 75S. Diffusion couples of these alloys with uranium were heated in vacuum while mechanically loaded, at temperatures up to 550° C. The extent of diffusion was determined by microscopic examination of the interfaces.

While the data obtained to date have been erratic, there are indications that silicon additions retard diffusion and that magnesium accelerates it. X-ray diffraction examinations of the diffusion layers from the unalloyed aluminum, aluminum-12% silicon, and aluminum-3% magnesium alloys are being made at present. The layer adjacent to the unalloyed aluminum has been identified as UAl_3. The layers adjacent to the silicon and magnesium alloys have not yet been identified.

2. *Hanford Slugs - Thermal Gradient Studies.* The problem of distortion in Hanford slugs is being investigated by means of equipment (to be referred to as "thermal gradient equipment") in which temperature gradients, analogous to those occurring in slugs in the Hanford reactors, are produced by means of electrical resistance heating. The principal objective is to determine whether the distortion which occurs in Hanford slugs in the reactors can be reproduced by thermally induced stresses in the absence of irradiation. If

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this can be shown to occur, the equipment can be employed as a rapid test for evaluating material which shows promise of dimensional stability.

A thermal gradient unit is shown in Fig. 1. It consists essentially of a co-axial electrical circuit in which a series of Hanford slugs, brazed end to end, serves as the resistance heating element. The line of slugs is held within an aluminum outer tube which serves as the other leg of the co-axial system. Cooling is accomplished by pumping demineralized water thru the annulus between the slug line and aluminum outer tube. Heat is removed from the cooling water in a film type heat exchanger. One end of the slug line is connected to a bellows arrangement which is a continuation of the aluminum outer tube, so that the slug line is free to expand and contract during thermal cycling. Flexible copper cable provides electrical contact between the slug line and outer tube. End loads from zero to 6000 pounds can be applied to the line by means of a small gas-oil accumulator system. The circuit is supplied by a 375 KVA variable primary 440/20-5 volt transformer. Controlled input to the unit is accomplished by use of ignitron tubes which are controlled by an air motor positioned selsyn motor.

The following controls are available:

(a) Power input can be varied according to a desired schedule by means of program controllers.

(b) Water velocity may be varied from zero to about 50 ft/sec.

(c) Cooling water temperature can be varied from approximately 40 to 85° C inlet temperature, under full load.

(d) End loading can be adjusted from 500 to 6000 lb.

Preliminary testing of the thermal gradient units, started in November, 1948, indicated that, in addition to expected mechanical difficulties, a serious electrical difficulty existed. It was found that much higher currents than those planned for were needed to develop a temperature gradient equivalent to that of slugs in the reactor. It appeared that these currents could be attained only by operating the transformers and other parts of the electrical system at about 200% of rated capacity. In order to avoid costly alteration of the system, it was decided to reduce the aluminum jacket thickness on the slugs by use of a contour turning tool. It was found that the removal of 0.010 in. of aluminum permitted a slug center temperature of 500° C, with the current on the secondary of the transformer within 160% full load rating.

To date, 37 gamma extruded eight inch slugs have been cycled. Because of the electrical and mechanical difficulties associated with the testing of

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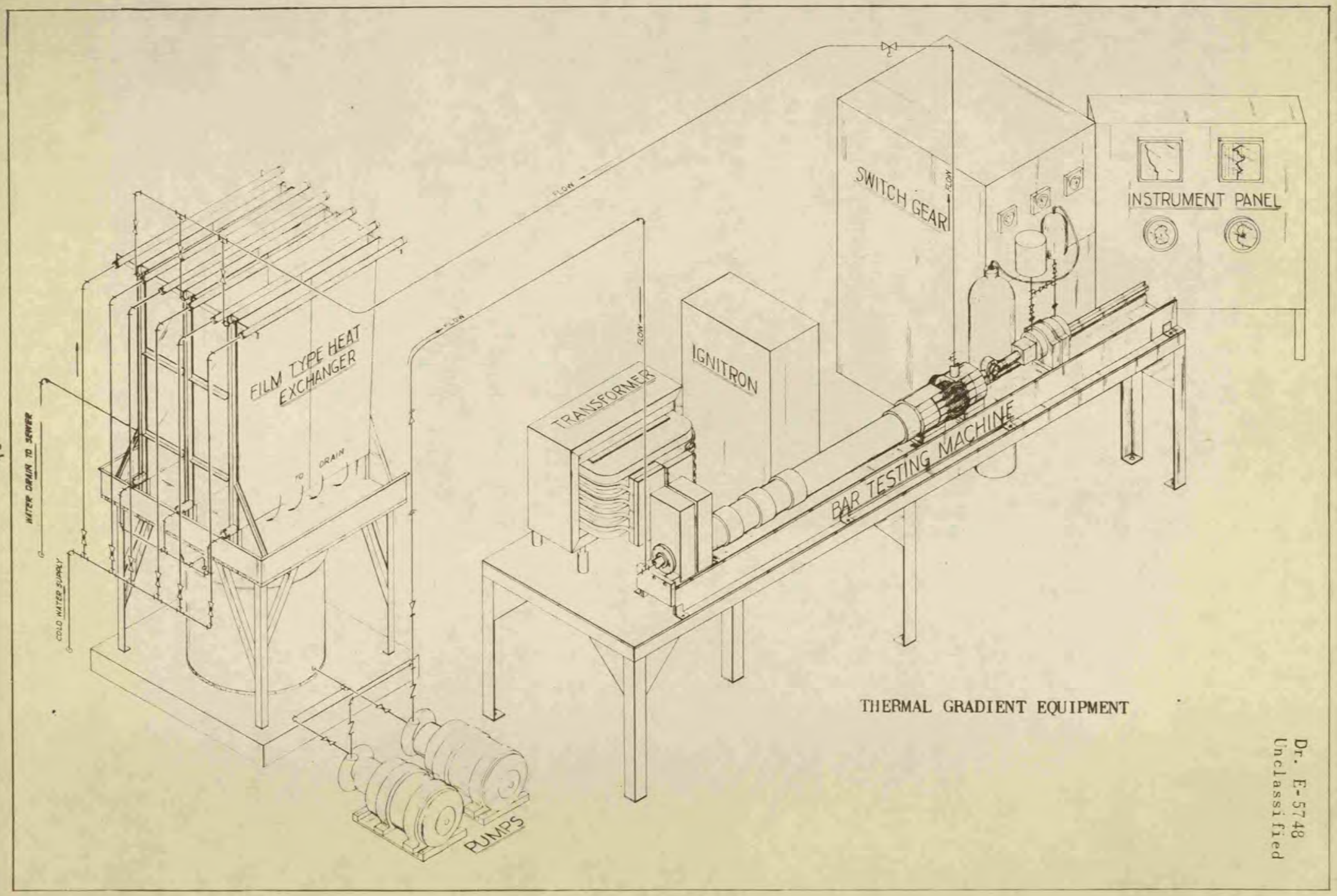

THERMAL GRADIENT EQUIPMENT

Dr. E-5748
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the first 12 slugs, the data for these are of doubtful quality. The results of cycling tests on the 15 remaining slugs are listed in Table I.

The procedure in testing slugs is as follows:

(a) Appropriate male and female joints are machined in the ends of the aluminum slug cans and aluminum end connections. These are coated with zinc by heating the slugs to 500° C, laying a 0.010 in. thick coin of rolled zinc on the end of the slug, and abrading the aluminum surface under the molten zinc with steel wool.

(b) The slugs and aluminum end connections are then brazed into a line by heating the joints in an induction coil while holding the slugs or end connections in a lathe to maintain alignment. In this operation, slug temperature is raised to 500° C and the male and female joints are rotated against one another to insure good bonding.

(c) The complete line, which consists of two eight inch slugs and two aluminum end connections, is then turned down in a lathe with a contour turning tool which removes 0.010 in. of aluminum from the slug cans along the length of the uranium cores.

(d) Length gauge marks are put on the ends of each slug, approximately at the ends of the underlying uranium core. The diameter of the slugs is measured at three 120° rotations at ½ in. intervals along the entire length. Finally, the surface contours of the slug along the entire length and at 60° intervals around the periphery are recorded by means of a feeler device hooked up to a recorder.

(e) The slugs are then cycled in the thermal gradient units described above. Slug center temperatures are measured with a thermocouple placed in a hole drilled to a depth of about 2½ in. from the end of one slug.

(f) In cycling, the lower temperature of the cycle is approximately 50 to 65° C at the center of the slug while the equipment is in steady operation. During shut-downs, the slug drops to the cooling water temperature. After completion of cycling, length changes are recorded, diameter measurements and surface contours are measured as outlined above.

The data obtained to date are listed in Table I. Because of the irregular nature of the deformation obtained in gamma extruded slugs, it is impossible to make a quantitative analysis of these data. Warping and blistering of slugs have been obtained which at least resemble similar effects occurring in the Hanford reactors. However, the test conditions used in this work, slug center temperatures of 380 to 550° C and end loads of 1460 to 4900 lb, were much more severe than those present in reactor environment—slug center temperatures of 175° C maximum and usual end loads of several hundred pounds—and it is not possible to make a direct comparison. It is planned to run tests at lower end loads and lower temperatures.

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TABLE 1

Cycling tests on gamma extruded, 8 in. Hanford slugs

ROD NUMBER	SLUG NUMBERS	SLUG NUMBERS	NUMBER OF CYCLES	LENGTH OF CYCLES	END LOAD (lbs)	CENTER TEMP. (Av. Peak °C)	TEMP. GRADIENT (Calc. °C)	CHANGE IN LENGTH	CHANGE IN AV. DIAMETER (in. x 1000)	POWER INPUT (kw/ft calc.)	COOLING H_2O Vel. ft/sec.	
9	27	32	44	2 hr	4530	415	290		27 + 0.000 32 + 0.001	105	44	Cycle - even increase in power for 1 hr and even decrease for 1 hr. Slight indication of change in contour on No. 32 slug.
9	27	32	44-65	2 hr	4500	450	310	27-0% 32-0.6%		130	48	Pulled end from No. 27 slug—Small swell in No. 32 about 1 in. from end.
10	25	31	64	1/2 hr	4900	380	260		25 + 0.011 31 + 0.002	90	42	Cycle - even increase in power for 15 min and even decrease for 15 min. Contour indicates swelling and uneveness about 2 in. long on No. 25 slug near junction of slugs.
10	25	31	64-76	1/2 hr	4800	440	310	25-4.0% 31-0.4%	25 + 0.030 31 + 0.002	100	41	No. 25 slug warped considerably and enlarged about 3 in. long on end near slug union.
11	28	30	45	2 hr	2550	460	310		30 + 0.002 28 + 0.003	120	42	Cycle - even increase in power for 1 hr and even decrease for 1 hr. No indications of change on contour or visible changes.
11	28	30	45-123	2 hr	2370	500	340		30 + 0.002 28 + 0.001	130	43	Slight indications of change or gradual swelling on contour, line broken was rewelded.
11	28	30	123-139	2 hr	2470	500	320	28-0.4% 30-0.4%	28 + 0.001 30 + 0.001	140	37	Line broken - beyond repair - very slight changes noticeable.
12	29	26	162	1/2 hr	2350	410	290		29 + 0.000 26 + 0.001	100	42	Cycle - even increase in power for 15 min and even decrease for 15 min. No changes picked up by contour or visible
12	29	26	162-302	1/2 hr	2500	400	280		29 + 0.001 26 + 0.001	100	46	Very slight change noticeable on exit end of No. 26 slug from contour.
12	29	26	302-370	1/2 hr	2500	500	320	29-0% 26-0%	29 + 0.001 26 + 0.001	130	37	Rod broke between U and Al end cap on No. 29 slug. Considerable oxidation on end of uranium.
13	36	35	63	1 hr	2200	530	380		36 + 0.006 35 + 0.007	110	38	Cycle - even increase in power for 15 min, held at max. power 30 min, and even cool in 15 min. Indications of distortion from contour of both slugs.

(Table continued on following page)

ROD NUMBER	SLUG NUMBERS	SLUG NUMBERS	NUMBER OF CYCLES	LENGTH OF CYCLES	END LOAD (lbs)	CENTER TEMP. (Av. Peak °C)	TEMP. GRADIENT (Calc. °C)	CHANGE IN LENGTH	CHANGE IN AV. DIAMETER (in. x 1000)	POWER INPUT (kw/ft calc.)	COOLING H_2O Vel. ft/sec.	
13	36	35	63-166	1 hr	2100	530	390		36 + 0.021 35 + 0.045	110	39	Both slugs had visible bumps. No. 36 slug is noticeably lopsided.
13	36	35	166-214	1 hr	2150	520	380	unable to measure	not measured	110	41	Both slugs swelled and ruptured
14	37	34	10 - 40 -	1 hr 1/2 hr	2500	550	370		34 + 0.001 37 + 0.005	140	36	Cycle - even increase in power for 5 min, held at full power for 20 min, and even decrease in 5 min. Contour indicates slight warp near center of No. 34 slug. Pulled end from No. 37 slug - replaced with No. 42 slug.
14	42	34	50-210	1/2 hr	2470	500	310	42-0.8% 34-1.0%	42 + 0.005 34 + 0.003	140	35	No indications of change to No. 42 - A slight amount of swelling is evident on No. 34 - line broke.
15	38	43	4	1/3 hr	1460	500	350	38-0 %	38 + 0.002	110	35	Cycle - even increase in power for 3-3/4 min, held at full power for 12-1/2 min, and even decrease in 3-3/4 min. Special thermocouple set 0.2 in., 0.4 in., and 0.6 in. deep in No. 43 slug - line broke at end weld.
15	38	43	4-38	1/3 hr	2350	500	340	43-0 %		100	33	No. 43 slug oxidized where couples were inserted - no change to No. 38 slug.

TABLE 1 (continued)

[redacted]

A photograph of typical deformed slugs appears in Fig. 2. It will be noted that there is a large welt at "B" on slug 35 and a very sharp ridge at "A" on slug 36. The welt at "B" is a real protuberance in the uranium as may be seen from the radiograph in Fig. 3. Slug 35 had a rupture in the aluminum can and some corrosion occurred which accounts for the ragged end seen in Fig. 3. The sharp ridge at "A" on slug 36 is almost entirely deformation in the can. This would appear to result from differential expansion and contraction of the uranium core and aluminum can.

Figure 4 is a plot of slug surface contours before testing and after cycling 66 times, as related to a radiograph taken after cycling 214 times. It will be seen that the contour of the final deformation can be found quite early in the cycling.

Some idea of the temperature gradient existing can be seen in Fig. 5. Temperature distribution across the diameter of the slug was obtained by inserting thermocouples in radial holes drilled into the slug at depths of 0.2, 0.4 and 0.6 in. The thermocouple holes were lined with thin aluminum tubing to prevent attack of the uranium by the cooling water. The center temperature was measured as usual by a thermocouple inserted in a longitudinal hole drilled into the center of the slug.

Some immediate experiments planned on this problem are:

(a) *Cycling of alpha-rolled, triple-dipped four inch Hanford slugs.* It is expected that the results of these tests will be very informative with regard to the validity of the tests on gamma extruded slugs; i.e., if the alpha rolled material shows the same sort of deformation behavior as occurs in the Hanford reactors, greater reliance can be placed on the reliability of thermal gradient testing as an indication of probable Hanford behavior than is now warranted.

(b) *Cycling of alpha-rolled, lead-dipped 4 in. Hanford slugs.* This material is known to have a highly preferred structure and should provide good quantitative data on the validity of the present tests.

(c) *Investigation of the possible correlation between bond failure at the uranium-aluminum interface and blistering in gamma extruded material.* It is thought that the local deformation occurring in this material may be accentuated in the present tests by hot spots which possibly develop at areas of bond failure between the uranium and aluminum jacket. It is proposed to frost test slugs before and after cycling, to check this point. Also, some frost test rejects slugs will be ordered from Hanford to be cycled. These slugs will have known areas of bond failure and should yield conclusive data.

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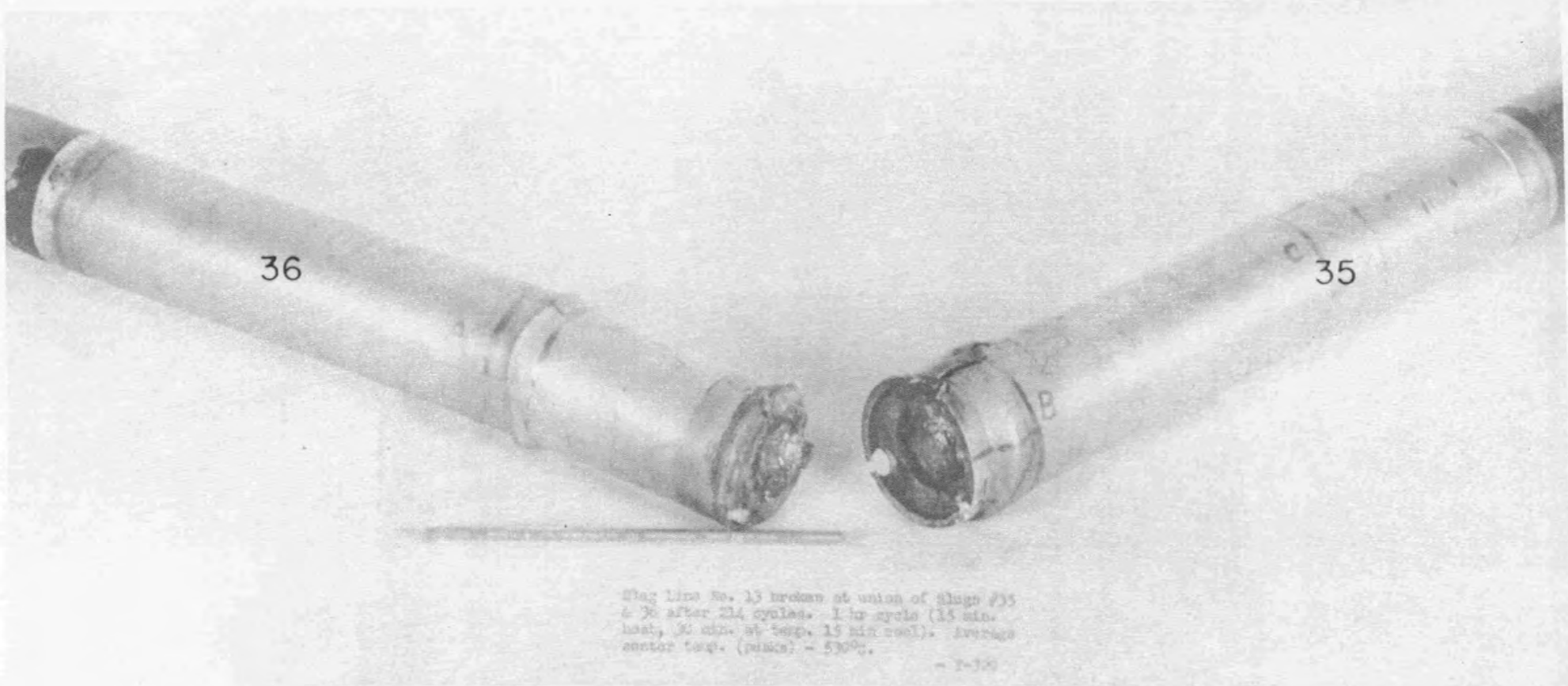
PHOTO Y-329



Slug Line No. 13 broken at union of slugs #35 & 36 after 214 cycles. 1 hr cycle (15 min. heat, 30 min. at temp. 15 min cool). Average center temp. (peaks) - 530°C.

