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DEVELOPMENT OF TECHNIQUES FOR ROLLING URANIUM METAL

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Work Done By:

D. E. Deutsch
G. S. Hanks
G. E. Jaynes
R. E. Anderson
V. Vigil
R. Osborn
G. Sweeney

Report Written By:

D. E. Deutsch
G. S. Hanks
J. M. Taub
D. T. Doll

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DATE MAR 26 1957
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W. D. Claus
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DEVELOPMENT OF TECHNIQUES FOR ROLLING URANIUM METAL

ABSTRACT

Uranium can be rolled from cast metal or forged ingot to sheet satisfactory for cupping, deep drawing, and similar fabrication procedures by a combination of hot breakdown in the neighborhood of 600°C and warm finishing at 225° to 325°C. Sheet may also be obtained by hot rolling alone and by warm rolling alone. However, it is difficult to secure a uniform, controlled grain size by hot rolling only and warm rolling is time consuming because of the limited reduction per pass obtainable. The combination of hot and warm rolling afforded the best and most practical method to secure good quality sheet in the quantity required.

The percent reduction by hot working does not appear to be critical, but at least 60 percent warm reduction is desirable to obtain complete and controlled grain size by recrystallization with high ductility and strength properties.

Except for research investigation, rolling of uranium below 225°C is not recommended. In the temperature range considered (below 225°C), reduction is difficult and must be limited to one to two percent if edge cracking is to be avoided.

Hot rolling of unplated uranium from the as-cast or as-forged surface is recommended, using a bath of 35 percent Li_2CO_3 plus 65 percent K_2CO_3 for a heating medium. No further preparation other than

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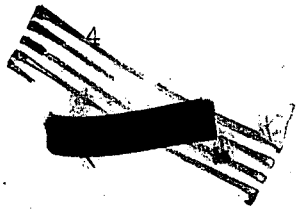
washing the salt from the hot rolled surface is required before warm rolling, and a bath of Meltemp No. 7 oil is recommended for warm rolling.

Work roll speeds and temperatures are not critical, and moderate to light reductions per pass are recommended for easiest rolling and best results. Alternate cross-rolling is recommended during hot breakdown, but continuous rolling in one direction during warm finishing is satisfactory.

Starting with an as-cast tensile strength averaging 60,000 psi, as-rolled sheet up to 230,000 psi has been thus produced, with elongation averaging 5-8 percent and tensile to yield strength ratios averaging 75 percent.

Uniform, equi-axed grains are produced upon annealing, and grain sizes can be controlled by choice of annealing temperature and time.



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INTRODUCTION

The present report covers work thus far completed in an investigation of procedures by which uranium metal can be rolled to plate or sheet. Additional work remains in respect to examination of the variables concerned in the rolling process. However, at the present time, a method has been established by which sheet possessing good surface and mechanical properties can be rolled from cast metal or from forged ingot.

In certain cases, it is recognized that additional data are required to substantiate present information. The behavior of the metal when undergoing deformation has presented some results not fully understood and further examination of the processes is required.

A study of casting procedures with a view towards improvement of the metal structure in the ingot to be rolled should be made. Likewise, the effects which impurities may impart to the finished sheet should be investigated.

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The major purpose of the rolling program was to investigate methods and techniques by which uranium sheet possessing good forming characteristics might best be obtained.

Metals Used

The uranium metal used in the program thus far was of two types:

1. Normal uranium - the normal mixture of the isotopes of uranium metal, known as tuballoy, containing approximately 630 parts per million of carbon. A typical spectrographic analysis is as follows:

Element	Parts Per Million
Carbon	630
Iron	80
Silicon	70
Calcium	10
Copper	7
Aluminum	5
Chromium	3
Magnesium	3
Manganese	2
Sodium	2
Boron	0.1
Vanadium	<10

Element	Parts Per Million (Cont'd)
Nickel	< 5
Cobalt	< 5 (not detected)
Lithium	< 0.1 (not detected)
Beryllium	< 0.1 (not detected)

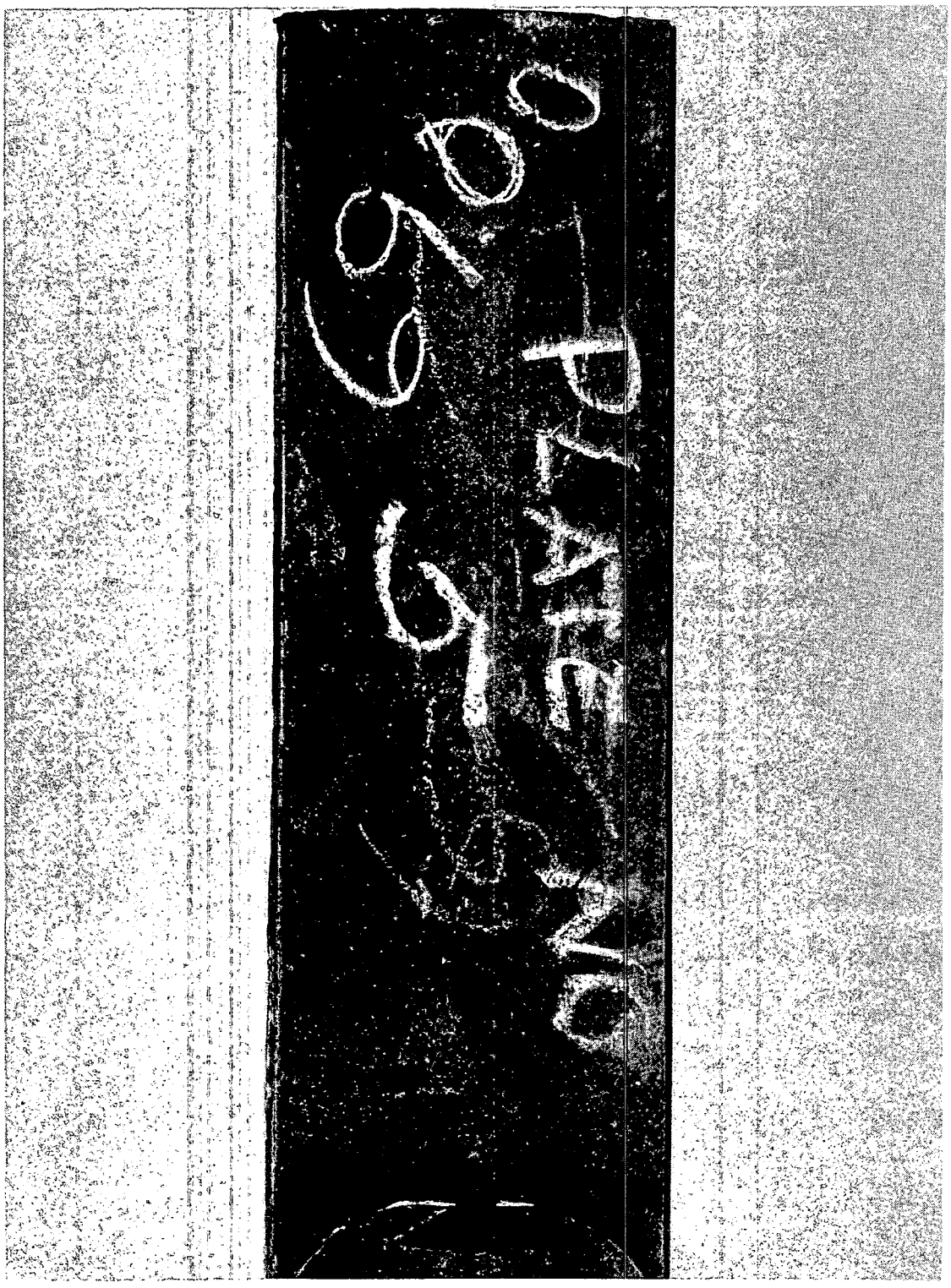
The presence of columbium, which may or may not be of importance, was not reported.