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FIG. 2
TYPICAL DEFORMED SLUGS

[redacted]

DWG. #7643

#13 Line

Slug
No. 36

Slug
No. 35

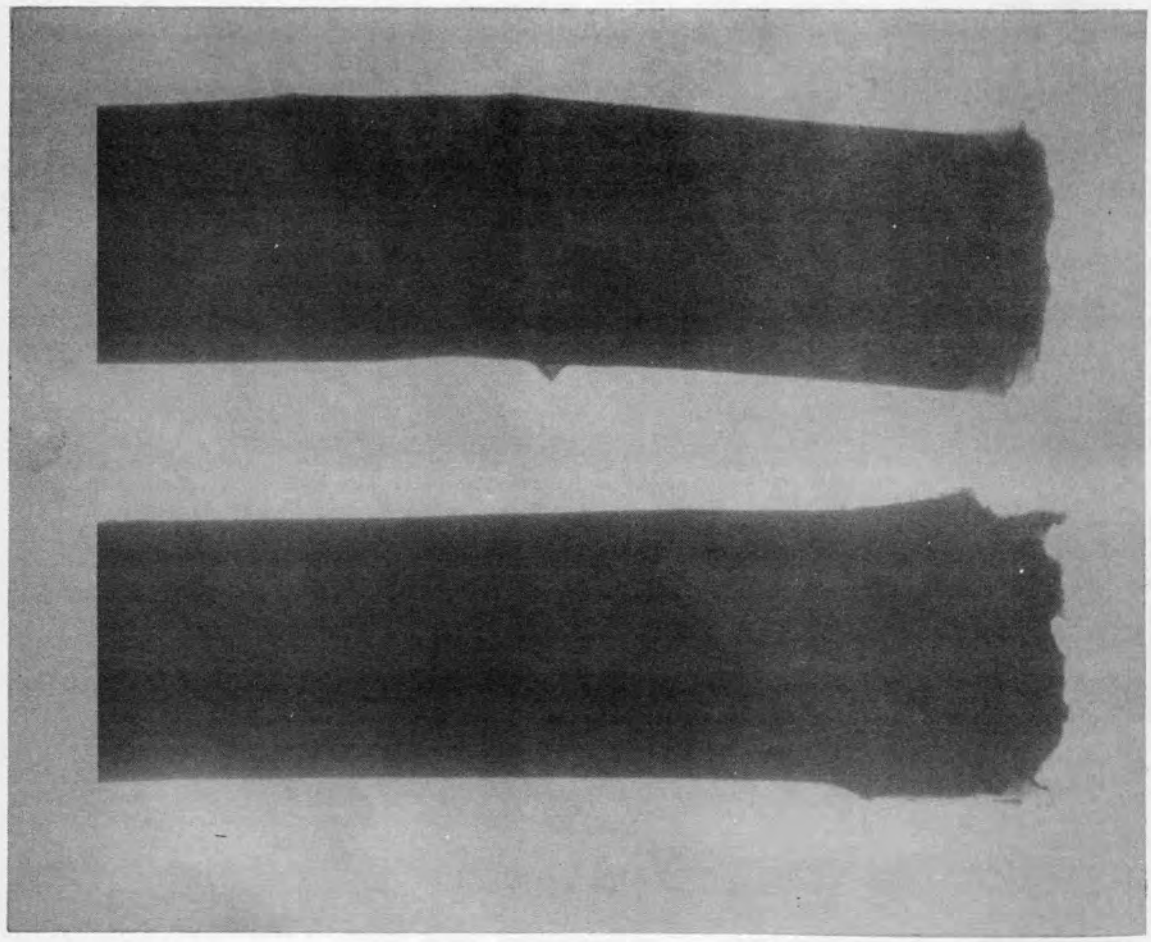

Fig. 3

Radiographs of Slugs 35 and 36
(No. 13 Slug line)

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DWG. 7662

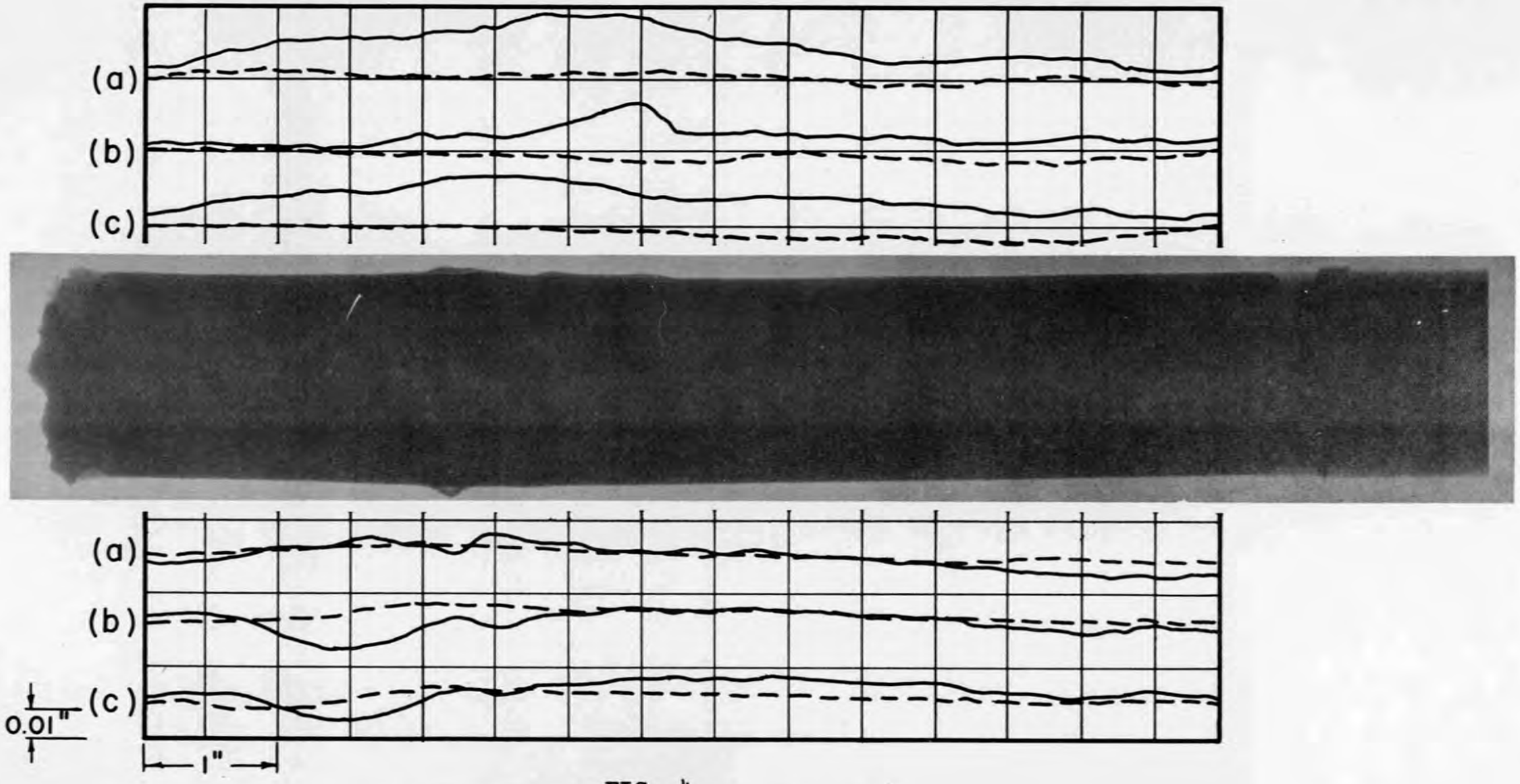
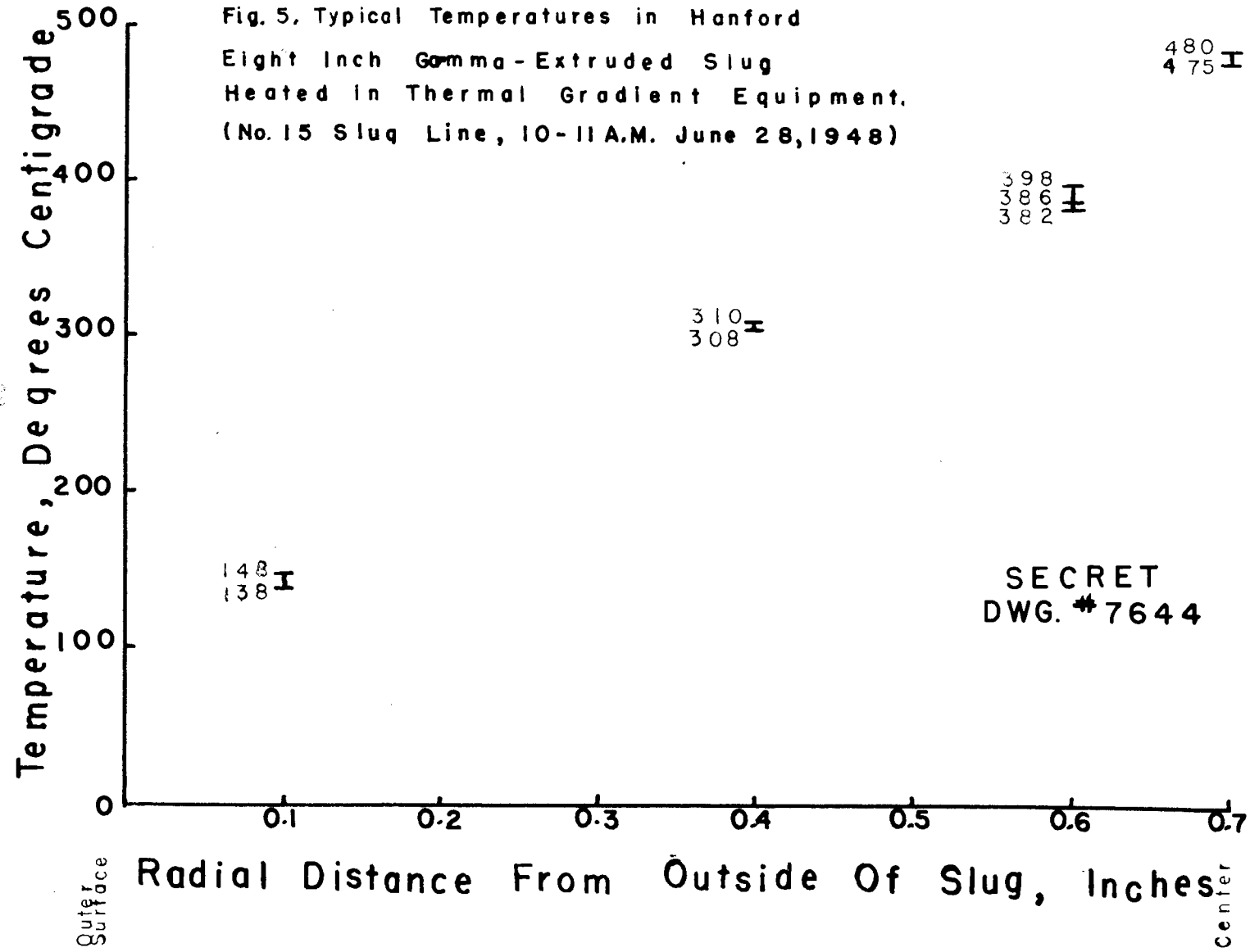


FIG. 4
X-RAY AND SURFACE PROFILES OF SLUG 36

Profiles a,b,c,were taken 60° apart. Profiles a',b',c' are mirror images of actual surfaces, and were taken 180° from a,b,c respectively. None is specifically oriented with respect to X-ray outline shown. Broken lines represent original profiles; solid lines, profiles after 66 cycles; X-ray outline, after 214 cycles. Vertical scale is 20 times horizontal scale.

Fig. 5, Typical Temperatures in Hanford Eight Inch Gamma-Extruded Slug Heated in Thermal Gradient Equipment. (No. 15 Slug Line, 10-11 A.M. June 28, 1948)



[redacted]

(d) Fabrication procedures aimed at producing fine grained, randomly oriented uranium. It is planned to extrude three inch uranium billets in the gamma range and drastically quench them, by means of water sprays, as the material leaves the die. The coarse grain size of gamma extruded material may result from slow cooling from the extrusion temperature. Six 3½ in. diameter billets have been cast for this work. Some work has been done on selection of a glass of proper fluidity which can be used as a heating bath for heating the billets for extrusion. A molten glass bath may be a convenient method of heating uranium without oxidation, and may also function as a lubricant for extrusion without jacketing.

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REACTOR COMPONENT DEVELOPMENT

Beryllium

1. *Melting and Casting - Beryllium Corporation, Reading, Pennsylvania.* The object of this work was to produce eight inch diameter beryllium billets free from cracks or porosity and having good surface quality, for extrusion into reflector elements for the Materials Testing Reactor. Some of the difficulties associated with the production of these billets and the corrective measures instituted were as follows

(a) **Shrinkage porosity.** Because of inadequate feeding of the solidifying casting, shrinkage porosity was present to a maximum of one-half the billet length. Clay hot-tops were substituted for the previously used graphite, and resulted in substantial reduction in the length of shrinkage pipe. In most castings, the shrinkage was entirely confined to the hot-top section.

(b) **Cracks.** Long radial cracks in the interior of the cast ingots have been and continue to be a major defect. It was found that close control of mold temperature and pouring temperature was essential. The optimum conditions found were to hold the top of the mold at about 600° C and the bottom at 500° C. It was necessary to supply heat to the mold by insertion of Calrod elements in the mold chamber and to insulate the inside of the chamber to meet these conditions. Rate of cooling of the solid ingot appears to be important, i.e., rapid cooling seems to promote crack formation.

There also appears to be a fair correlation between shrinkage porosity and crack formation—those billets which have deep shrink pipes show radial cracks more often than those which are adequately fed during solidification.

(c) **Poor surface quality.** Poor surfaces appear to arise from the use of dirty or heavily oxidized metal in the charge, poor vacuum, low mold temperature, and moisture in the charge or furnace refractories. Considerable improvement was obtained by attention to these items.

(d) **Carbide reaction.** The reaction of molten beryllium with the graphite molds used in casting to form beryllium carbide, can be avoided by using heavy walled molds, and by coating the bottom of the mold where the molten stream from the bottom-pour crucible impinges with a wash of beryllium oxide.

Thirty-two billets were cast during the period from March 22 to May 25, 1949. Because of uncertainty about the radiographic technique used for

[redacted]

[redacted]

examination of the billets, it is not possible to state how many billets were completely sound. Responsibility for the plant research phase of beryllium casting has been taken over in its entirety by the New York Operations Office, and studies are presently being carried out of techniques required to produce crack-free eight inch diameter billets.

The plant research phase of this problem has been reported in ORNL 261.

2. *Laboratory Melting and Casting.* Two vacuum furnaces, 12 in. in diameter and 36 in. long, have been put in operation for casting three to four inch diameter billets for experimental extrusion work. These furnaces are essentially the same as the one described by Kaufmann and Gordon ("Vacuum Melting and Casting of Beryllium", Metal Progress, Sept. 1947, pp. 387-390). The furnaces are powered by a 50 kw. 3000 cycle frequency changer. The vacuum system consists of a NRC B-6 oil diffusion booster pump backed by a 100 cfm Kinney pump.

Because of the breakage of the thin BeO stopper rods used in the original top stopper rod arrangement, a bottom stopper rod technique has been adopted. A rod of BeO 2½ in. long, is threaded onto a water cooled pipe which runs through the bottom plate of the furnace. This BeO plug fits into a hole in the bottom of the BeO crucible used for melting, and is pulled down and turned to the side when casting.

Thirty-one melts of beryllium have been made. The pressure, just prior to the introduction of helium for casting, is usually in the neighborhood of 20 microns. The billets produced have been 4½ in. diameter by 12 in. long. Alundum hot tops backed with MgO, have been used with success. Optimum casting conditions appear to be as follows:

Pouring temperature - 1400° C

Mold temperatures - 600° C at top, 500° C at bottom. These are regulated by Calrod heaters in the mold chamber.

Atmosphere - 3-7 psi helium

Ratio of raw beads to remelted metal - This factor has not been definitely fixed. Satisfactory billets have been made with 100% raw metal, 100% remelted metal, and various mixtures of both.

The major difficulty encountered to date has been the violent blowing out of the molten metal from the molds during casting. It was felt that this reaction might arise from contact of the molten metal with volatile material.

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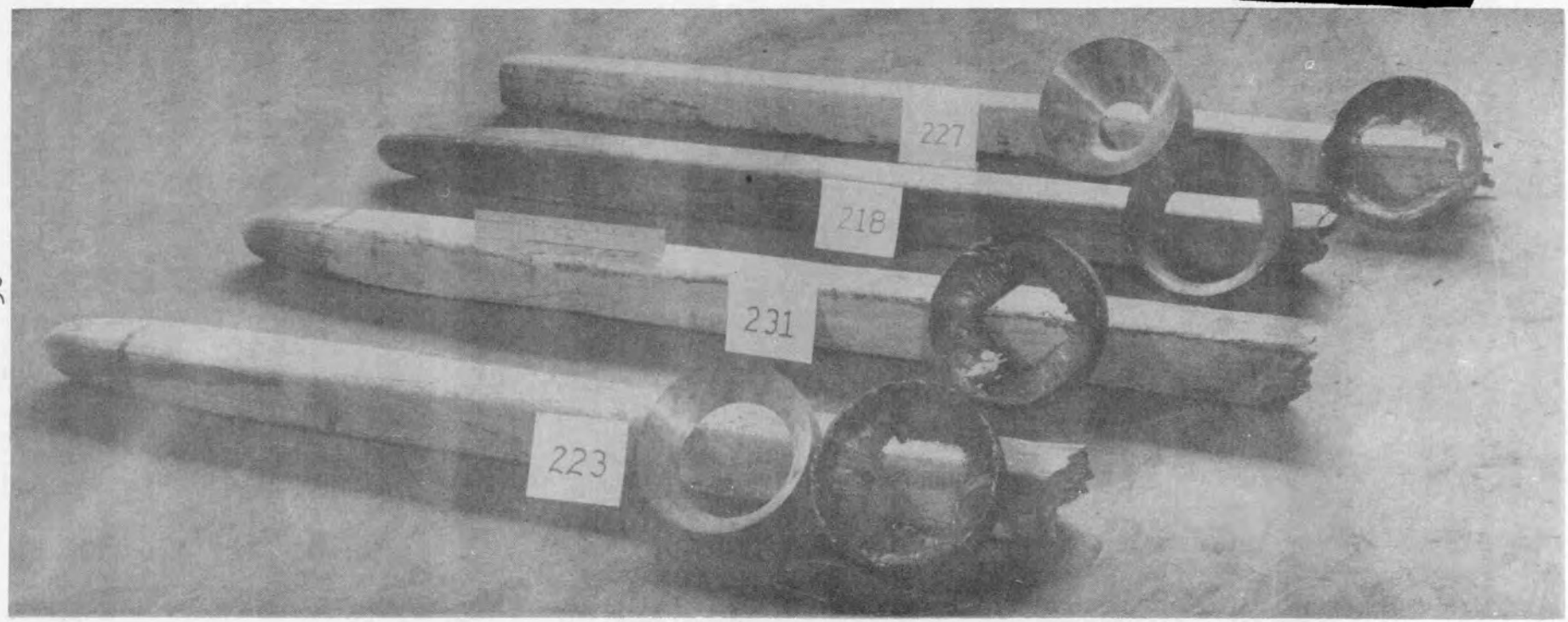

FIG. 6
BERYLLIUM EXTRUSIONS FOR MTR SECTIONS
SHOWING CONES BEFORE AND AFTER EXTRUSION

[redacted]

such as magnesium, which condensed on the mold walls. It has now been found that the blowing of the metal from the mold can be prevented by casting with a pressure of 3.7 psi of helium in the furnace, instead of casting in vacuum.

3. *Extrusion - Gerity-Michigan Corporation, Adrian, Michigan.* An active program is in progress to determine the feasibility of extruding the approximately 3½ in. × 3½ in. × 40 in. beryllium pieces desired for machining reflector sections for the Materials Testing Reactor. The work is being done on a 2750 ton extrusion press in a War Assets Administration-owned plant in Adrian, Michigan. The Gerity-Michigan Corporation is operating the equipment under the technical direction of representatives of this division. The procedures are based upon the beryllium extrusion practices developed by Kaufmann and co-workers at MIT. Some modifications have been made in this practice as a result of increased press capacity and the experience gained in extruding the large size cross section. The work performed during this period is described in a memorandum, "Extrusion of Beryllium", J. L. Gregg to J. H. Frye, Jr., July 26, 1949, ORNL - C.F. 49-7-250, from which this account has been largely condensed.

(a) **Billets.** Thirty-three 8 inch diameter beryllium billets, cast by the Beryllium Corporation at Reading, Pa., were received and extruded during this quarter. One additional billet, extrusion 32 of Table II, was a badly cracked casting into which iron wires were inserted for subsequent radiographic study of the extrusion flow pattern. Twenty-six of the 33 billets were reported to contain internal cracks on the basis of radiographic examination at Reading. Three of the remaining billets contained internal defects of a type which result typically from casting defects, but which were not detected in the standard radiographic examination. In this examination, three radiographs are made, with the X-ray beam rotated 120° for each successive exposure. This serves to show internal cracks that are approximately parallel to the beam, but some few cracks escape detection.

(b) **Jackets.** All billets were jacketed in steel cans for extrusion. About half the jackets were made at Reading and the other half at Adrian. The cans from Reading were made of 1/8 in. and ¼ in. mild steel; in either case, the same gauge of steel was used for the entire can. In the extrusion of a large cross section, the leading portion of the extrusion receives relatively little work, particularly in the center. To avoid extrusion of unworked beryllium, a short length of steel was placed in the can ahead of the billet,

[redacted]

TABLE II

Extrusion of 3¾ in. round-cornered squares at Adrian

EXTRUSION NUMBER	BILLET NUMBER	DATE EXTRUDED	TEMPERATURE °F	RATE (in./sec)	PRESSURE (10^3 lb/in.^{2})	JACKET THICK. (in.)	PLATE	CONE, INCLUDED ANGLE, DEGREE	LEAD	DIE	DIE AFTER EXTRUSION	CONDITION OF EXTRUDED SECTION (Measurements from leading end)
1	167	5/11/49	1600	1+	50.2	1/8	Cu	60	none	1002	clean	Heavy cracks 0-4 in. and 30 in. -47 in., balance good
2	168	5/11/49	1600	1+	48.0	1/8	Cu	60	none	1001	clean	Heavy cracks except 8 in.-15 in. and trailing 18 in.
3	178	5/11/49	1600	1+	50.2	1/8	Cu	60	steel	1001	clean	Excellent
4	174	5/11/49	1400	1	69.3	1/8	Cu	60	none	1001	bad	Heavy cracks except trailing 18 in.
5	176	5/11/49	1400	<0.1	92.3	1/8	Cu	60	steel	1000	bad	Trailing 16 in. cracked, otherwise good
6	175	5/24/49	1600	2	74.2	1/8	Cu	60	steel	1001	med. bad	Heavy cracks 9 in. to 39 in.
7	163	5/24/49	1600	1.3	71.1	1/4	none	60	steel	1001-2	med. bad	One crack at 37 in., otherwise good
8*	158	5/24/49	1600	<1	51.1	1/8	Cu	60	none	1001	med. bad	Cracks entire length
9	160	5/25/49	1800	1	47.5	1/8	Cu	60	none	1000	clean	O.K. except for longitudinal crack 12 in. to end
10	164	5/25/49	1800	1	38.3	1/8	Cu	60	none	1002	clean	Crack 0-18 in. and 9 in. from trailing end
11	170	5/25/49	1800	1	41.1	1/8	Cu	60	none	1001-1	slight P.U.	Cracks 0-8 in., longitudinal crack 12 in. to end
12	155	5/27/49	1700	<0.1	83.0	1/4	Cu	60	none	1002	bad	Few cracks near ends - much Be bare
13	172	5/27/49	1700	2+	51.2	1/8	none	60	steel	1001-1	bad	Heavy cracks to 13 in. from trailing end
14	156	6/21/49	1700	1/2	51.0	1/4	Cu	no cone	none	1008	slight P.U.	Rattlesnaked entire length
15	187	6/21/49	1700	1	56.5	1/8	Cu	no cone	steel	1008	bad	Rattlesnaked entire length
16	210	6/22/49	1700	1	60.0	1/4	none	no cone	new can	1008	med. bad	0-19 in. cracked, otherwise good
17	186	6/22/49	1700	1/2	49.0	1/4	none	no cone	new can	1008	med. bad	Heavy transverse cracks 5 to 10 in. apart entire length
18	166	6/23/49	1700	<0.1	54.3	A	none	90	new can	1001-3	bad	Light corner crack at 36 in., otherwise good
19	173	6/23/49	1700	1	47.5	A	none	60	new can	1001-2	slight P.U.	Heavy cracks 21 in.-29 in., otherwise good
20	214	6/23/49	1700	1	45.7	A	none	90	new can	1001-2	slight P.U.	Crack at 46 in. due to billet defect, otherwise good
21	212	6/23/49	1700	1	45.7	A	none	60	new can	1001-2	slight P.U.	Excellent
22	213	6/23/49	1700	1	49.4	A	Cu	60	new can	1001-2	slight P.U.	Rattlesnaked all over
23	215	6/23/49	1700	<1/2	54.0	A	Cu	90	new can	1001-2	slight P.U.	Crack at 14 in., otherwise good
24	220	7/12/49	1725	1	54.7	A	none	90	new can	1001-2	slight P.U.	Light corner crack at 19 in., otherwise good
25	221	7/12/49	1725	1	54.7	A	none	75	new can	1001-2	med. bad	Light corner cracks 27 in.-41 in., otherwise good
26	224	7/12/49	1725	1	58.2	A	none	75	new can	1001-2	med. bad	Cracks on 2 corners
27	223	7/13/49	1800	1/2	47.4	A	none	75	new can	1000	clean	Excellent
28	231	7/13/49	1800	1/2	46.5	A	none	75	new can	1000	clean	Light corner cracks 7 in.-17 in., otherwise good
29	218	7/13/49	1800	1/2	38.3	A	Cu	75	new can	1000	clean	Excellent
30	227	7/13/49	1800	1/2	49.2	A	none	75	new can	shear	deformed	Excellent
31	225	7/14/49	1800	1/2	40.0	A	Cu	75	new can	1000	good	Excellent
32	X	7/14/49	1800	1/2	45.5	A	Cu	75	new can	1000	good	Rattlesnaked 18 in.-52 in.
33	226	7/14/49	1800	1/4	48.3	A	none	75	new can	1000	good	Excellent
34	222	7/14/49	1800	1/2	36.5	A	none	none	new can	1008	good	Heavy cracks at 27 in. and 42 in.

*Glass used with 90° cone heated to 800° F

A - Jackets of variable thickness, but averaging about 3/16 in.

so that the steel would be extruded first. This proved effective and the subsequent cans, which were made at Adrian, contained the steel leading piece as an integral part of the can.

As ultimately developed, the bottom of the can consists of a piece of soft steel, eight inches in diameter and 2½ in. thick, on which a truncated cone is machined. The cylinder is rolled of 3/16 in. steel, welded, slipped over the cone section, and arc-welded to the cone, to form the can.

In making some of these cans, ¼ in. thick steel was used by mistake, and the billets were a little too large to go into the cans. The cans were then turned to a slightly larger inside diameter with the result that the jacket was not of uniform thickness. Apparently this did not affect the extrusion. Some of the cans were plated with approximately 0.005 in. of copper on the outside only, which appears to be a desirable practice. A chromium plate was used on one can but its use was not successful.

Soft steel rings ("cones") are tacked onto the jacket and heated with the jacket and billet. During extrusion they serve as a part of the die. The outside diameter of the cone is either 8¼ in. or 8-3/8 in., and the inside diameter, generally 5½ in. The cross section of the ring, on a longitudinal axial plane, forms two opposite right triangles. The included angle formed by the sloping faces of the ring has usually been 60°. An angle of 90° was tried, but seemed to increase the extrusion pressure. A cone with an included angle of 75° has been used and found satisfactory. The billets and the carbon cut-off, 8 in. diameter by 8 in. long, are heated in a Hevi-duty nichrome-resistance furnace, with heating elements in both the back and door as well as in the top, bottom, and sides. Excessive scaling of the steel jacket is prevented by maintaining an atmosphere of partially burned natural gas in the furnace. Billets are charged when the furnace is between 1000° and 1350° F, and remain in the furnace for at least four hours before extrusion. The furnace has a capacity of five or six billets plus carbon cut-offs.

(c) **Container and Dies.** The container used in the extrusion press has an internal diameter slightly more than 8½ in. It is heated to approximately 800° F by a gas flame, and just before each extrusion is swabbed with a graphite-oil emulsion. The die holder is heated in close proximity to the container. The dies and backers are heated in a separate furnace, normally to

[redacted]

800° F.

The dies are made of hot die steel, hardened to approximately 50 Rockwell C. Five different designs have been used, as follows:

No. 1000 - One inch thick plate. Simple bell-mouthed die with 0.2 in. land and 1.125 in. bell radius. This is an MIT design.

No. 1002 - Plate 1.500 in. thick. Bell-mouthed die with 6 in. diameter circular entrance.

No. 1008 - Large concial die with circular entrance 8½ in. diameter. Used with auxiliary cone.

Shear Die - Plate 1 in. thick, with 3-3/4 in. round-cornered square cut-out.

(d) **Extrusion.** Table II lists the data and results of extruding the thirty-four billets at Adrian. Temperatures shown are the temperatures of the billet furnace and are believed to be true temperatures of the billets, since the billets were well soaked and less than one minute was required to remove a billet from the furnace and apply ram pressure to it. The rate represents the approximate rate of travel of the ram. The pressure was read from a dial gage and is also approximate. A recorder has been made in the ORNL Instrument shop for use in future extrusions. It will record pressures on both pistons and rate, all with reference to ram position.

(e) **Results and Comments.** Temperature of the billet appears to be the most important variable. Sound sections can be produced at 1800° F much more readily than at lower temperatures. Extrusion difficulties at lower temperatures may be due to properties of steel rather than the properties of beryllium. At lower temperatures there is more tendency for the dies to gall, the steel jacket to weld to the cone, and the beryllium to weld to the jacket.

The large die without auxiliary cone has been used five times, but has not been successful. However, in the first four attempts, the die was not seated properly at the container, and flash resulted. The shear die used with a three inch inside diameter cone, produced a sound but undersized section. The die became red hot and deformed to approximate a bell-mouthed die. The available data are probably insufficient to evaluate relative merit of the dies used. It does not appear, however, that die design is a major critical factor.

Copper plating of the jacket appears to have a beneficial effect, resulting in a 20% reduction in pressure required at 1800° F, and a generally more satisfactory extrusion. The chromium plated jackets stuck to the container wall, much of the extrusion was bare of steel, and a very high pressure was

[redacted]

required to push the billet.

(f) **Straightening.** The bars are straightened immediately after extrusion, on a hydraulic straightening press near the runout table. They are pressed until the concave side just touches the flat press table. One or two bars cracked during straightening, probably because they became cold in spots.

(g) **Dejacketing.** Jackets can be stripped by clamping the bars in a vise and tearing the jackets off with steel bars, but a more satisfactory method is to cut the two diagonal corners open with an acetylene torch. The flame normally does not touch the beryllium. A ventilated cabinet can be built if required. The leading end of the beryllium tends to form a pointed insert into the steel leading piece, and the heavy steel nose is sometimes difficult to remove.

(h) **Inspection.** After dejacketing, transverse sections are sawed from the ends of the bars. The sawed surface is chemically polished, using a chromic acid-phosphoric acid solution developed by Battelle. The surface is then etched with 5% sulfuric acid revealing both the grain size and internal cracks. The samples are then fractured, as a further check on internal cracks.

Corrosion, mechanical, and annealing tests are being made on beryllium extruded at different temperatures, but the tests are not yet completed. Material extruded at 1700° to 1800° F tends to be relatively fine-grained, whereas material extruded at lower temperatures contain larger deformed grains, but conclusions regarding this must wait completion of the tests in progress.

(i) **Prospective Program.** At least one more extrusion run on perhaps a dozen billets is planned. The practice employed will probably involve the use of 1/8 in. or 3/16 in. thick steel jackets, copper plated, with a thick steel end, and with a cone having an included angle of 75° and an internal diameter of 5½ in. The billets will be heated to 1800° F and extruded at a rate of ½ in. to 1 in. per second, using a simple bell-mouthed die. They will be straightened while hot, and dejacketed by burning diagonal corners and then prying off the steel. Subsequent development work will depend upon the results of this extrusion run, the prospective availability of crack-free billets, and the progress made in the production of hot-pressed beryllium sections.

4. *Laboratory Extrusion.* Nine of the 31 ingots produced in the work described above in "Laboratory Melting and Casting", were machined to 3 in. diameter and used for tests of the feasibility of using glass as a lubricant in beryllium extrusion. The use of glass was suggested by Dr. A. B. Kinzel,

President of the Union Carbide and Carbon Research Laboratories. Significant data are contained in Table III.

It appears, that for the same temperature and reduction ratio, glass lubrication reduces extrusion pressure by about 30%. In the case of the two billets extruded without glass, the cans were torn from the extrusion, rattlesnaking was very pronounced, and the die showed severe galling and had to be reground after each run. When glass was used as a lubricant, the cans remained completely intact, no rattlesnaking occurred, and the die showed no evidence of galling. Figure 7 illustrates the differences in surface quality between the unlubricated and lubricated extrusions. It will be noted that the bar extruded without jacketing was also free of rattlesnaking.

The glass used in these tests was scrap window glass cast into one inch thick cylinders three inches in diameter. In extruding, the glass plugs were preheated to 550° C and inserted into the extrusion press container between the die and the billet.

A memorandum, "Use of Glass as a Lubricant in Beryllium Extrusion", June 13, 1949, C.F. 49-6-202, has been issued by ORNL.

5. *Hot-Pressed Beryllium.* The grade of hot-pressed beryllium generally known as QM or QRM, now produced by the Brush Beryllium Company of Cleveland, Ohio, represents a distinct improvement over the hot-pressed material formerly produced by Brush and known as Q metal. Corrosion tests, both in simulated reactor water and in an autoclave, indicate that the corrosion resistance of QM metal is on a par with extruded vacuum cast metal for part or all of the beryllium sections required for the Materials Testing Reactor. At the present time Brush cannot produce sections longer than 24 inches, but is now building equipment that will permit production of sections as long as 48 inches.

Materials, both extruded and hot-pressed, are being prepared for irradiation in the Hanford Reactor in contact with process channel cooling water. Because of the time involved in making irradiation tests, it is not certain that the results of the combined irradiation-corrosion tests will be available in time to contribute information regarding the relative merits of QM and extruded material for incorporation into the Materials Testing Reactor.

Consideration has been given to the possible effects of neutron irradiation on QM metal, particularly as it may affect the beryllium oxide content, roughly ¾ to 1½%, of the hot pressed beryllium. Siegel and Billington (TID-66) report that no significant changes were observed in electrical resistivity, density, elastic modulus, or damping in short time irradiation tests on extruded beryllium.

[redacted]

TABLE III

Beryllium extrusion using glass as a lubricant

BILLET NUMBER	EXTRUSION TEMPERATURE (°C)	REDUCTION RATIO	PRESSURE* ON BILLET (tons/in^2)	RAM SPEED	THICKNESS OF CAN PLUG**	LUBRICANT
A56	1050	17:1	72.0	--	2 in.	none
A53	1050	''	48.0	--	2 in.	glass
A54	950	''	56.0	--	2 in.	glass
A68	950	''	59.2	--	1 in.	glass
A58	950	''	83.2	--	1 in.	none
A60	870	12:1	64.0	--	1-1/2 in.	glass
A61	760	''	57.6	1/4 in./sec	1-1/2 in.	glass
A75	760	''	64.0	1/4 in./sec.	1/2 in.	glass
A59	1050	''	--		bare	glass

*Running pressure - not maximum

**All billets canned with 1/16 in. mild steel on sides; mild steel plug of thickness shown, on leading end. The steel cans were not copper plated. A59 extruded bare.