2. High purity uranium - the normal mixture of uranium isotopes, in which the carbon content is kept low (around 100 ppm, as compared to 630 ppm in normal U). Other elements are present in approximately the same amounts as in the normal uranium.

For the first series of experiments, the metal was cast into plates (see Figure 1), whose dimensions after cropping were 18 by 6 by 3/4 inches. The method of casting these ingot plates was as follows:

Scrap metal was heated in a graphite crucible to 1325°C and held in the liquid state at temperature for one-half hour under a vacuum ranging from 500 to 1000 microns in order to eliminate dissolved gasses. A graphite pouring rod which closed the pouring gate in the bottom center of the crucible, was then mechanically raised and the molten tuballoy cast into a graphite mold situated directly under the crucible. The graphite molds were coated on the inside with MgO and

FIG. 1 - PHOTOGRAPH OF AN AS-CAST URANIUM INGOT, DIMENSIONS 18 BY 6 BY 3/4 INCHES (AFTER CROPPING). MARKINGS ON PLATE ARE FOR IDENTIFICATION.

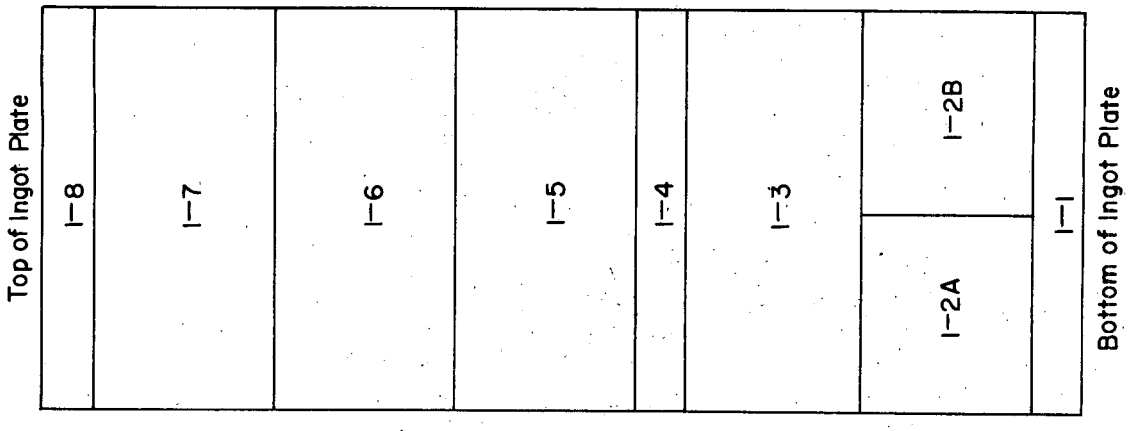


were preheated to an average temperature of 900°C by casting time, the top of the mold being at approximately 1100°C and the bottom around 700°C.

The melting and casting of the metal was performed in an evacuated electric induction furnace. After pouring, the metal was cooled slowly in the furnace to 300° or 400°C (or down to room temperature if cooling took place overnight). The furnace was opened and the mold quenched in water, after which the casting was extracted.

For casting plates, the graphite mold was made in two halves held together by a circular sleeve. The mold could be opened, the casting extracted, and the mold reused for subsequent castings.

In order to evaluate the properties of sheet rolled from different portions of an ingot plate, each plate was sectioned by sawing, as indicated in Figure 2 and the sections numbered for identification purposes as shown in the sketch below.



Numbering of Cast Plate No. 1

Sections of the plate were numbered consecutively from the

FIG. 2 - AS-CAST URANIUM INGOT SHOWN AFTER SECTIONING INTO LENGTHS FOR USE AS ROLLING STOCK WITH 0.505 INCH TENSILE SPECIMENS ALREADY MACHINED FROM TOP, MIDDLE, AND BOTTOM SECTIONS. CROP IS SHOWN AT LEFT.