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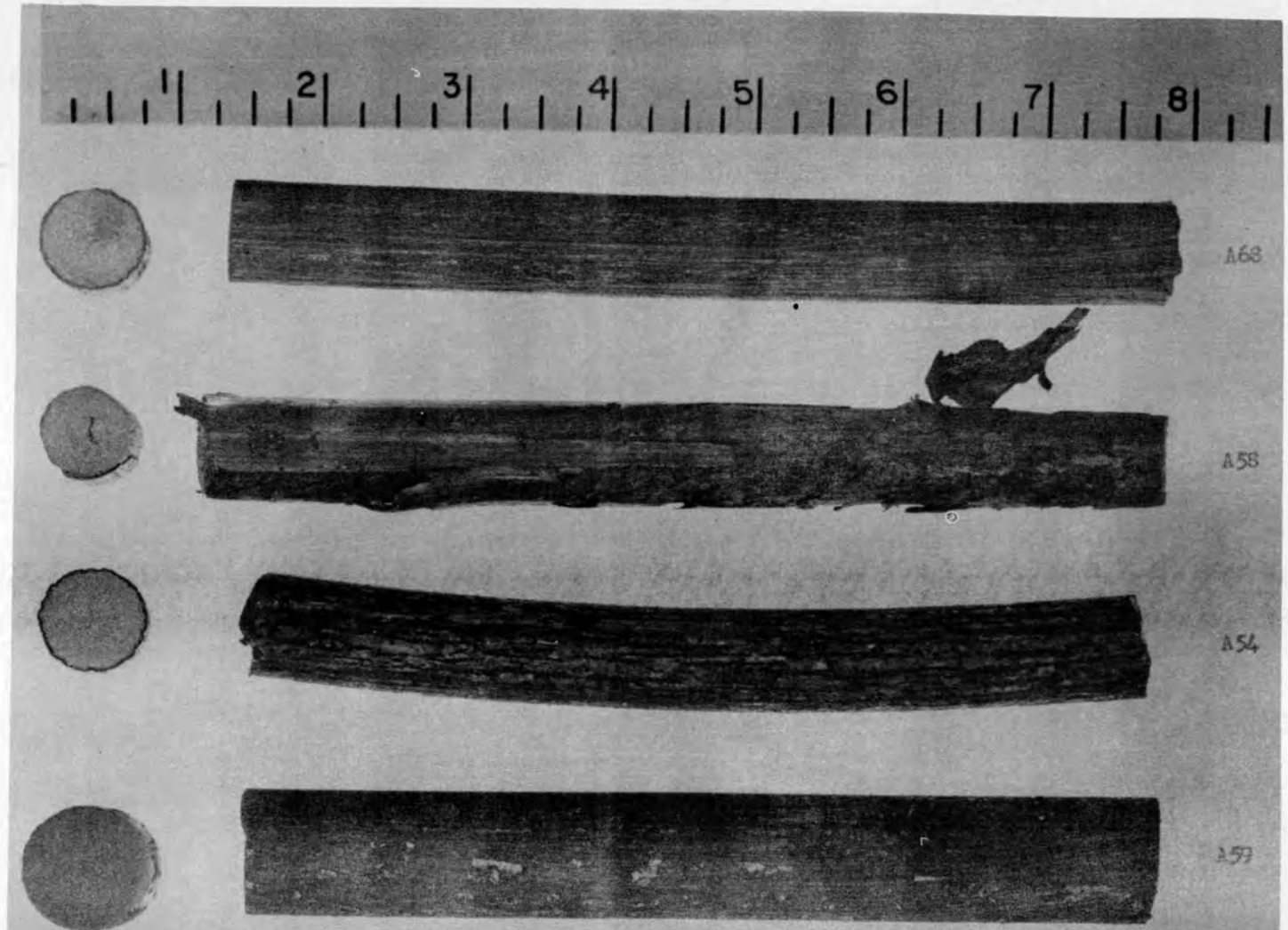
[redacted]

PHOTO Y-328-G



Billet No.	Extrusion Temp.-°C.	Reduction Ratio	Pressure on Billet-Tons/in.^{2}	Thickness of Can Plug	Lubricant
A68	950	17:1	59.2	1"	Glass
A58	950	17:1	83.2	1"	None
A54	950	17:1	56.0	2"	Glass
A59	1050	12:1		Bare	Glass

FIG. 7
BERYLLIUM EXTRUSIONS PRODUCED IN LABORATORY WITH AND WITHOUT GLASS AS A LUBRICANT

Information regarding the irradiation behavior of BeO alone, reported in ANL-4000, ANL-4005, ANL-4006, and CF-3807, indicates that neutron bombardment of pure BeO does not produce changes in density, dimensions, or elastic modulus greater than the limits of error of the measurements. Only in the case of thermal resistivity, which increased by a factor of about 1.6 during the course of the exposure, was any change in properties of the BeO observed. The information available gives little if any reason to believe that QM will be less satisfactory than the extruded vacuum cast beryllium in the MTR reflector.

6. *Beryllium Machining.* The fabrication of large cross-section pieces of beryllium has been predicated on the probability that long water-cooling holes can be drilled in the beryllium. Previous work showed that a 3/16 in. diameter by 40 in. length hole can be drilled by the gun barrel technique, and latest efforts are now concentrated on reducing the time of operation and minimizing the amount of hole run-out from straightness. During recent attempts, some tool breakage at the drill bit shaft joint has been experienced; however, it is felt that this difficulty can be overcome by the use of welding instead of brazing at the tip joint.

Deep-hole drilling experiments are being conducted at both the High Standard Manufacturing Company of Hamden, Connecticut, and Conner Tool Company of Detroit, Michigan. The companies are confident that a satisfactory tool can be designed and manufactured for drilling the desired water cooling channels in beryllium metal.

Fuel Elements

The thirty mock fuel assemblies, containing natural uranium as the active material, have been completed, inspected, and incorporated into the Materials Testing Reactor mockup. The current phase of the fuel element program is designed to diagnose and remedy the blistering of the aluminum-clad fuel plates and assemblies. This blistering occurs when the plates are annealed after fabrication, or during the subsequent brazing operation.

1. *Effect of Humidity on Blister Formation.* Because of the tendency of the aluminum-clad uranium-aluminum alloy fuel plates to form blisters during the brazing process, plates are tested after cladding and rolling, by annealing at 1100° F for three hours. Plates which blister in this test are reject-

ed. It has been observed that rejections are much higher in the spring than during the winter months. Information obtained from the Weather Bureau at the Knoxville Airport shows the relative humidity to be about 70 in the winter and about 50 in the summer. The absolute humidity values for 1948 ranged from 2.3×10^{-4} lb/cu ft in January, to 10.6×10^{-4} lb/cu ft in July. These represent the means of readings taken at 1:30 P.M. daily. A tabulation was then made of the humidities prevailing at the time of rolling about 1100 plates. These data, summarized and plotted in Fig. 8, indicate the importance of maintaining a low absolute humidity. A similar study of humidity at the time of casting showed no correlation with blistering.

As a result of these studies, a dry furnace atmosphere is now maintained, by passing compressed air through calcium chloride before introduction into the preheating furnace.

2. *Effects of Processing Variables on Blistering.* Table IV contains data pertinent to the casting, rolling, and aluminum cladding of a number of ingots of uranium (14 to 15%)-aluminum alloy. This work was done to study the effects of modification of certain processing variables on the tendency of blisters to form on clad fuel plate surfaces during subsequent reheating operations.

The usual practices employed in making these plates have been described previously (ORNL 323, Pt. I). Briefly, this practice involves the reduction of U_3O_8 with an excess of aluminum, with or without some scrap alloy, in a graphite crucible and using a protective cover of cryolite at atmospheric pressure. The slag is removed, and the alloy is pigged, remelted and cast in a rectangular graphite mold, to form an ingot approximately 1¼ in. × 5 in. × 10 in., and weighing about 2200 grams. The ingot, after cropping and inspection, is partially rolled at 1100° F; then clad with 2S aluminum and rolled to just under 0.3 in., the last few passes being made cold. Round cornered rectangular cores, 2 in. × 2.3 in., are punched out of this material on a press and placed in tightly fitting frames of 2S aluminum. This core-frame combination is partially rolled, and a flat sheet of aluminum is folded over it to cover both sides. The entire combination is then preheated and rolled, resulting in a well bonded aluminum-clad plate with a total thickness of 0.064 in. This is followed by a three hour anneal at 1100° F, which serves in part as an inspection for blister-forming tendencies.

The practices outlined above were used in the work reported in Table IV, except where variations, as noted in the table, were made for purposes of

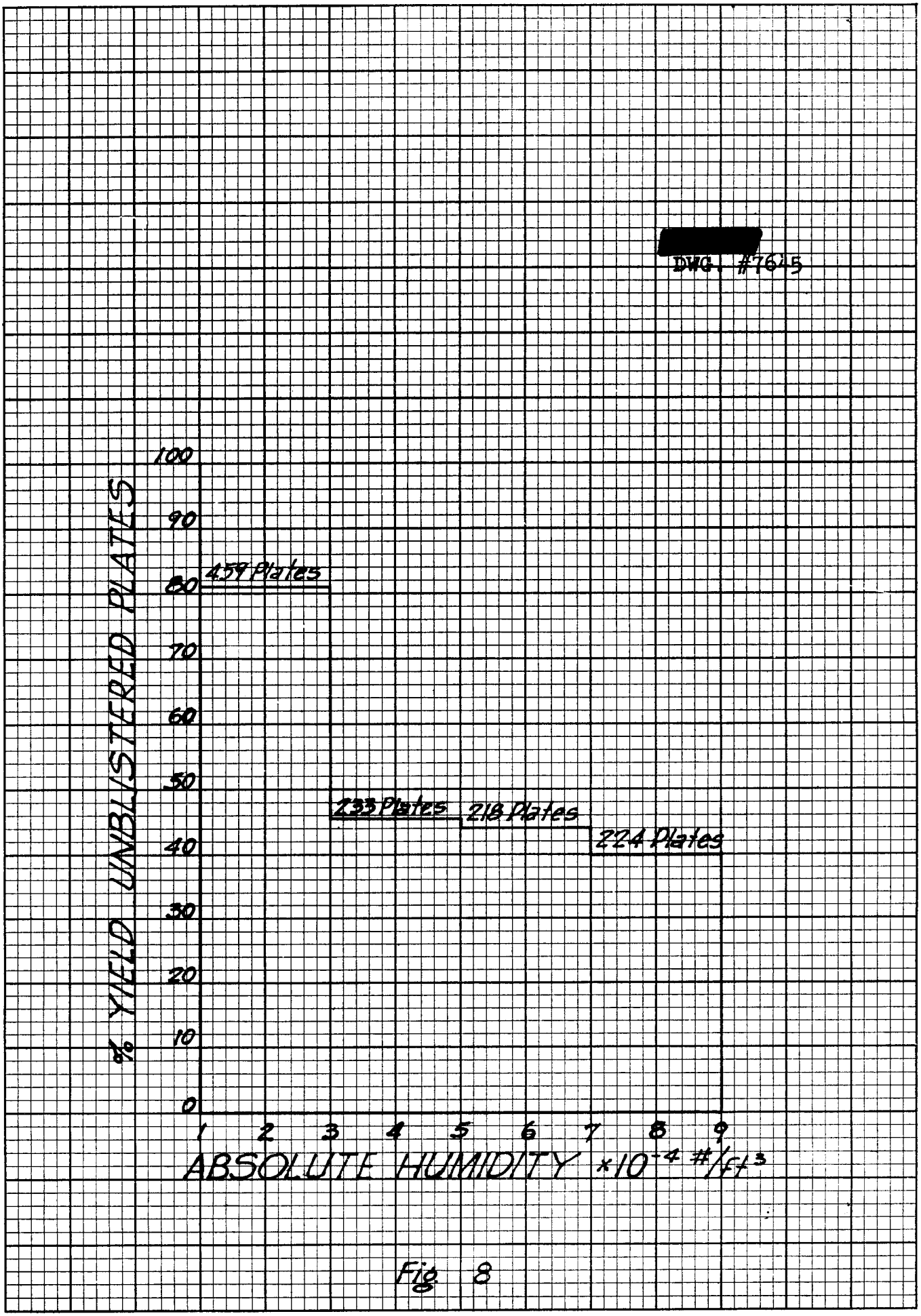

[redacted]
DWG. #7615
% YIELD UNBLISTERED PLATES
100
90
80
70
60
50
40
30
20
10
0
459 Plates
233 Plates
218 Plates
224 Plates
1
2
3
4
5
6
7
8
9
ABSOLUTE HUMIDITY $\times 10^{-4}$ #/ft^3

Fig 8

TABLE IV

Fuel Plates: Melting, rolling, and cladding data

	HEAT NUMBER	CHARGE IN REDUCTION HEAT	CASTING TEMPERATURE, °C	CASTING ATMOSPHERE	PREHEATING ATMOSPHERE FOR CORE, FRAME AND COVER	NUMBER OF CORES, TOTAL	NUMBER OF UN-BLISTERED CORES	
					Atm=Ordinary Atmosphere Dry=Dried Air prelim.=2 hrs. at 300°F, then 45 min. at 1100°F			
INGOTS QUENCHED IN WATER Cores, frames, and covers preheated for cladding roll in ordinary atmosphere	N1R	U_3O_8 + Al	800	Air	Atm	20	1	Numerous blisters, 10 to 20 per plate on both N1R and N2R
	N2R	U_3O_8 + Al	800	Air	Atm	20	2	N2R cores variously treated with oil, stopoff compound, and furnace desiccant as described in text
INGOTS AIR COOLED c, f & c preheated for cladding in dried air	N3R	U_3O_8 + Al + Scrap (N1R)	775	Air	Dry	22	7	Majority of blisters away from core ends. Three to ten blisters per plate.
	N4R	U_3O_8 + Al + Scrap (N2R)	775	Air	Dry	22	12	N124R had poor ingot surface and showed segregation in radiograph
	X124A	Scrap	722	Vacuum	Dry	22	15	
INGOTS AIR COOLED c, f & c preheated for cladding roll as indicated	N5R	U_3O_8 + Al + Scrap(N3R)	775	Air	Dry Prelim. Atm	7 6 7	6 3 1	Only one to three blisters per plate.
	N6R	U_3O_8 + Al + Scrap(N4R)	775	Air	Dry Prelim. Atm.	7 6 7	5 2 4	Blisters largely at core ends
	X124B	U_3O_8 + Al + Scrap(124A)	775	Vac. melt Air cast	Dry Prelim. Atm	7 6 7	5 1 2	

[redacted]

experiment. The principle modifications whose effects were studied include: drying of furnace atmosphere, water cooling of cast ingot, extent of recycling of scrap alloy, melting and casting under a vacuum as opposed to atmospheric pressure, and variations in preparing and processing the materials for cladding and rolling.

The results confirm the desirability of keeping the material and furnace atmosphere dry throughout processing. Quenching the ingots immediately after casting appeared detrimental. There is no evidence that remelting of the U-Al alloy influences the blister forming tendency. The vacuum melted ingot yielded a slightly higher proportion of blister free plates, but in some cases showed a somewhat increased segregation within the ingot. The use of commercial stop-off compound on the alloy core before cladding increased the seriousness of blistering; oil, similarly used, had little effect one way or the other; and plates heated in a furnace containing a commercial furnace desiccant, "Alorco", were both blistered and corroded.

In a run not listed in the table, sixteen plates were prepared in which aluminum cores were substituted for the uranium-aluminum alloy cores. Eight plates were preheated for cladding in the furnace containing dried air, and eight were preheated by conventional methods. One plate developed three blisters, which were located along the core edge, during the test anneal. This plate was one which had been preheated in a furnace with normal atmospheric moisture. Otherwise no blisters developed in the aluminum-cored plates.

Microscopic examination was made on eighty-two samples cut through blisters formed under practically all conditions represented by the tests reported in Table IV and in the preceding paragraph. In almost every case the blisters originated in the U-Al alloy core. In the case of the plates containing an aluminum core, the blisters originated at the core-frame interface, indicating entrapped air to be the cause of blistering in this instance. Typical blisters are shown in Figs. 9-12.

3. *Brazing*. After cladding and rolling to a thickness of 0.064 in., the fuel plates, whose production is outlined in the preceding section, are sheared and machined to 24-5/8 in. × 2.839 in. (Two plates in each group of 18 plates, are machined to a length of 28-5/8 in.). The shearing and machining do not expose the uranium-containing alloy. The 2.839 in. dimension is then curved to a 5.5 in. radius. Eighteen fuel plates, together with the necessary 2S aluminum side plates, are then assembled and furnace brazed to form the fuel assembly (described in MonT-433). The blistering which occurs during this

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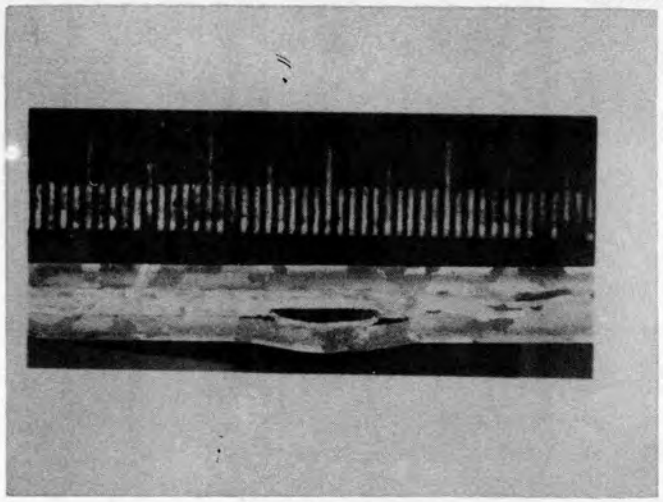

Fig. 9
Blister in Core of N1R Plate

Y-405

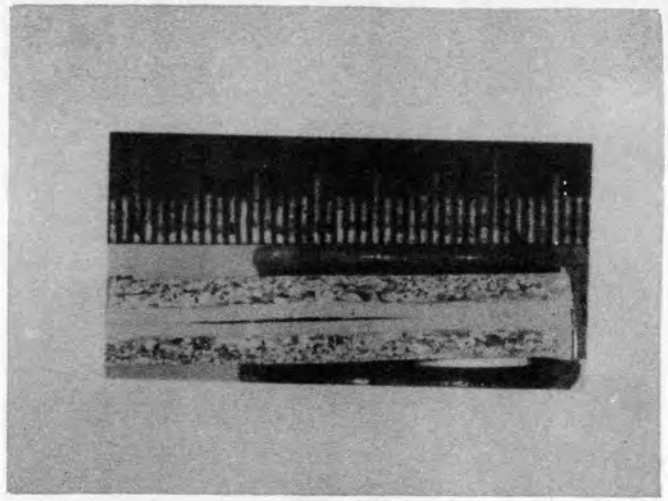

Fig. 10
Blisters in Core and in Cladding of 365R Plate

Y-404

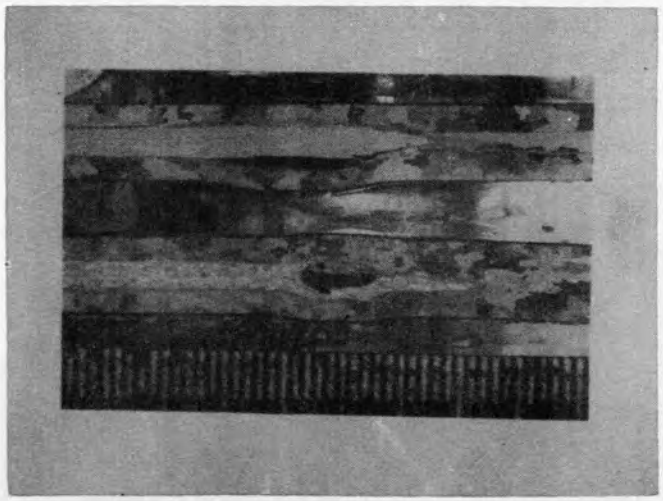

Fig. 11
Blisters at Ends of Two Cores in N6R Plates

Y-407

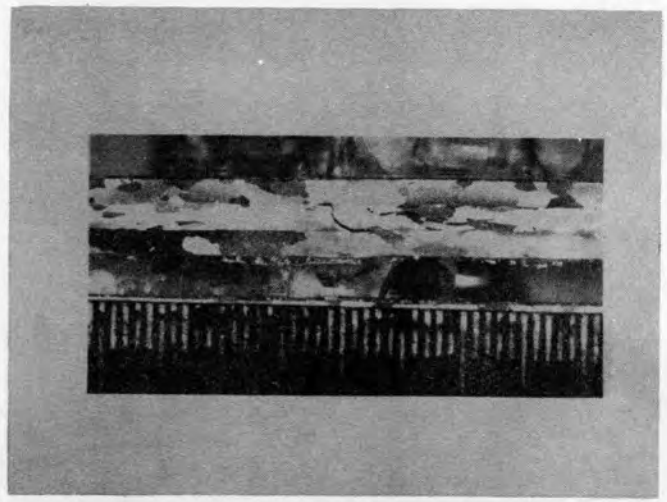

Fig. 12
Blister at Core-Frame Interface of Aluminum-Core Plate

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furnace brazing, has been responsible for several blistering investigations and process modifications.

In earlier work it was found that cleaning in an aqueous media—specifically, a hot caustic dip followed by immersion in nitric acid—promoted blistering. Fuel plates are now cleaned for brazing by means of vapor degreasing alone. Also, it has been observed that blistering tends to occur in the vicinity of areas to which the brazing flux has been applied. Tests have been made to obtain a better understanding of the effects of the use of brazing flux on plate quality.

Samples were prepared of regular plates, and of dummy plates containing 2S aluminum cores. These were subjected to the earlier cleaning cycle of dipping successively into hot caustic, nitric acid, and hot water, followed by drying at 300° F. Flux was then applied by either coating one edge, or by coating one entire surface, with a slurry containing three grams of Eutectic 190 Flux per two cubic centimeters of methyl alcohol. The plates were dried at 300° F, preheated at 850° F, and then heated for ¾ hr at 1100° F. Except for coating the entire surface with flux, this procedure simulated earlier fuel assembly fluxing practice.

Examination of the plates thus treated indicate that blisters are produced adjacent to, but not in the fluxed areas. No blisters occurred on those plates having one entire surface coated with flux. The blisters occur at the cladding interfaces. Blistering was more common in the plates which contained the uranium alloy as the core material.

A similar series of tests was made in which the drying temperature, before fluxing, was varied. No blisters appeared on the plates dried at 1100° F; only a few appeared on plates dried at 1000° F.

The results emphasize the importance of keeping the plates and material dry throughout processing, and of using suitable cleaning and preparation methods preliminary to brazing.

Materials Testing Reactor - Metallurgical Design

In addition to conducting the programs on beryllium reflector and fuel assembly development, representatives of the Division provide assistance to

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the Design Section of the Technical Division in other metallurgical and engineering problems pertaining to the MTR. In some cases this requires experimental investigation; in other cases the assistance is primarily of an advisory character.

1. *Materials Testing Reactor Window.* Plans for the MTR call for two windows, each 6½ ft square, to be made of a material transparent to neutrons but with high gamma radiation absorption. These units are to be installed vertically, and the maximum operating temperature is estimated at about 300° F. From the standpoint of radiation, bismuth and lead appear best suited to this service.

If bismuth is used, the window should be four inches thick, and the bottom loading will be 27.3 psi. Little information is available concerning the physical properties of bismuth at elevated temperatures, but, with a melting point of 520.3° F, it is unlikely that much can be expected in the way of strength or creep resistance at 300° F. This and the fact that absorption of neutrons by bismuth produces an alpha-emitting polonium isotope, indicate that the bismuth, if used, should be canned. Efforts to can the material by casting into 4 in. × 4 in. square aluminum tubes produced cracked castings due to the expansion of metal upon solidification. A modified casting technique can probably overcome this tendency.

A lead window, if used, should be but 2 in. thick, because of greater neutron absorption by lead. Such a window would have a bottom loading of 32 psi. More data regarding creep and physical properties of lead are available than for bismuth, although none are available in the range of loading specified in this design. A lead producer has recommended a maximum allowable fiber stress of 80 psi at 300° F for chemical or for tellurium lead, and 180 psi for a lead alloy containing 5% tin.

Additional information is being obtained and submitted to the Design Group. In view of the possibility of creep and buckling of the relatively thin slab, suspension of the slab in the design has been recommended.

2. *Shim Safety Rod, Cadmium Section.* A considerable portion of the shim safety rod which is used as a control for the MTR is, as presently designed, to be made of stainless steel. It has been suggested that the stainless steel portion be replaced by aluminum to facilitate machining and to decrease

[redacted]

galvanic corrosion.

Fabrication of a suitably protected cadmium unit is a problem. As a part of this proposal, an aluminum clad tubular cadmium insert has been made, Fig. 13. A sheet of cadmium was sandwiched between sheets of aluminum and formed into an open cornered square tube, measuring 2-1/8 in. × 2-1/8 in. × 28 in. A heliarc sealed joint was made along the contacting edges, and the edges were also sealed at the ends to provide complete protection for the cadmium.

Consideration has also been given to the possibility of fabricating a cylindrical unit by casting cadmium in the annular space between concentric aluminum tubes. Such a unit can be altered in shape after final sealing. Preliminary tests indicate that Alcoa X 62 Flux promotes bonding of cadmium to aluminum, but that gassing, due to the presence of flux, occurs during casting.

3. *MTR Mockup Tank Gaskets.* When the MTR Mockup was first placed in operation, leakage occurred at some of the large tank gaskets. The use of gaskets with a circular cross section was proposed, since the initial contacting areas are small and plastic flow accommodates surface irregularities. A test was made on a one inch piece of ¼ in. diameter annealed 2S aluminum, which showed that at 6000 lb loading, the rod flattened to a thickness of about 0.17 in. producing a contact width of about 0.24 in.

4. *Plate Spacing Gauges.* The cam type plate spacing gauge has been reported in ORNL 255. This type of gauge will be used where accurate measurement is necessary in the development of fuel assembly fabrication techniques. In addition, two types of go-no go cam gauges have been designed to permit faster inspection of plate spacing during actual production operations. These gauges will indicate only whether or not space dimensions fall within the ten percent tolerance now specified.

5. *Upper Adapter Retaining Rings.* It was observed after MTR Mockup tests, that some of the upper adaptor retaining rings of the fuel assemblies stuck and kept the spring compressed. This may be due largely to unequal spring pressure. A suggestion has been made that splines be used to decrease the contacting surface area. This should minimize the tendency for seizure if corrosion products build up during operation.

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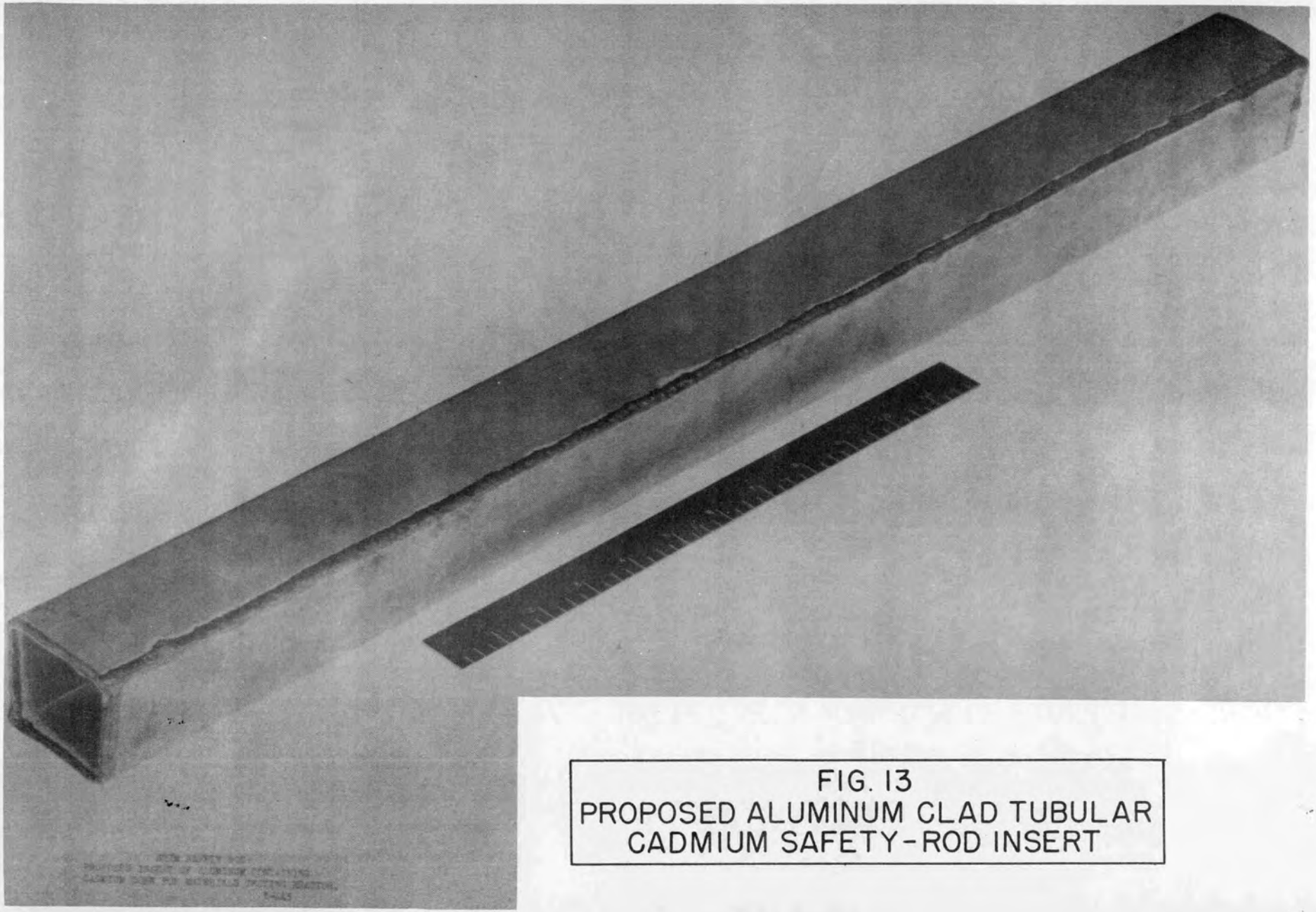

FIG. 13
PROPOSED ALUMINUM CLAD TUBULAR
CADMIUM SAFETY-ROD INSERT

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THORIUM PROGRAM

The division is undertaking an extensive investigation of thorium and its alloys. Fundamental physical metallurgy studies will be directed toward clarification of the physical constants of the metal; for example, the melting point, boiling point, modulus of elasticity, and Poisson ratio of pure thorium are still in doubt. The effects of small amounts of elements such as carbon or oxygen on precipitation hardening and on recrystallization, will be studied.

The development phase of the program will be designed to devise techniques for melting, casting, and fabrication of the metal, and to develop useful thorium alloys. Melting and casting will be studied in both a vacuum induction furnace and an arc furnace. An induction furnace using a tilting crucible is being designed. The initial experiments will be directed toward obtaining suitable crucible refractories. A vacuum arc furnace with a water cooled copper crucible has been put into operation and preliminary tests have been made on steel melts.

The first alloy systems to be considered will be the following:

1. *Th-Be, Th-Si, Th-Be-Si.* These systems are of interest because of reported improvement in corrosion resistance, and because substantial lowering of the melting point of thorium results from the addition of either beryllium or silicon. There is some possibility that a ternary eutectic, of relatively low melting point, may be formed.

2. *Th-Zr, Th-Ti.* Zirconium and titanium may be interesting from the standpoint of grain refinement. Titanium is reported to reduce the hardness of Ames cast metal.

3. *Th-Mg, Th-In.* The atomic size factors for magnesium and indium indicate the possibility of appreciable solid solubility.

4. *Th-Ce.* Cerium is reported to be completely soluble in the liquid and solid states.

5. *Th-Mn.* While manganese is undesirable from the standpoint of nuclear properties, it is reported to improve cold workability.

6. *Th-Cb.* Columbium is also reported to improve workability, presumably by removing carbon from solid solution and the formation of stable carbides.

The experimental alloys will be prepared by arc melting, induction melting or powder metallurgy. After fabrication by rolling, forging or extrusion,

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interesting alloys will be tested for mechanical properties, physical properties and corrosion resistance.

Continued development effort will be applied to the cladding or other protective coating of thorium shapes and the fabrication of assemblies for breeding applications in reactor.

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FUNDAMENTAL STUDIES

Mechanical Properties of Pure Metals

An urgent demand exists for knowledge of the behaviors of materials of construction, particularly metals, in reactors. Since the effects of neutron bombardment appear in some respects to be analogous to those induced by cold work, a thorough understanding of plasticity of metals is required. Plasticity is being studied in many laboratories and is under rapid development, but much clarification of controversial and uncertain points must be accomplished. It is believed that knowledge of the fundamental plastic laws will be advanced by obtaining a better understanding of testing methods and by the establishment of fundamental data regarding the mechanical properties of pure metals. For these reasons, an extended study of the plasticity of unalloyed metals has been undertaken by the Division.

One of the first requirements of this projected work was the design and construction of a gauge for recording true stress strain data and of straining rates. During the fall of 1948 a previously designed gauge was built and tested. This gauge embodies the use of a motion picture and single frame camera for taking simultaneous photographs of test specimens and load gauge, and a time piece for rate recording by measuring the film.

By measuring the film with a densitometer and photographing a comparison standard of length adjacent to the specimen, a precision of measurement equal to that obtained by direct reading on a knife edge micrometer is obtained. Tests have revealed that various changes will yield a larger photographic image of the test piece and also give a stronger boundary contrast.

The purposes of the gauge are to photograph the test piece, the time piece, and the load gauge, on a 35 mm strip film at regular intervals during stressing, and to derive stress-strain curves at various stipulated rates of loading or straining (0.1 in. sec^{-1} to 0.00001 in. sec^{-1}). Figure 14 shows a photograph of the gauge mounted on the Baldwin Southwark 120,000 pound testing machine, with a tensile test specimen in place. Originally the pressure indicating dial pointer shown was mechanically connected to the regular indicating dial gear. At present it is indirectly connected to the regular dial gear by selsyn motors; but it will eventually be directly operated from the capsule through a special Bourdon tube.

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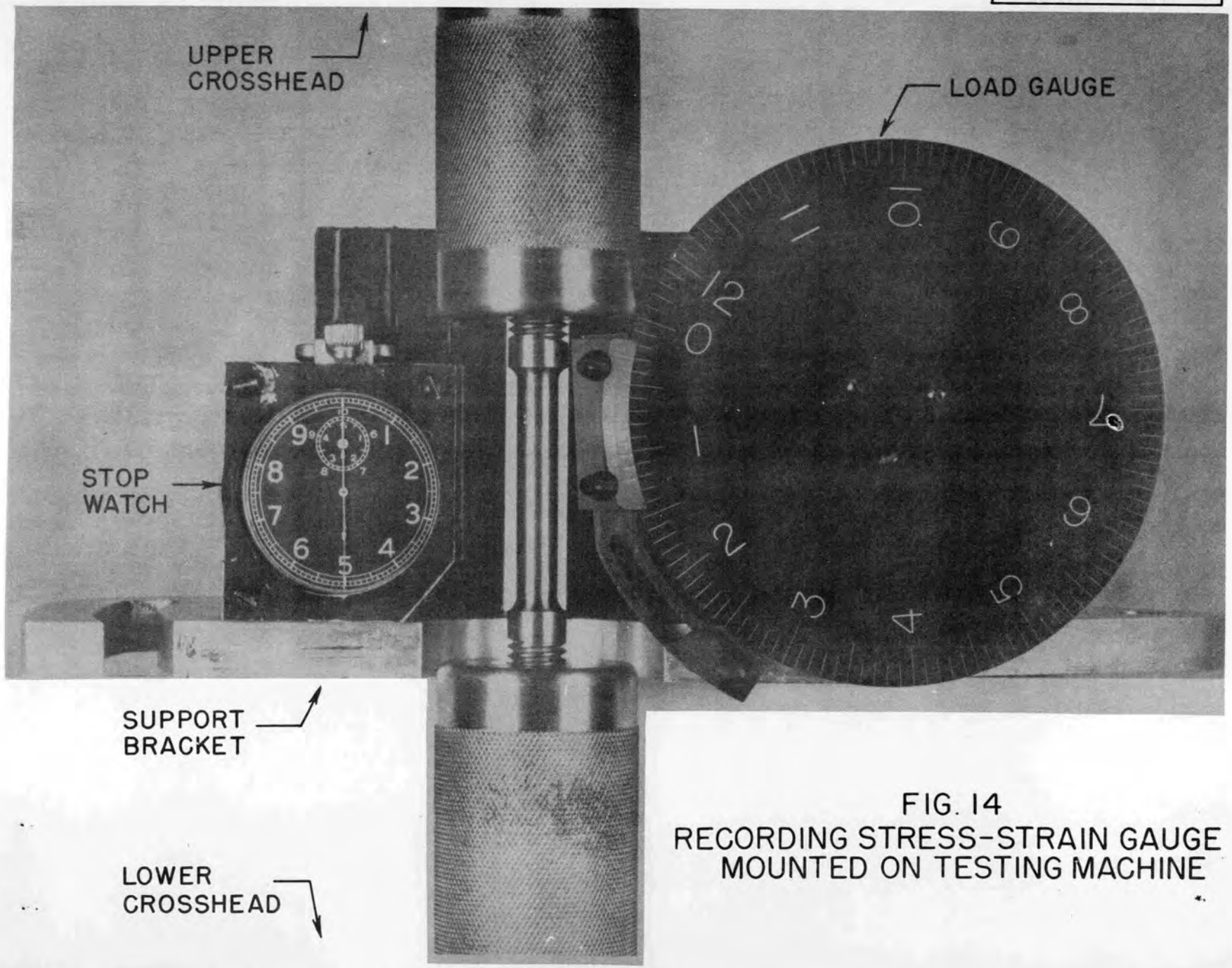

FIG. 14
RECORDING STRESS-STRAIN GAUGE MOUNTED ON TESTING MACHINE

The test specimen is illuminated from both front and rear so that it appears in silhouette against a light background. The size of the image of the specimen will be proportional to its actual dimensions. The films may be measured by means of a densitometer. Figure 15 shows a diagram taken from a film by a densitometer. As this procedure is slow and expensive, measuring the films by a microscope is being tested. Projection on a ground glass screen and measurement by a micrometer have also been tried. An internal standard length is used, and the measurement is essentially a comparison of two lengths, one of which is known to 0.0003 in. on the same film.