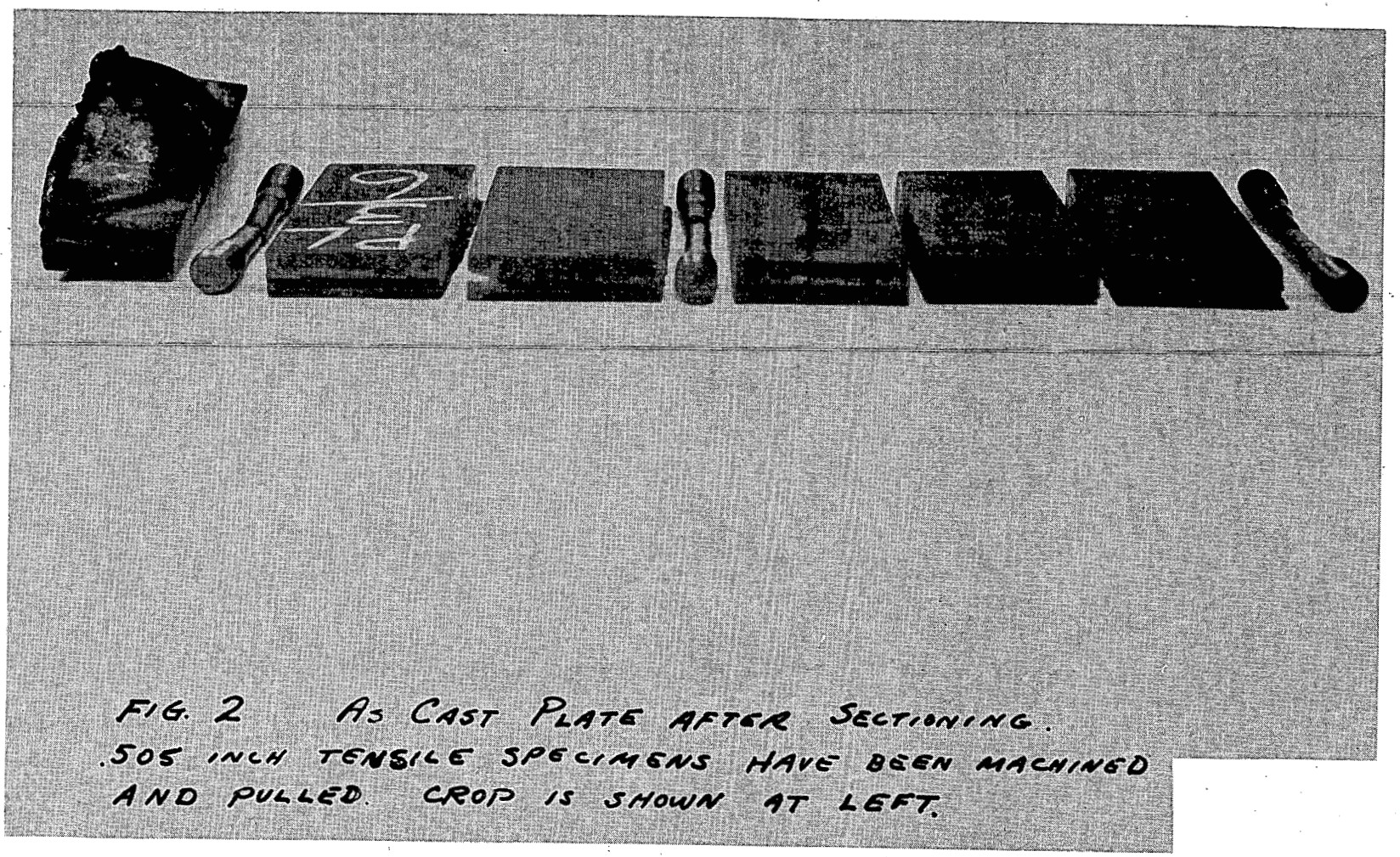


FIG. 2 AS CAST PLATE AFTER SECTIONING. .505 INCH TENSILE SPECIMENS HAVE BEEN MACHINED AND PULLED. CROP IS SHOWN AT LEFT.

bottom of the casting to the top. Where only half of one section was used, such as 1/2 of section 1-2, the halves were numbered "A" and "B", viz., 1-2A and 1-2B.

The As-Cast Plate

Before rolling results could be evaluated, it was necessary to know the properties of the as-cast metal used for rolling stock. Metallographic studies were made, tensile specimens were pulled, and all castings were radiographed to check for soundness. Only sound castings were rolled; unsound castings were rejected.

Figures 3, 4 and 5 are photomacrographs taken after machining as-cast Plate No. 2 to a smooth surface, and etching in a solution of nitric acid. These pictures show the typically coarse as-cast grain structure on the flat sides and on the cross section of the plate. All the plates used for rolling showed similar as-cast grain structure.

Figures 6 and 7 show some typical photomicrographs of the as-cast grain structure of the normal and high purity tuballoy used.

It can be seen that the grain structure for both the normal and high purity tuballoy is quite similar. The distribution of carbides is also fairly similar, but a significant difference in the size of the carbides can be noticed.

As might be expected, the carbides in the high purity sample, which contains a much lower carbon content than normal tuballoy, are smaller in size.

FIG. 3 - PHOTOMACROGRAPH SHOWING AS-CAST GRAIN STRUCTURE ON FLAT SIDE OF NORMAL URANIUM INGOT PLATE AT 2X.