The front illumination of the specimen serves to outline a grid of fine lines, reproduced photographically, on the surface of the specimen. Diametral as well as length, extension and change in shape of specimen are thereby recorded. It is planned to expose ten to thirty frames during any one test. Excellent illumination is required, and means of illumination are now under study. While using a Sept camera, microfile film and D-11 developer gave images of excellent definition. The Sept camera gives good pictures at single frame exposures during slow loading, such as will be taken periodically by a timing device and an automatic trigger mechanism. Used as a motion picture frame, at high rates of loading, it is not quite satisfactory, and a 35 mm De Vry Model A motion picture camera may be used for study of loading rates of 0.1 in./sec to 0.01 in./sec.

Physical Metallurgy of Beryllium

At the time the Division undertook the development work on beryllium for the Materials Testing Reactor, a limited amount of physical metallurgy research on the metal was also started. This research was intended to investigate some of the suspected causes of cracking and brittle failure and to study means of improving the properties of beryllium.

1. Microscopic Study. Samples of extruded beryllium from various lots and with different degrees of purity were studied microscopically. These showed no evidence of a second phase at the grain boundaries which might be responsible for the brittleness, although microradiographic work has given indication of such a second phase. (Also, MIT-1018, page 22, reports a grain boundary constituent in vacuum cast beryllium.)

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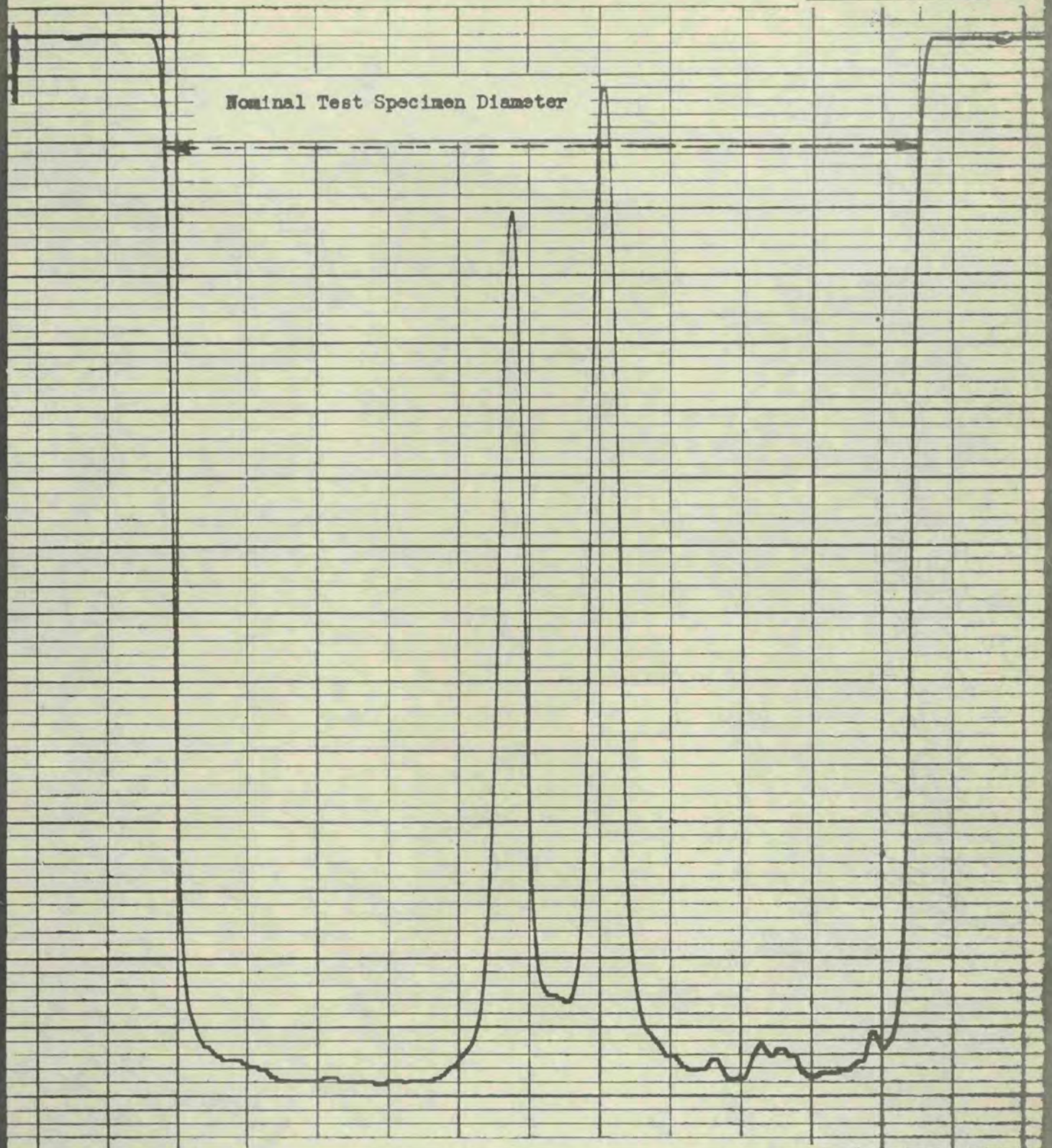

FIG. 15
MEASUREMENT OF TEST SPECIMEN DIAMETER
FROM FILM, WITH A DENSITOMETER

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A study was made of a number of fractures of samples of extruded beryllium which were polished for microscopic examination and then broken. The broken ends were carefully fitted together and the fractures examined. Cracks were observed to follow grain boundaries and to cut across the grains, to an approximately equal extent (Fig. 16). Specimens of very coarse grained beryllium were broken in compression and these fractures also were about equally trans- and intergranular. Small cracks have also been observed entirely within coarse beryllium grains.

2. *Preparation of Beryllium Phosphide.* It is planned to study the effects of various fluxes, slags, and alloy additions on the purity and properties of beryllium. As a part of this program, the beryllium-phosphorus system will be investigated. One of the first requirements in this phase of the work is the preparation of beryllium phosphide.

From a literature survey, there does not seem to have been much interest in beryllium phosphide. Wöhler [Pogg. Ann. 13:580 (1827)] first prepared it in 1827 by passing phosphorus vapor over finely divided beryllium metal. Lebeau [Comp. Rend. 120:1272 (1898)] prepared Be_3P_2 by two methods. The first was the same as Wöhler's and the second was by reacting phosphorus with beryllium iodide. R. Paulus [Z. Phys. Chem (B) 22:305 (1933)] prepared Be_3P_2, also using the method of Wöhler, to determine the X-ray diffraction patterns of beryllium phosphide.

Beryllium phosphide is brown to brown-black, with a density of 2.25 ± 0.02 gm/cc and a lattice constant of 10.15 ± 0.03 A. The material is extremely reactive with water, forming beryllium oxide and phosphine gas. Even with a short exposure to air, the reaction with water vapor proceeds rapidly. The melting point has not been determined but is higher than the melting point of beryllium metal. Even at these high temperatures there is no apparent breakdown of the compound.

The reaction between phosphorus and powdered beryllium proceeds rapidly at the temperature at which red phosphorus vaporizes. The beryllium phosphide produced is then melted with pebble beryllium. As yet none of the chemical analyses or metallographic results are available.

The reaction between phosphine and beryllium was attempted. Powdered metal was placed in a boat and brought to a red heat in a stream of helium. PH_3 was then introduced. Unfortunately, after the gas had passed through only a short time, there was a backflow into the PH_3 generator, which stopped the

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POLARIZED LIGHT

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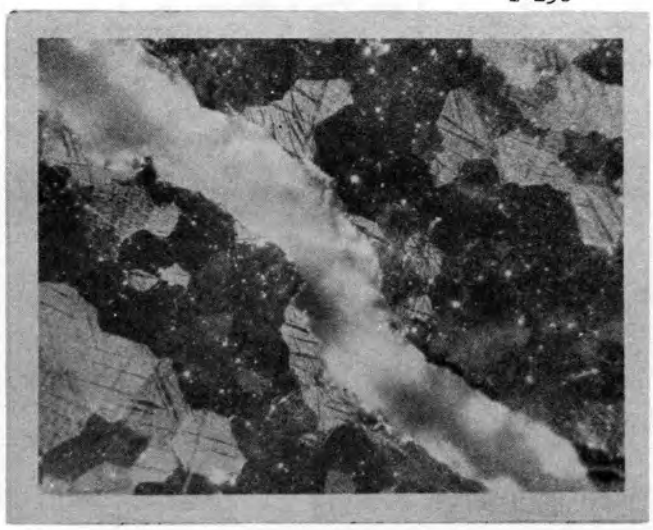

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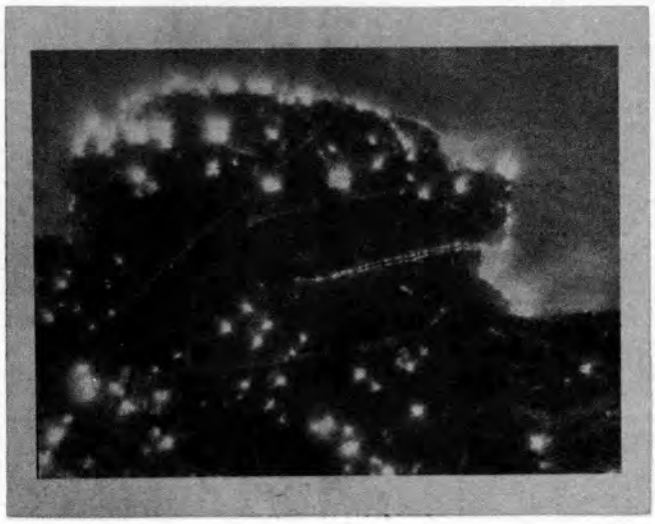
75X

Y-290



500X

FIG. 16
FRACTURES ACROSS POBISHED SURFACES
OF EXTRUDED BERYLLIUM SPECIMENS

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experiment. Upon removal of the Be powder there was no visible sign of any reaction having taken place.

The work of Holtje and Meyer [Z. Anorg. Chem. 197:93 (1931)] on the reaction of beryllium chloride and phosphine was confirmed. They stated that there was no reaction between the two, even at high temperatures and pressures. PH_3 was passed through a quartz tube in a Burrell tube furnace at 800° C, and vaporized $BeCl_2$ introduced. The $BeCl_2$ condensed on the cool walls of the tube after passing through the furnace, and there was no reaction product.

A single run was made to determine if there was any reaction between beryllium oxide and phosphorus. Beryllium oxide and phosphorus were placed in a closed end tube and the phosphorus refluxed at approximately 900° C under a helium atmosphere. After two hours there had been no apparent reaction. This was confirmed by a qualitative analysis.

Preferred Orientation and Anistropy in Properties of Metals

As a consequence of the preferred orientations developed in polycrystalline metals during plastic deformation alone or with subsequent recrystallization, the mechanical as well as other properties differ for different directions in the metal. A study of this anisotropy in mechanical properties for various metals was recently initiated with a view to attempting a quantitative treatment of the correlation of anistropy with type and degree of preferred orientation resulting from various types of deformation. Such a treatment might be attempted, for instance, on the basis that the deformation resistance of the metal in various directions, particularly in the early stages of a tension or compression test, is related to the percentage of the grains in the various orientations and the shear stress on the slip systems of the grains in each orientation. Investigation has been started on uranium, and later will be extended to beryllium, thorium, and perhaps other metals.

Work to date has centered on standardizing testing variables, such as strain rate, to obtain reproducible results, and on eliminating or at least evaluating conditions that might lead to spurious anisotropy, such as mechanical fibering and residual stress patterns. There are indications that the tensile stress-strain curve is affected by the rate of cooling of uranium from the working or annealing temperature. Presumably the thermal stresses set up

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as a result of the anisotropy of thermal expansion are retained at higher levels in material cooled at the faster rate. These indications are now being checked in more carefully controlled experiments.

Internal Energy Measurements

Plans are in progress for the development of equipment for measuring internal energy changes in metals. A modification of Sykes' method is being considered. Some changes will be made in the design of the equipment and accuracy will be improved by the use of automatic control. An analysis of the method and errors involved indicated an expected accuracy of determination of 0.01 cal/g. For cold worked metals (0.5 to 1.5 cal/g internal energy due to cold work) this gives an error of approximately 1%, increasing as the internal energy decreases. This is adequate for the experimental work planned.

If it is possible to obtain the anticipated accuracy, internal energy measurements will be made and the data utilized in fundamental studies on the types of distortion produced in metallic lattices by cold working, transformations, solid solution, and radiation. This work is being done as part of the joint program with the Physics Division on Physics of the Solid State.

Effect of Manganese on Rates of Growth and Rates of Nucleation of Pearlite

The effect of manganese on the rate of growth and rate of nucleation of pearlite in high purity Fe-C-Mn alloys, has been undertaken as a fundamental problem on reaction rates in the solid state. This work is being done by graduate students placed in the Metallurgy Division for research during the summer by the Oak Ridge Institute of Nuclear Studies.

A high purity Fe-C-Mn (0.75% C, 1.02% Mn) alloy has been made from electrolytic iron and manganese, by vacuum melting and subsequent forging and rolling to 3/8 in. round. Microscopic and chemical examinations have shown the material to be satisfactorily homogeneous. Samples are austenitized in a purified nitrogen atmosphere and isothermally transformed in a stirred lead bath. All thermocouples have been regularly checked against Bureau of Standards pure

[redacted]

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metals, allowing an accuracy of reading to ± 0.5° C with control of the lead bath held to ± 0.3° C. Long transformation times are being obtained by an electric stop clock activated by contact of the sample with the lead bath. Shorter transformation times will be determined by a special Brown controller activated by a thermocouple welded to the specimen. Transformation times and temperatures will be taken directly from the controller chart.

The experimental data will be compared with previous data on pure iron-carbon alloys, and an attempt made to study the mechanism whereby alloying elements influence the pearlite transformation in particular, and hardenability in general.

Some work is being done on improved accuracy of determination of grain size in eutectoid steels and the relation of grain size distribution to grain surface area. If time permits, similar work on 1% Cr eutectoid steel will be made.

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FABRICATION OF URANIUM—SERVICE WORK

Rolling Foil from Depleted Uranium

Two ingots, approximately ½ in. × ½ in. × 0.153 in., of depleted uranium (0.01% U^{235}) were rolled to foil for the Physics Division. The cast material was jacketed with 0.055 in. mild steel, the edges of which were welded. This was hot rolled at 1200° F to 0.028 in. with reductions of 5% per pass. The cover was removed and the pieces, now 0.012 in., rejacketed with 0.055 in. mild steel. This jacketed assembly was then rolled at 1200° F, also to 0.028 in., with reductions of 10% per pass. The jackets were removed, the uranium pieces measured 0.002 in. thick. One was rejacketed and hot rolled as before, at 10% per pass, to a total reduction of 50%. When the jacket was removed the foil was 0.001 in. thick.

One piece of 30% uranium (0.001% U^{235}), measuring approximately ½ in. × ½ in. × 0.125 in. was hot rolled to 0.003 in. This material was inserted in an aluminum frame and rolled, without a cover, at 1100° F.

Preparation of Small Square Wires of Enriched Uranium for Neutron Exposure Tests

At the request of the ORNL Solid States Group, enriched uranium wires have been prepared for irradiation studies, by rolling and swaging the uranium from one inch to 0.12 in. square at 300° C. The procedure, which was first developed using natural uranium, includes (1) alpha-rolling to 3/8 in. round rod, (2) alpha-swaging to 0.162 in., (3) squaring in a hand-cranked rolling mill, and (4) drawing through square dies to finished size. Hardness tests and microscopic examination of the metal were made at different stages of fabrication.

The natural uranium used was in the form of 1-1/16 in. round billets (ORNL reactor slugs). Enriched (2.7 and 6.5% U^{235}) uranium was in the form of ingots one inch square. These ingots or slugs were annealed in vacuum at 700° C for two hours. Prior to rolling in a 6 in. × 10 in. rolling mill, they were heated to 300° C, in either silicone fluid or quenching oil. The billets were rolled to ¾ in. round in five roll passes; the roll pass openings were adjusted to give 3 to 5% drafts with each opening. The ¾ in. rods were reannealed in

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[redacted]

vacuum at 900° C and rolled to ½ in. diameter in six rolled passes, using 10 to 20 settings and 3 to 5% drafts per pass with each setting. After annealing at 900° C the rods were similarly rolled from ½ in. to 3/8 in. diameter, and reannealed.

The rods were than swaged to 0.163 in. diameter, using 15 high speed steel dies at a die temperature of 150 to 200° C. Rods were heated in oil to 300° C before each die pass, heated with a gas torch during swaging, and reannealed after passing through four or five dies with a maximum total reduction of 30%. For rods less than 3/8 in. diameter, annealing was done at 675° C for three hours. Swaging was advantageously continued to 0.163 in. diameter, although in some cases rods were swaged to both larger and smaller sizes.

Following the swaging operation the round rods were squared by rolling through diamond shape passes in a 3 in. × 4 in. hand-crank driven rolling mill. After squaring, they were rolled to 0.133 in. or 0.125 in. squares using drafts and temperatures comparable to those used in swaging.

The repeated alpha-working and alpha-annealing of the metal resulted in a fine grained structure with equi-axed grains, with no trace of the mosaic of grains within grains which results from heating through allotropic changes. The hand rolling mill did not permit finishing to close dimensions. Straightening and dimensional finishing were accomplished by drawing the square rods on a Baldwin Southwark 120,000 lb tensile machine, using an appropriate die holder and square dies. The temperature for successful drawing is limited to a narrow range, in the vicinity of 150° C. Good lubrication of wire and die was necessary; a "Stancoat No. 1" drawing compound gave better service than oils or stearates. The square rods were heated on a hot plate and drawn through two dies with about an 11% draft in each. The load required ranged from 96,000 psi to 130,000 psi, and was very near the yield point of the metal. Drawing speed was about 14 in. per minute, the speed of the testing machine cross head.

Table V shows the distribution and recoveries of uranium accomplished in the preparation of the specimens. A close check of weights of the enriched material was kept at all stages of working, annealing, cutting and cleaning.