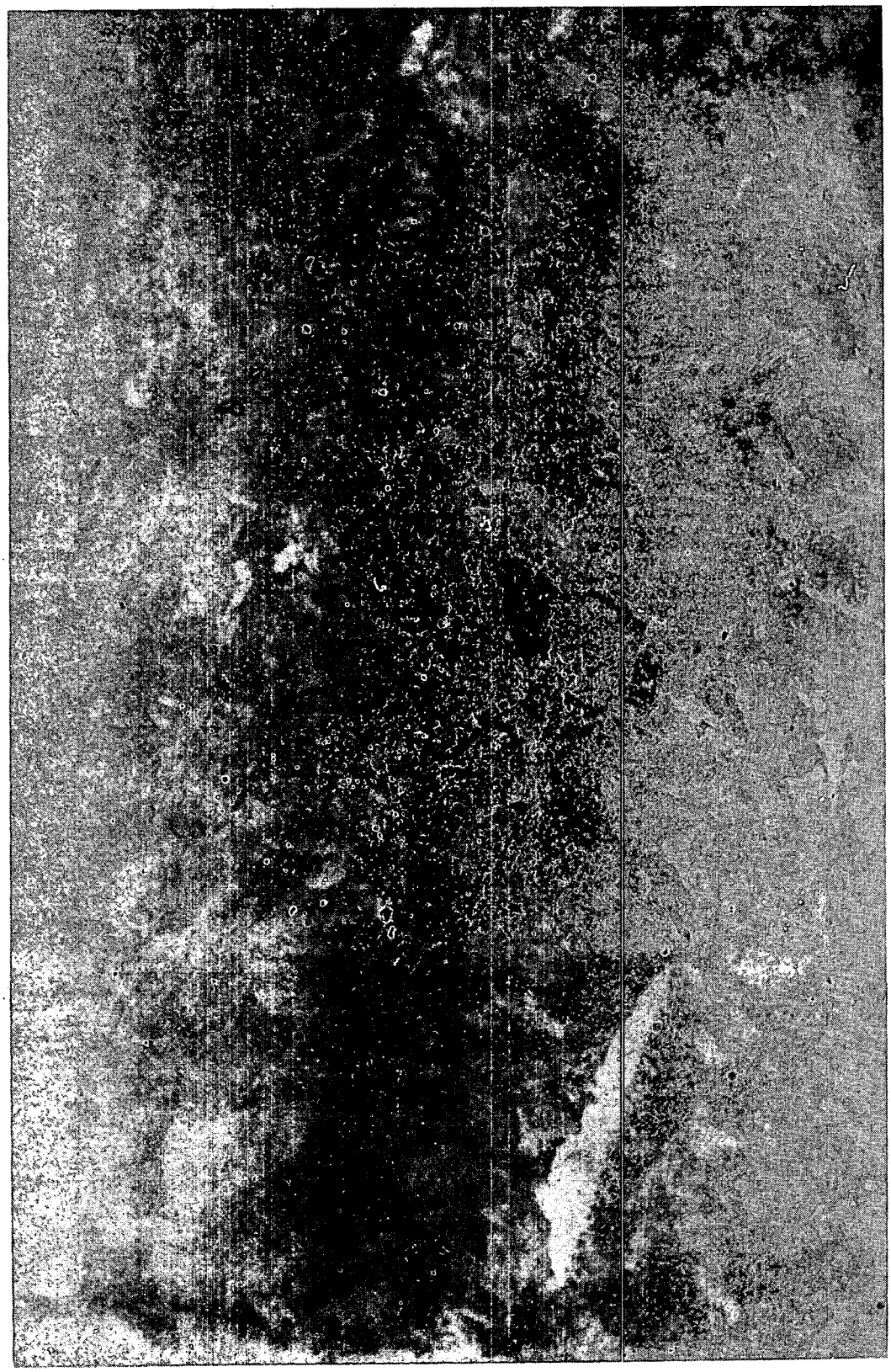


FIG. 4 - PHOTOMACROGRAPH SHOWING AS-CAST GRAIN STRUCTURE ON FLAT SIDE OF A NORMAL URANIUM PLATE.