Mechanical Properties. Rockwell hardness values for different samples in various stages of treatment are shown in Table VI. Samples 9 and 10, enriched material, responded in a different manner to working and heat treatment than did samples 6, 7 and 8, normal uranium. Photomicrographs, Figs. 17-20, show a considerable difference in microstructure. The presence of a eutectic-like

[redacted]

TABLE V

Distribution and recovery of uranium in preparation of irradiation specimens

	LOT NUMBER	U^{235} CONTENT	VACUUM FURNACE	WORKING EQUIPMENT	CLEANING SOLUTIONS	METAL CHIPS, FILINGS	CROPPING SCRAP	METALLOGRAPH SPECIMENS	3 mm sq WIRE	TOTAL
WEIGHT DISTRIBUTION (grams)	10	2.7%	.485	12.722	2.950	27.116	465.340	69.743	482.004	1060.35
	9	6.5%	.362	15.560	2.112	27.566	398.029	62.465	551.925	1058.02
		TOTALS	.847	28.282	5.062	54.682	862.369	132.208	1033.929	2118.37
PERCENT DISTRIBUTION	7	Normal	.19	.55	.31	17.0	24.0	18.5	39.95	
	6	Normal	.11	.38	.34	6.18	11.0	10.58	70.58	
	10	2.7%	.046	1.20	.28	2.56	43.88	6.56	45.46	
	9	6.5%	.034	1.47	.20	2.60	37.62	5.90	52.17	

[redacted]

TABLE VI

Rockwell hardness of uranium—
Normal and enriched

U^{235} CONTENT	NORMAL No. 6	NORMAL No. 7	NORMAL No. 8	2.7% No. 10	6.5% No. 9
Annealed at 900° C, 2 hours	B-81	B-84	B-82	B-91	B-89
Annealed at 700° C, 2 hours	B-88	B-89			
Annealed at 625° C, 3 hours	B-94	B-96	B-90	B-97	B-96
Rolled 45% at 280° C, rounds	C-32		C-32	C-35	C-34
Rolled 45% at 280° C, squares	C-34				
Swaged 30% at 300° C, rounds			C-26	C-28	C-26
Drawn 10% - 20%, squares	C-33		C-30	C-31	C-31

Y-413

Fig. 17
Normal Uranium. Annealed at 900°C for 2 hours after rolling at 300°C. Electrolytic polish. Vertical Illumination. X750

Y-414

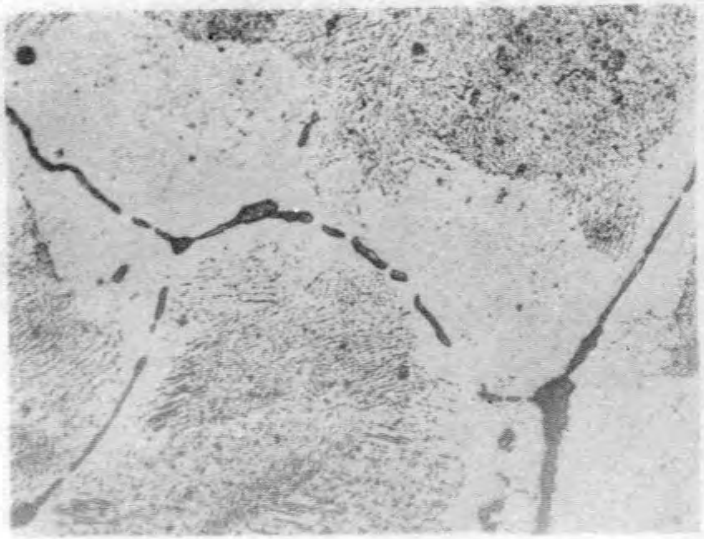

Fig. 18
Enriched Uranium (2.7% U^{235}). Annealed at 900°C for 2 hours after rolling at 300°C. Mechanical polish. Vertical Illumination. X750

Y-416

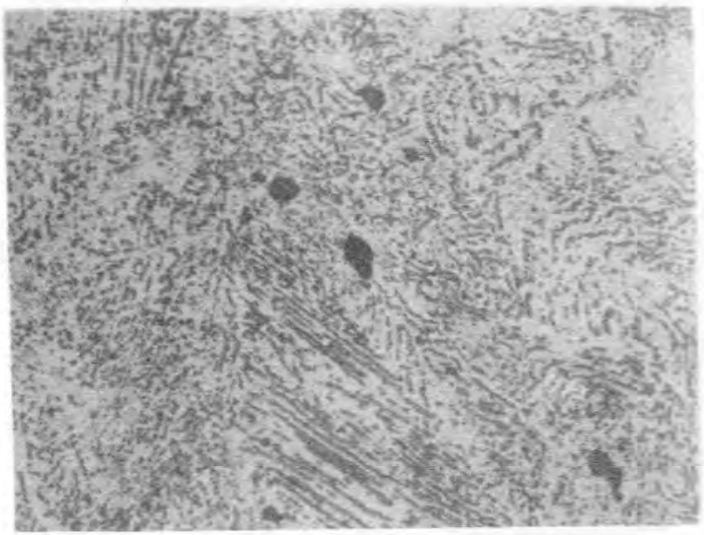

Fig. 19
Enriched Uranium. 6.5% U^{235}. Annealed at 900°C for 2 hours after rolling at 300°C. Mechanical Polish. Vertical Illumination. X750

Y-415

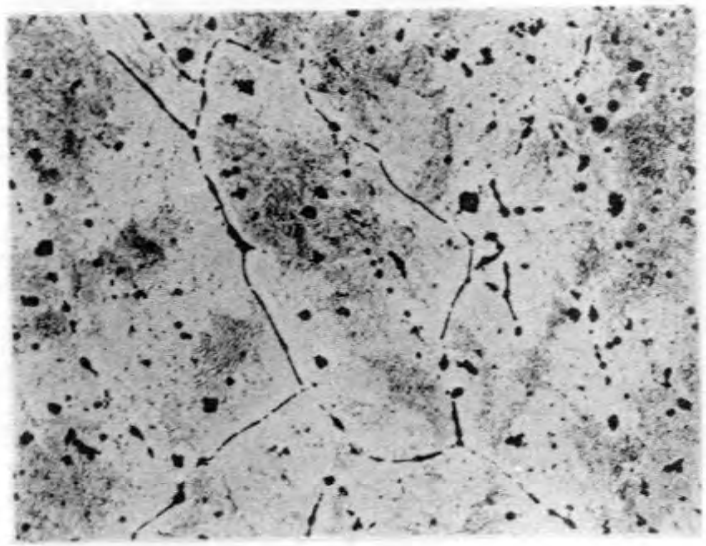

Fig. 20
Enriched Uranium. 2.7% U^{235}. Annealed at 900°C for 2 hours after rolling at 300°C. Mechanical Polish. X250

[redacted]

structure in Figs. 18-20, representing the enriched material, indicates the probability of the presence of impurities. Chemical analyses are being obtained but are not yet available. The enriched samples pitted so badly in electrolytic polishing that polishing had to be done mechanically. Photomicrographs, Figs. 21-26, show the natural uranium in various stages of processing. Tensile test data on normal and on enriched uranium are presented in Table VII. Figures 27-30 show typical load strain curves for the specimens tested, reproduced directly from recorder charts. Reduction of area and elongation values of some of the normal uranium specimens are low and show much scatter. This is possibly due to the physical condition of specimens, which were not entirely free from corner cracks. The enriched metal specimens were prepared later, using the experience gained in preparing the normal uranium specimens. Both tensile strength and fracture stress values represent load divided by area at fracture. Tensile strengths compare well with similar values for natural alpha-rolled uranium. Load strain curves were obtained using Peters Company standard microformer automatic recording devices attached to a Baldwin Southwark 120,000 lb tensile testing machine.

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Y-72

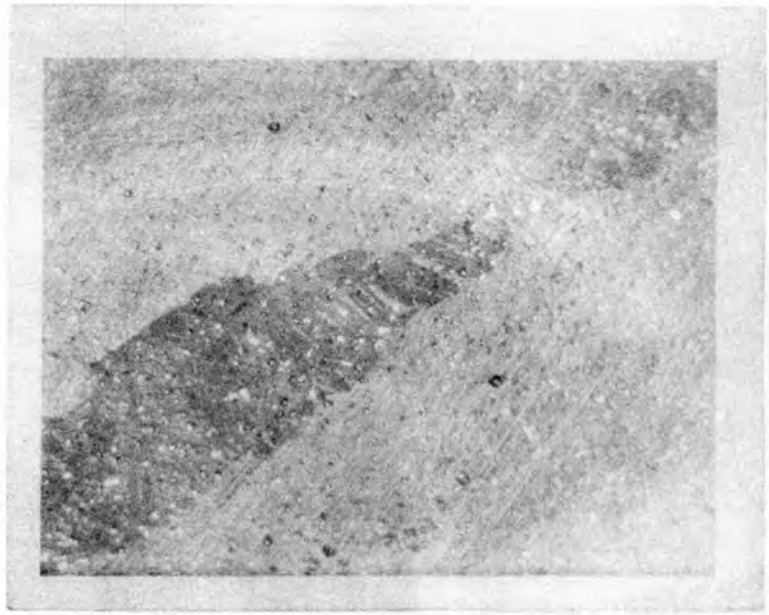

Fig. 21
Natural Uranium Cross-section. Annealed at 900°C for 2 hours. Rolled at 300°C from 1-1/16" to 3/4". Polarized light. X100

Y-145

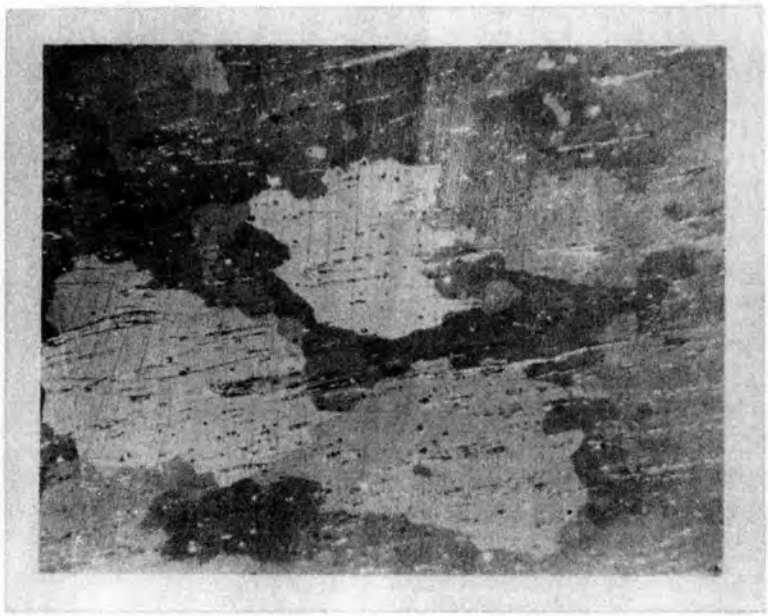

Fig. 22
Natural Uranium. Cross-section. Annealed at 900°C and rolled from 3/4" to 17/32" at 300°C.. Polarized light. X100

[redacted]

[redacted]

Y-150

Fig. 23
Natural Uranium. Cross Section. Annealed at 700°C for 1/2 hour. Swaged from 0.258 in to 0.229 in. at 300°C. Polarized light. X100.

Y-138

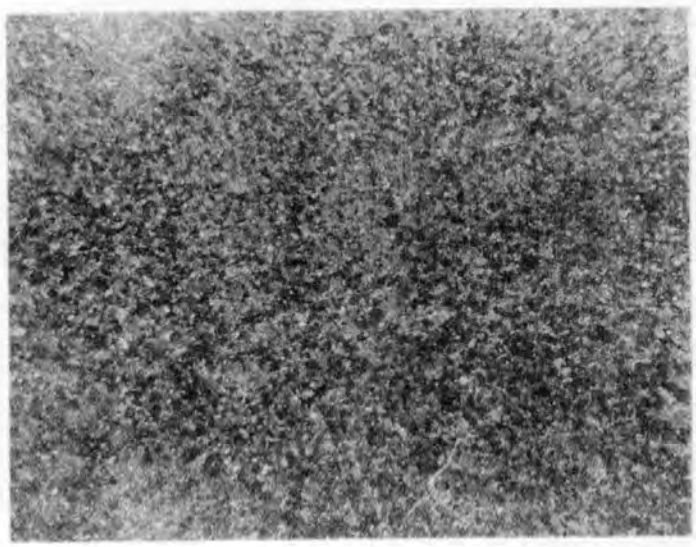

Fig. 24
Natural Uranium. Rolled from 0.205 in. to 0.153 in. and annealed at 625°C for 3 hours. Polarized light. X100

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[redacted]

Y-147

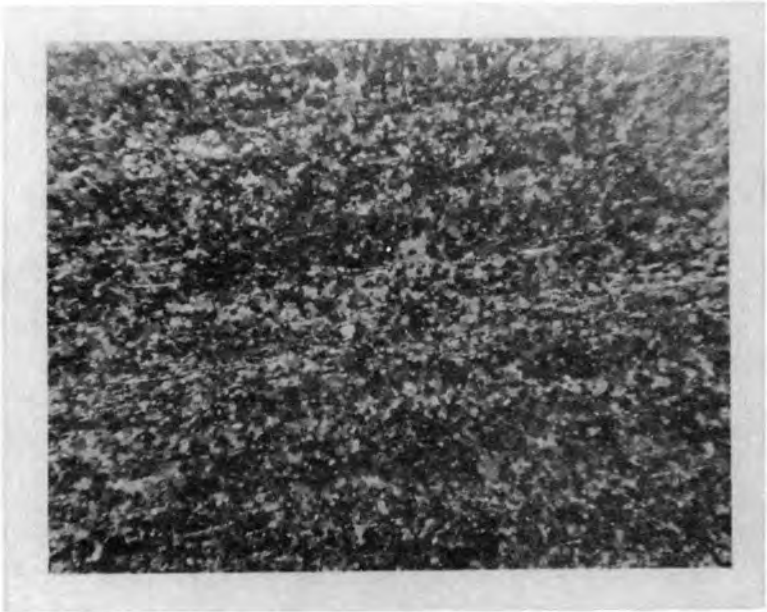

Fig. 25
Natural Uranium. Longitudinal Section. Annealed 625°C for 3 hours. Rolled from 0.181 in. to 0.132 in. at 300°C. Polarized light, X100

Y-136

Fig. 26
Natural Uranium. Same as Fig. 24 but longitudinal section.

TABLE VII

Tensile tests on normal and on enriched uranium

	WIRE LOT NUMBER	THICKNESS (in.)	REDUCTION IN AREA (percent)	TENSILE STRENGTH (psi)	FRACTURE STRESS (psi)	EXTENSION AND GAUGE LENGTH	AMOUNT OF COLD WORK SINCE LAST ANNEAL			ANNEALING TIME AND TEMPERATURE
							By Hand Rolling Mill	By Wire Drawing	Total Reduction	
NORMAL URANIUM - LOT 6	6B1	.1182 square .1170 square	1.8	153,000	154,000	3.1% in 1 in. 2.3% in 2 in.		21%	21%	
	6B1	.1175 square .1140 square	5.1	108,300	113,000 (Broke in grips)				0	2 hrs., 625°C
	6B1	.1165 square		138,000		4.7% in 2 in.			0	2 hrs., 625°C
	6B2	.1190 square .1175 square		144,800	146,000 (Broke in grips)			37%	37%	
	6B2	.1263 square		125,000				37%	37%	
NORMAL URANIUM - LOT 7	7B	.1182 square .1155 square	4.0	138,000	142,000	5.0% in 2 in.		20%	20%	
	7B	.117 square .111 square	10.	125,000	135,000	6.0% in 2 in.			0	2 hrs., 625°C
	7B	.1175 square .1142 square	5.6	125,600	131,900	6.0% in 2 in.				2 hrs., 625°C
	7A2	.1185 square .1120 square	10.7	149,500	163,500 (Broke outside gauge marks)	6.0%	35.5%	7.5%	40%	
	7A2	.1185 square .116 square	4.1	153,500	160,000	2.4% in 2 in.				
	7A1	.1188 square .1174 square	2.4	171,000	172,000	3.1% in 2 in.	54%	5%	56.5%	
	7A1	.1175 square .1065 square	10.5	120,000	144,500	27% in 2 in.			0	2 hrs., 625°C
	7A1	.118 square .110 square	12.9	160,000 112,500	128,000	12.5% in ½ in. 13.0% in 1 in. 12.0% in 2 in.			0	2 hrs., 625°C
	7A2	.1185 square .113 square	9.1	106,000 112,200	124,000	(Broke outside gauge marks)			0	2 hrs., 625°C
ENRICHED URANIUM 6.5% U^{235} - LOT 9	9-1	.1185 square .100 square	15.6	133,500	177,000	12.7% in 1.4 in. 9.0% in 2 in.		10.1%	10.1%	
	9-2	.1185 square .108 square	8.9	130,500	171,500	15.6% in 2 in. 22.5% in ½ in.		10.1%	10.1%	
	9-4	.119 square .105 square	22.2	144,000	180,000	22.0% in 1 in. 21.0% in ½ in. 20.0% in 2 in.		15.0%	15.0%	
	9-4	.119 square .111 x 113	11.3	118,000	132,000	18.75% in 1 in. 20.8% in 1½ in. 18.75% in 2 in.				