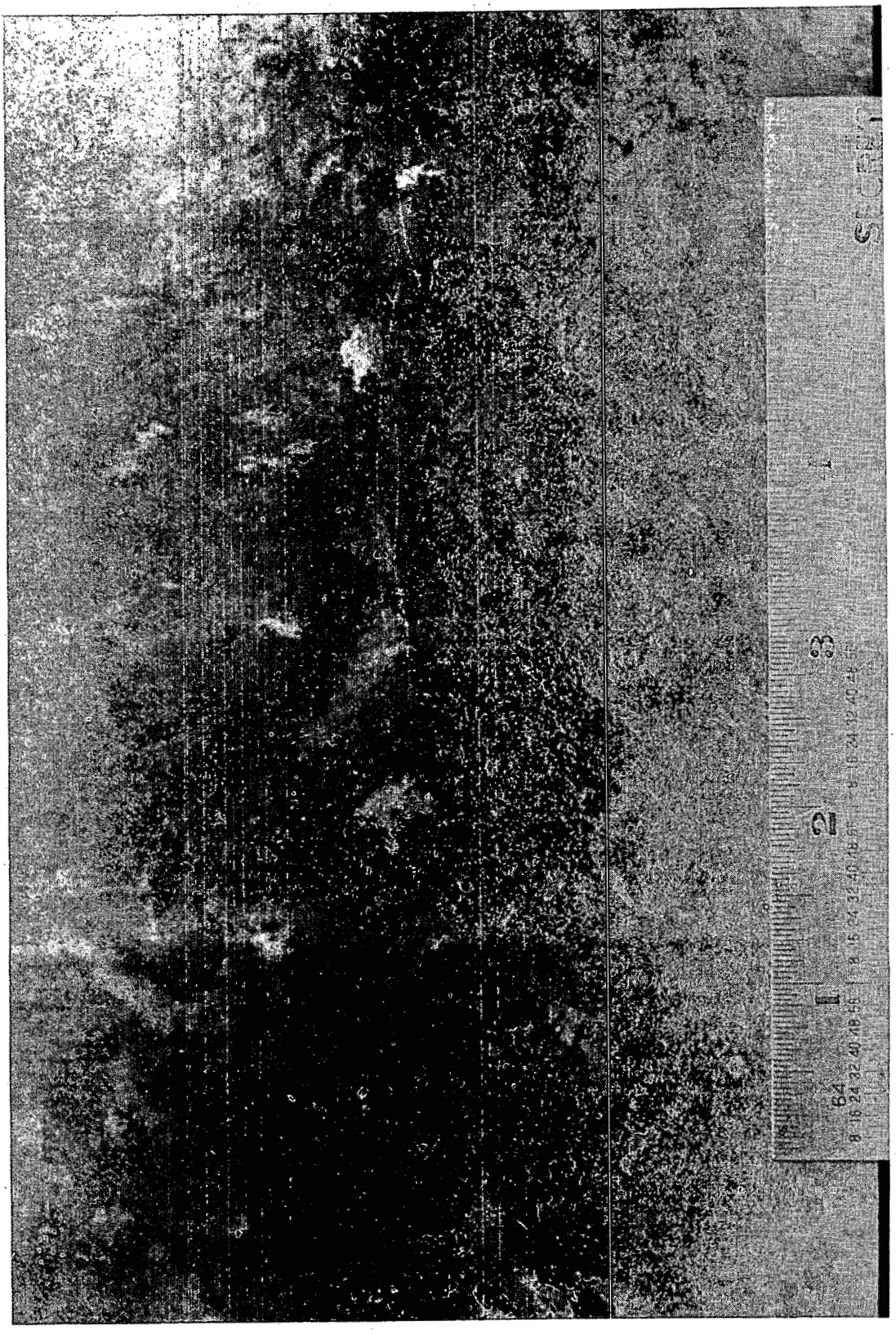
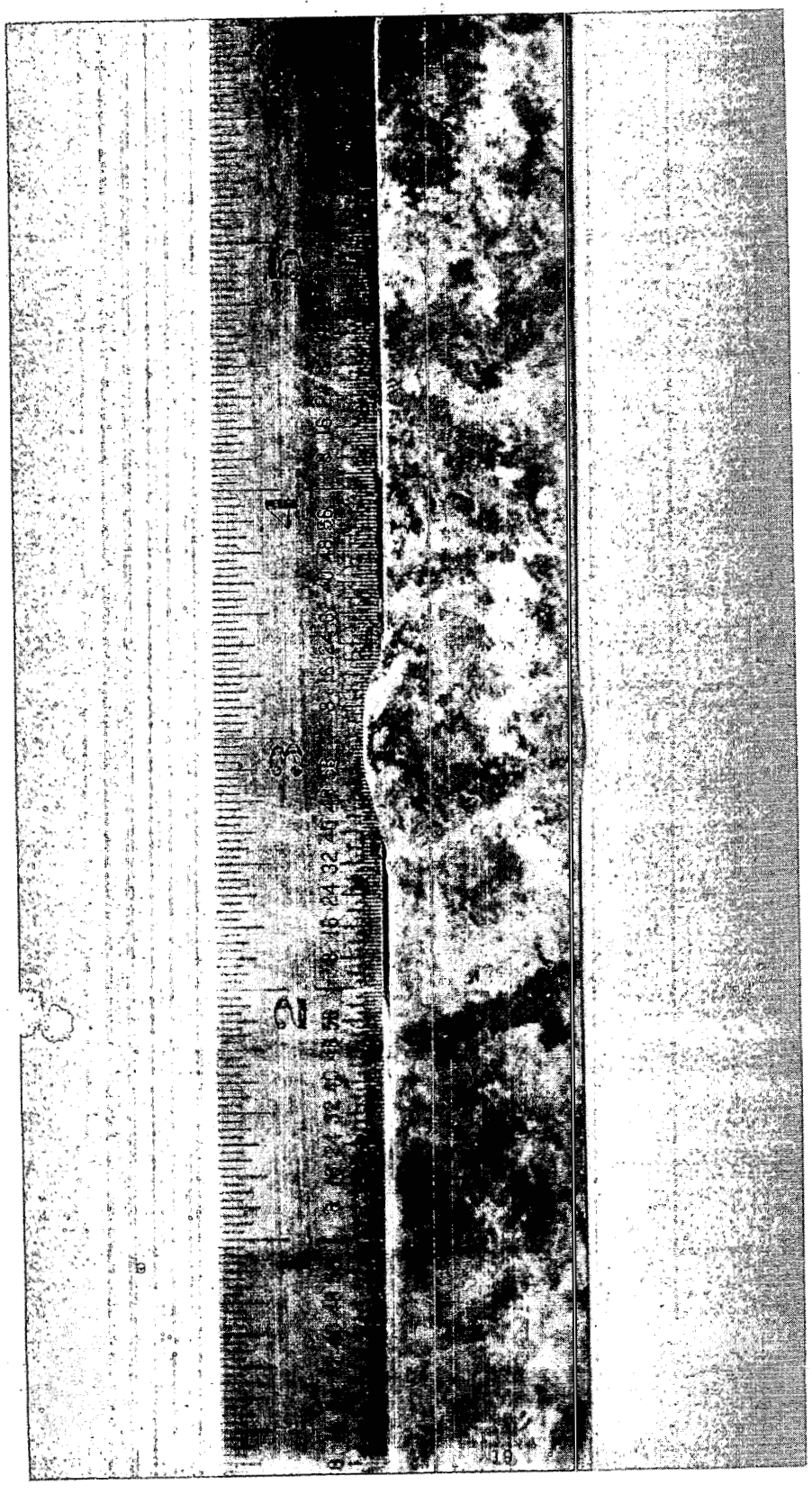


FIG. 5 - PHOTOMACROGRAPH SHOWING AS-CAST GRAIN STRUCTURE ON CROSS SECTION OF THE TOP OF A CAST INGOT OF NORMAL URANIUM. MAGNIFICATION APPROXIMATELY 2X.



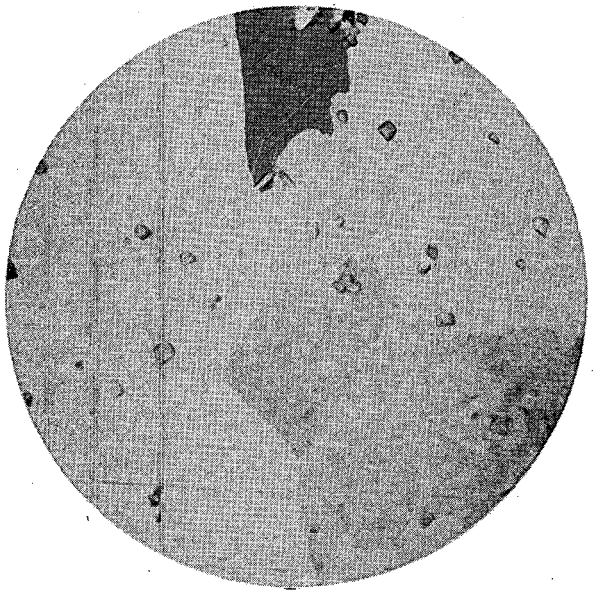


PLATE NO. J131-2-1

MAGNIFICATION: 250X
NO ETCH; POLARIZED LIGHT.

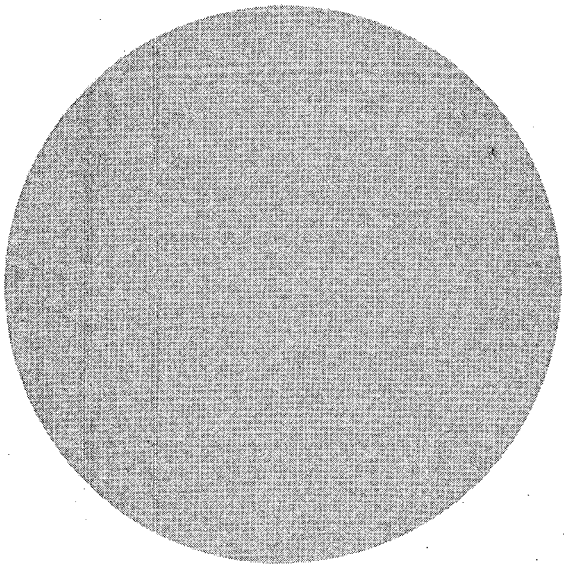


PLATE NO. J131-2-2

MAGNIFICATION: 100X
NO ETCH; BRIGHT FIELD.