(Table continued on following page)

TABLE VII (continued)

Tensile tests on normal and on enriched uranium

ENRICHED URANIUM - 2.7% U^{235} - LOT 10

WIRE LOT NUMBER	THICKNESS (in.)	REDUCTION IN AREA (percent)	TENSILE STRENGTH (psi)	FRACTURE STRESS (psi)	EXTENSION AND GAUGE LENGTH	AMOUNT OF COLD WORK SINCE LAST ANNEAL			ANNEALING TIME AND TEMPERATURE
						By Hand Rolling Mill	By Wire Drawing	Total Reduction	
10-13	.1185 square		139,000		6.5% in ½ in.				
	.1130 square	8.9		149,000	9.4% in 1 in. 8.8% in 2 in.		10.1%	10.1%	
					(Dark spot, perhaps oxidized crack, showed up in corner of fracture)				
10-11	.1188 square		142,800		2.5% in 2 in.				
	.1150 square	6.4		150,000			10.1%	10.1%	
	(10-11 after fracture displayed longitudinal split, due to overwork)								
10-12	.119 square	9.2	130,500		9.3% in 1 in.			0	3 hrs., 625°C
	.114 square			140,500	8.3% in 1½ in.				
	(Small inside crack)								
10-21	.1185 square		137,800		18.6% in ½ in. 15.6% in 1 in.		10.1%	10.1%	
	.113 square	8.9		146,000	12.5% in 1½ in.				
	(10-21 after fracture displayed a narrow longitudinal split)								
10-22	.119 square		126,000		18.75% in 1 in.		10.1%	10.1%	
				142,400	13.4% in 1½ in.				
	.112 square	12.			13.5% in 2 in.				
10-31	.1185 square		115,500		28.0% in ½ in.				
	.1185 square		123,200		27.0% in 1 in.				
	.105 square	21.5		155,000	25.5% in 1½ in. 25.0% in 2 in.				
10-4	.119 square		174,000		9.5% in 1½ in.	0	15%	15%	
	.1145 square	7.5		182,000	6.5% in 1 in. 4.2% in 1½ in. 3.9% in 2 in.				
10-4	.119 square	24.	127,500	166,000	18.75% in ½ in. 15.9% in 1 in.				
	.101 x .1065				16.3% in 1½ in. 15.6% in 2 in.			0	3 hrs., 625°C

FIG. 27

LOAD-STRAIN CURVES-NATURAL URANIUM-ANNEALED

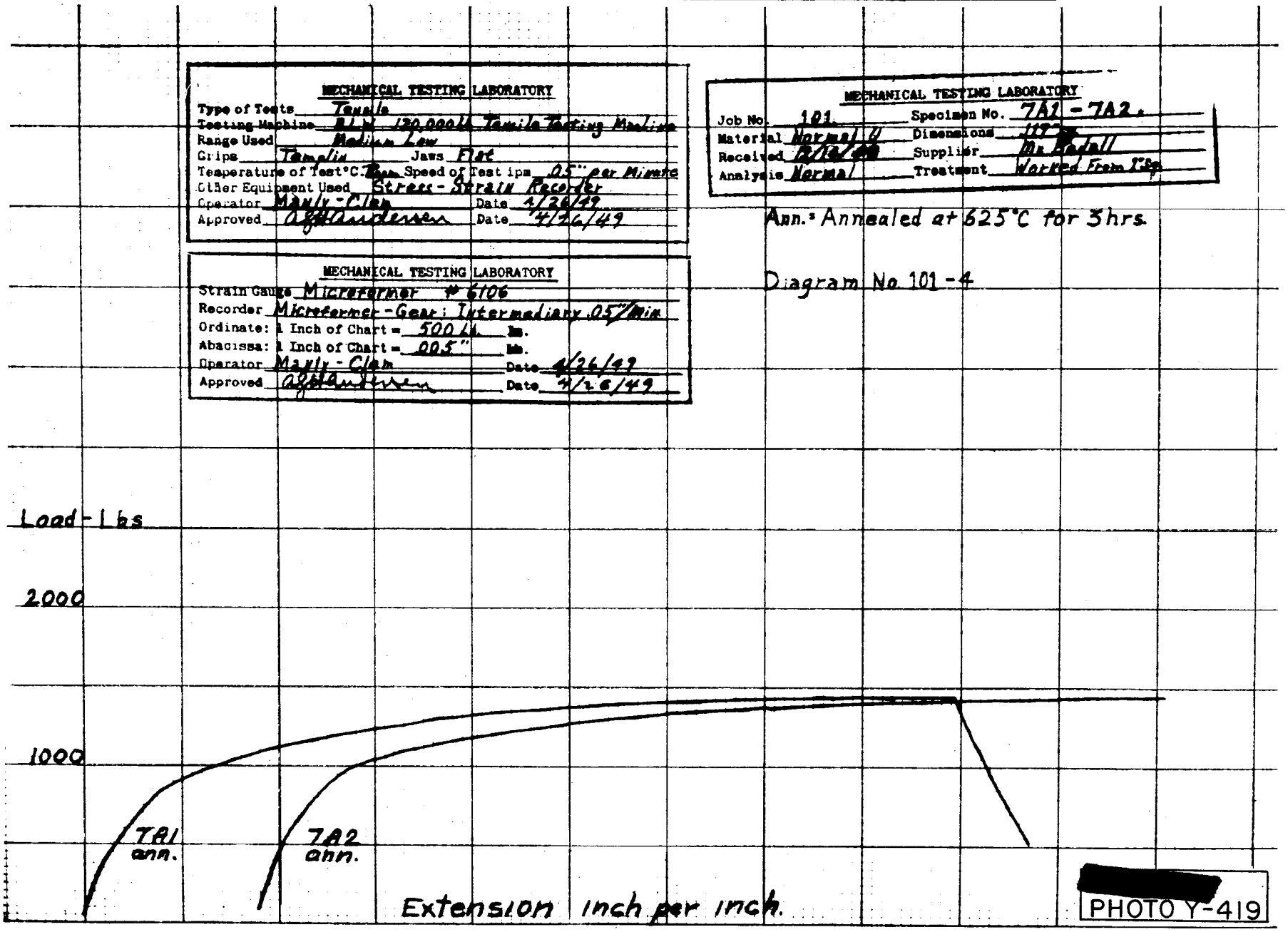

FIG. 28

LOAD-STRAIN CURVES-ENRICHED URANIUM (2.7% U^{235}) - ANNEALED

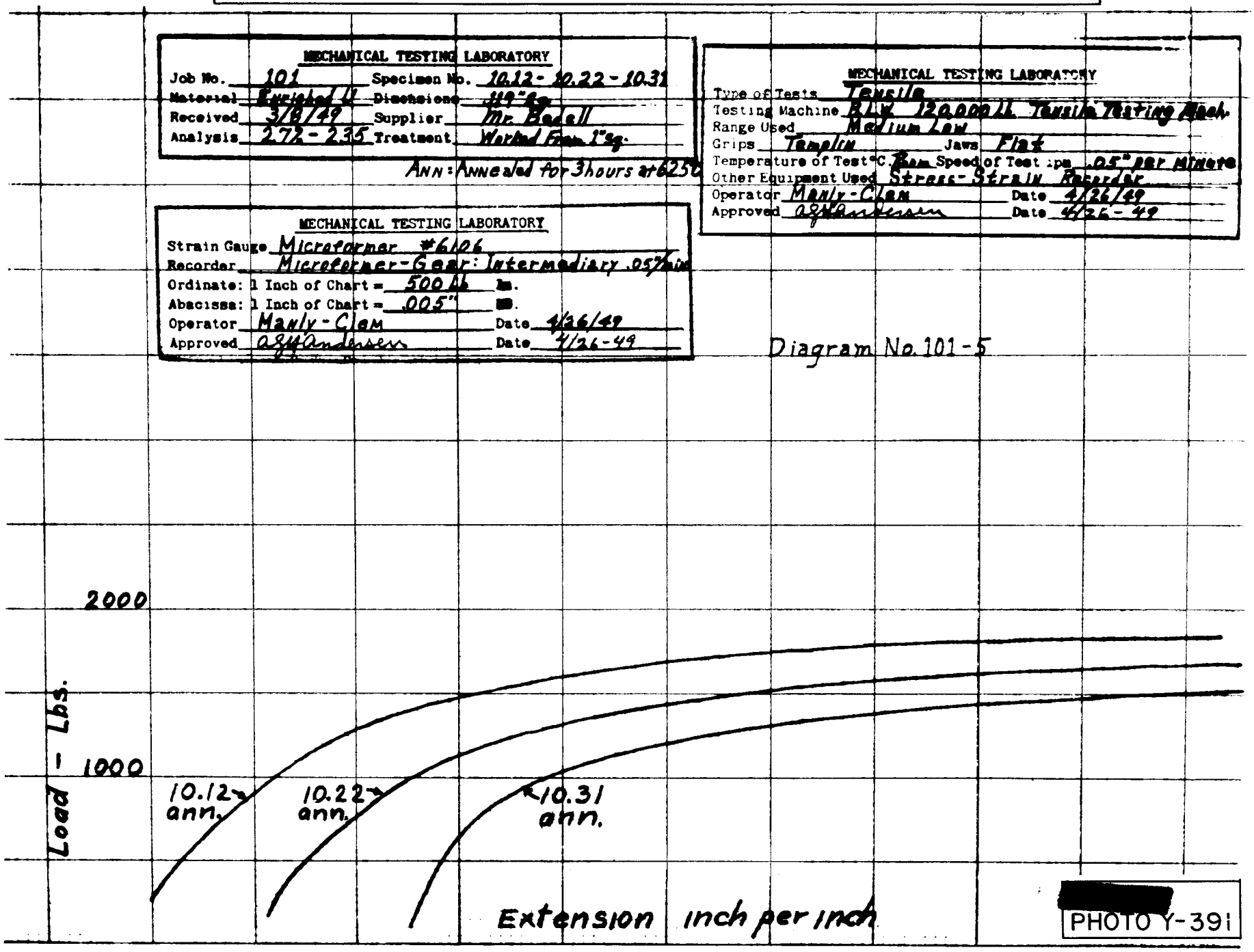

FIG. 29

LOAD-STRAIN CURVES - ENRICHED URANIUM (2.7% U^{235}) - COLD WORK

MECHANICAL TESTING LABORATORY

Job No. 101 Specimen No. 10.13 - 10.21
Material Enriched U Dimensions .119" sq.
Received 3/8/49 Supplier Mr. Bedell
Analysis 2.7% - 235 Treatment Worked From 1" sq.

MECHANICAL TESTING LABORATORY

Type of Tests Tensile
Testing Machine [redacted] 120,000 lb. Tensile Testing Machine
Range Used Medium Low
Grips Tensile Jaws Flat
Temperature of Test °C. Room Speed of Test ips .05 per minute
Other Equipment Used Stress - Strain Recorder
Operator Manly - Clem Date 4/25/49
Approved [illegible] Date 4/25/49

MECHANICAL TESTING LABORATORY

Strain Gauge Microformer #6106
Recorder Microformer - Gear: Intermediary .05/min.
Ordinate: 1 Inch of Chart = 500 lb.
Abscissa: 1 Inch of Chart = .005" in.
Operator Manly - Clem Date 4/25/49
Approved Date

Note: C.W. = 10.1% reduction at 300°C to 200°C.

Diagram No. 101-3

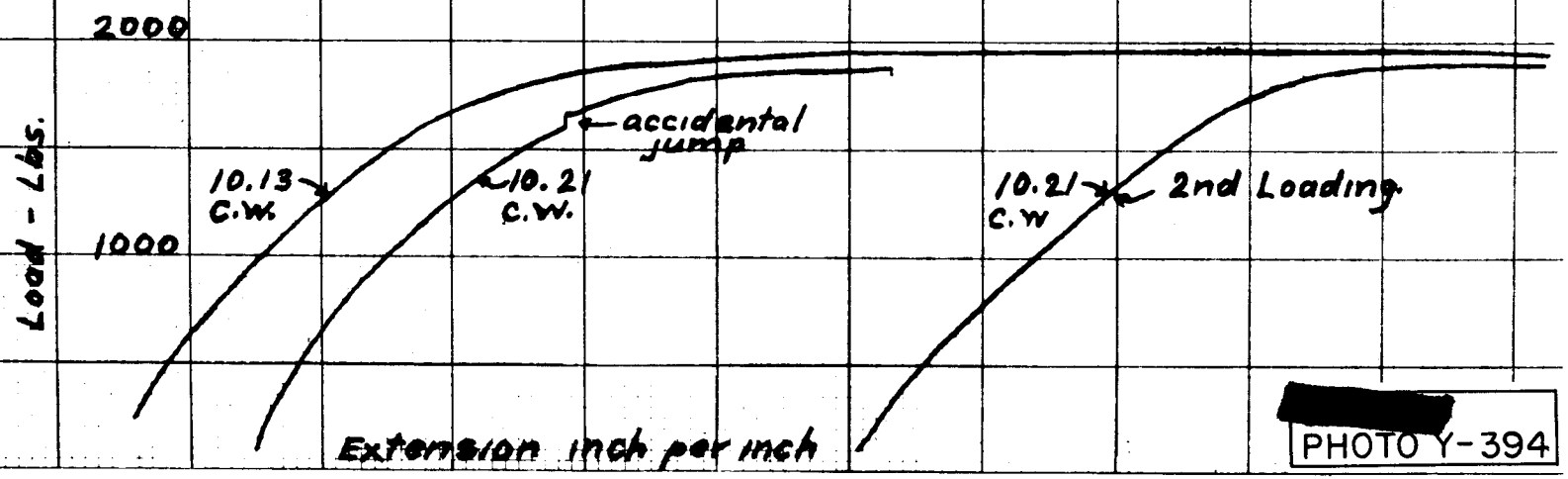

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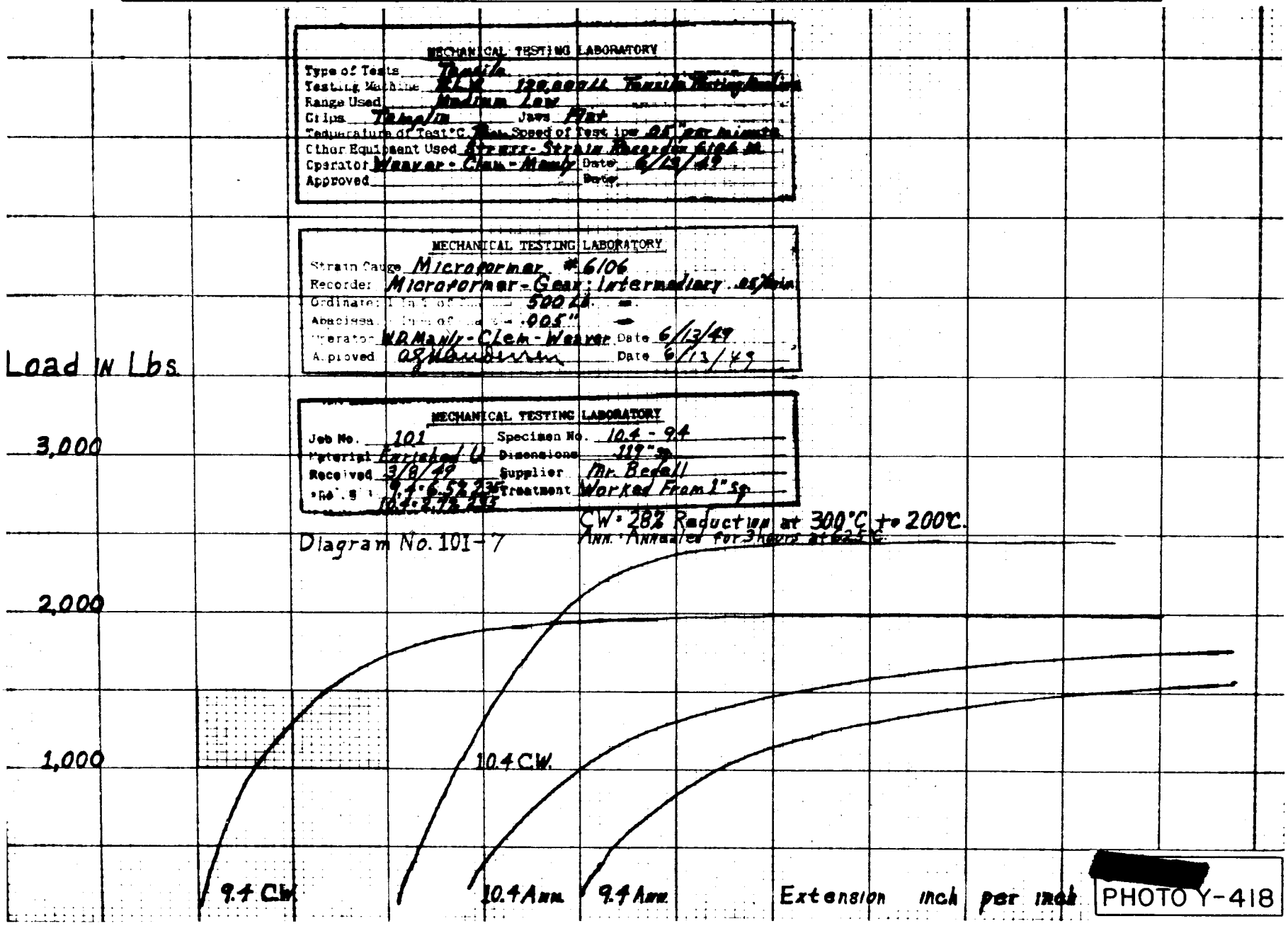
PHOTO Y-394

FIG. 30

LOAD-STRAIN CURVES-ENRICHED URANIUM (9.4-6.5% U^{235}; 10.4-2.7% U^{235})-COLD WORKED AND ANNEALED



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PERSONNEL

Table VIII lists the members of the Metallurgy Division whose work is presented in this report. It includes the organization as of July 31, 1949, and technical research personnel who have terminated employment with the Laboratory since the previous progress report.

Table VIII

Metallurgy Division Organization

J. H. FRYE, *Director*

Roberta Hudson, *Secretary*

L. E. Banker, *Administrative Assistant*

Helen Carr, *Secretary*
Delores Poe, *Librarian*
Ruby Bullard, *Secretary*
Doris Jacox, *Stenographer*

		TECHNICIANS
A. G. H. Anderson	*Fundamental Research: Beryllium, Thorium; Pure Metals*	
W. H. Bridges	*Purification of Beryllium*	W. C. Weaver
W. D. Manly	*Physical Metallurgy: Thorium; Beryllium; Mechanical Testing*	R. T. Clem
J. C. Wilson	*Mechanical Properties of Pure Metals*	
E. J. Boyle	*Beryllium Production, ORNL and Hanford Slug Problems, Thorium Alloy Development*	
G. H. Boss	*Hanford Slug Problem*	
D. E. Hamby	*Thorium Alloy Development; Melting and Casting*	E. E. Layne
J. H. Erwin	*Hanford Slug Problem; Metal Working and Heat Treatment*	G. D. Goldston J. D. Hutchins D. E. Rossen J. P. Turner

[redacted]

R. O. Williams	*ORNL Slug Problem; Thorium Alloy Development*	C. L. Smith
R. J. Gray	*Metallography*	E. R. Boyd E. P. Griggs
R. S. Crouse	*Metallography*	B. C. Leslie
L. K. Jetter	*Fundamental Physico-Metallurgical Research*	
G. T. Murray	*Fundamental Physico-Metallurgical Research*	
F. Kerze, Jr.	*Reactor Development*	
C. D. Smith	*Melting, Rolling, Cladding*	C. F. Cutcher
H. J. Wallace	*Melting, Rolling, Cladding*	F. M. Blacksher
F. W. Drosten	*Brazing, Fabrication*	G. E. Cooley J. N. Hix
J. E. Cunningham	*Beryllium Fabrication; Thorium Procurement*	T. W. Fulton
G. M. Adamson	*Beryllium Fabrication; Thorium Procurement*	
J. T. Howe	*Metallurgical Inspection and Design; Maintenance*	
E. C. Miller	*Extrusion of Beryllium*	

ACTIVE CONSULTANTS

J. L. Gregg	*Metallurgical Coordinator of Beryllium for the Materials Testing Reactor*
E. E. Stansbury	*General Consultation*

GRADUATE STUDENTS (Summer Thesis Research)

M. L. Pickelsimer	*University of Tennessee*
T. M. Kegley	*University of Alabama*

[redacted]

TERMINATIONS (Since ORNL 69)

W. A. Johnson

W. S. Pellini

J. A. Kies

D. K. Stevens

D. S. Billington

J. E. Draley

R. B. Goodrich

[redacted]