FIG. 6 - AS-CAST GRAIN STRUCTURE AND CARBIDE DISTRIBUTION OF NORMAL URANIUM METAL.

PLATE NO. J131-1-2 MAGNIFICATION: 250X
NO ETCH; POLARIZED LIGHT.

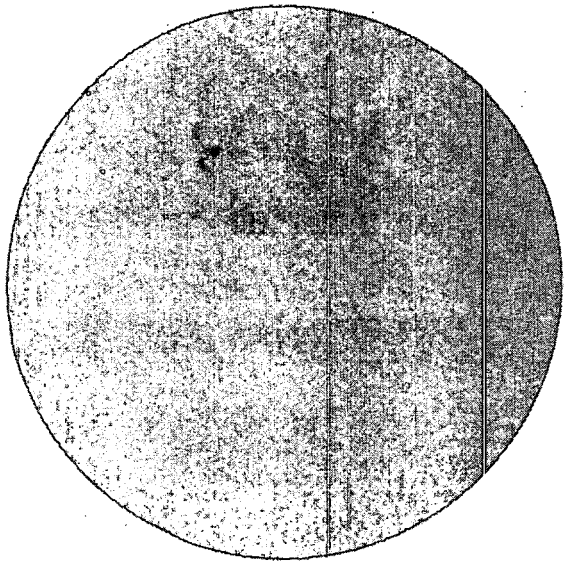


PLATE NO. J131-1-1 MAGNIFICATION: 100X
NO ETCH; BRIGHT FIELD.

FIG. 7 - PHOTOMICROGRAPHS OF AS-CAST STRUCTURE AND CARBIDE DISTRIBUTION OF TYPICAL HIGH PURITY URANIUM METAL.

A series of photomicrographs was taken of the bottom, middle and top regions of a normal uranium ingot plate to determine if there was any significant variation in grain structure, carbide distribution or amount and distribution of inclusions and porosity at these locations. The photograph of Figure 8 shows the ingot plate (Ingot Plate No. 1) to be fairly homogeneous from top to bottom. The tensile data listed below also shows absence of any significant variation in properties due to position in the ingot.

Sections 1, 5 and 8 of each ingot plate were machined to provide 0.505 inch tensile specimens to determine if any variation in the as-cast tensile properties occurred relative to section position in each casting. Table I presents data from tensile tests made on bars taken from a number of normal and high purity uranium plates.

TABLE I

Tensile Data for Normal and High Purity Cast Uranium

Spec. No.	Composition	Tensile Strength, psi	Yield Strength 0.2% Offset, psi	% Elongation 2" Gauge	Ratio Y/T x 100	Modulus of Elas. x 10^6
1-1	Normal	64,200	26,500	17.2	0.40	26
1-5	"	88,800	36,700	13.3	0.41	27
1-8	"	72,000	20,800	19.5	0.29	20
3-1	"	67,500	36,900	6.3	0.55	--
3-5	"	78,500	22,100	19.0	0.28	25.4
3-8	"	70,500	26,600	8.5	0.38	24

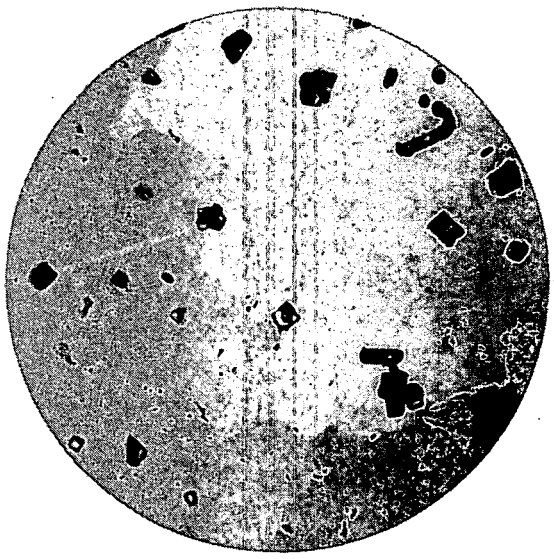


PLATE NO. J86-1-1
BOTTOM OF INGOT PLATE

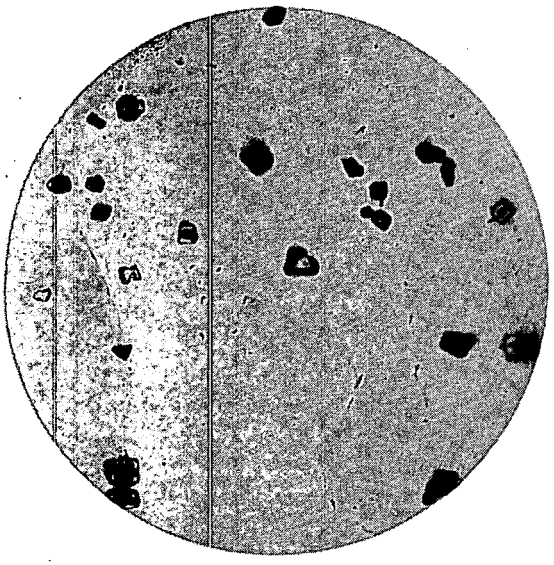


PLATE NO. J86-1-2
BOTTOM OF INGOT PLATE

PLATE NO. J86-2-1
MIDDLE OF INGOT PLATE

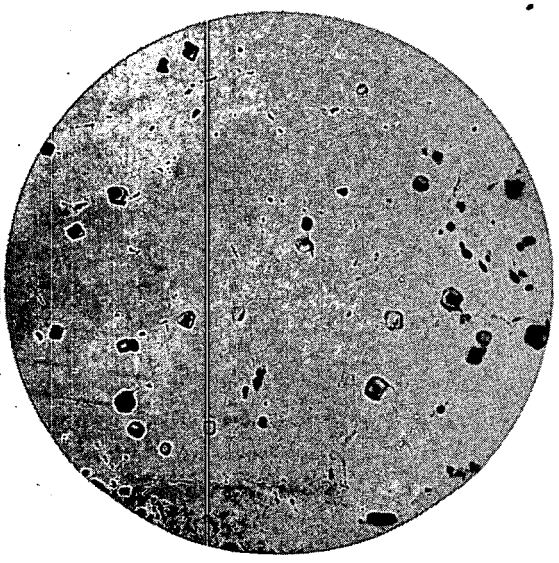


PLATE NO. J86-3-1
TOP OF INGOT PLATE

FIG. 8 - PHOTOMICROGRAPHS OF THE AS-CAST STRUCTURE OF NORMAL URANIUM METAL FROM BOTTOM, MIDDLE AND TOP OF AN INGOT PLATE. BRIGHT FIELD, STANDARD URANIUM ETCH. MAGNIFICATION 250X.

Tensile Data For Normal and High Purity Cast Uranium (Cont'd)

Spec. No.	Composition	Tensile Strength, psi	Yield Strength 0.2% Offset, psi	% Elongation 2" Gauge	Ratio Y/T x 100	Modulus of Elas. x 10^6
4-1	Normal	65,000	30,500	7.5	0.48	23.3
4-5	"	78,500	21,200	16.0	0.27	19.5
4-8	"	67,500	33,100	-	0.49	25
5-1	"	70,000	33,200	7.5	0.47	27
5-5	"	71,400	20,300	-	0.28	21.1
5-8	"	79,200	20,100	14.0	0.25	22.8
8-1	High Purity	53,000	21,600	7.5	0.41	24
9-1	" "	58,000	19,600	10.0	0.39	28
9-5	" "	45,600	16,800	8.5	0.42	19
9-8	" "	53,700	23,200	6.5	0.43	23

Hardness readings were also taken on all as-cast plates and sections prior to rolling. The as-cast hardness of both normal and high purity tuballoy was found consistently to be in the neighborhood of 57 R_A.

Equipment Used

4-High Standard Mill

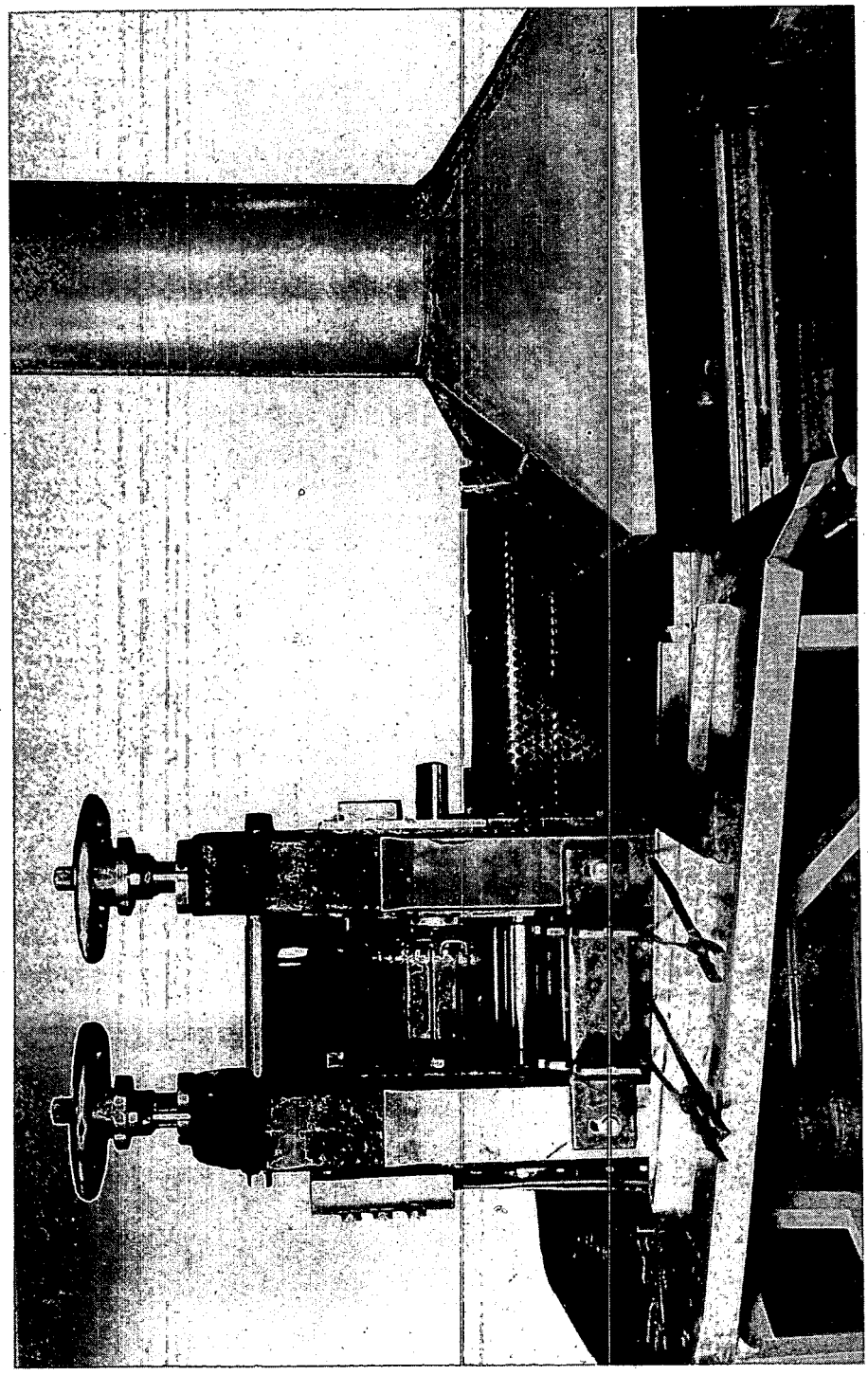
For the first series of experiments in which uranium was warm rolled at temperatures below its recrystallization range, a 4-High Standard rolling mill (Figure 9) was used. This mill has work rolls 2.5 inches in diameter and 8 inches long with backing-up rolls 8 inches in diameter and the same length as the work rolls. The mill is driven by a 30 horsepower, AC motor giving a constant speed of 62 RPM on the work rolls and a constant delivery speed of 40.5 feet/minute. The mill could be run both forward and in reverse.

A reversible mill is desirable because specimens would occasionally tear while passing through the rolls, or even stall the mill on very heavy reductions. The mill could then be reversed and the piece backed out with no difficulty.

Initially, no provision was made for heating the work rolls. However, experience using another mill having heated rolls indicated that heated rolls gave sheet with improved surface quality. It was decided to heat the rolls on the 4-High Standard mill. To this end, electric strip heaters were installed along the exposed circumference of the top and bottom back-up rolls (see Figure 9), enabling work roll temperatures up to 100°C to be attained.

The rolls were raised and lowered by hand using two hand wheels on two screws connected to the top back-up roll. The top work

FIG. 9 - 4-HIGH STANDARD ROLLING MILL



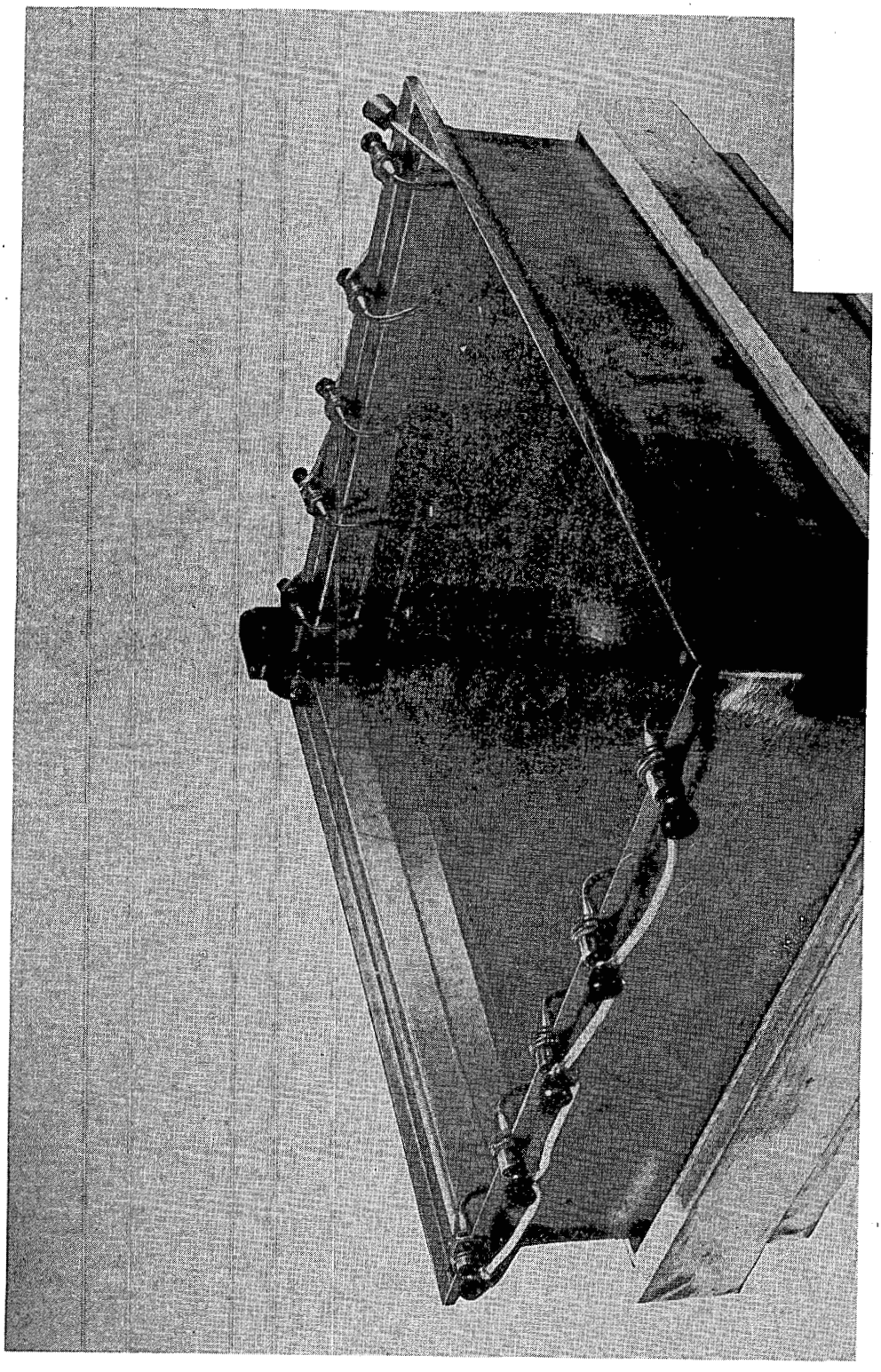
roll is held snug up against the top back-up roll by means of two springs. The bottom work roll and back-up roll are stationary. This arrangement was satisfactory on this small mill.

In conjunction with the 4-High Standard mill, a stainless steel tank, in which Calrod immersion heaters were placed, was used as a bath to heat the metal preparatory to rolling, Figure 10. The temperature of the bath was controlled by a De Khotinsky Thermo-Regulator. DC 550 silicone oil was first used as a heating medium. This material was later replaced by Meltemp No. 7, a high flash-point oil suggested by the Argonne Laboratories and marketed by Metal Lubricants Company of Chicago. The metal being heated rested on a stainless steel screen. The screen was placed immediately above the immersed Calrod heaters to prevent contact between the metal and heaters, which contact would result in local overheating. A hood, as shown in Figure 9, was connected to an exhaust fan and placed over the tank to draw off the fumes present when either DC 550 or Meltemp No. 7 was brought up to temperature.

Ordinary blacksmith tongs were used to remove the metal from the heating bath and place it on the rolling table prior to delivery into the rolls.

On several occasions, the uranium metal caught on fire while passing through the rolls. This occurred when a fin or leading edge caught on the roll table or mill housing. For this contingency, a can of G-1 powder was placed convenient to the rolling mill and used to smother the flames.

FIG. 10 - BATH FOR HEATING SPECIMENS IN OIL. USED IN CONJUNCTION WITH 4-HIGH STANDARD MILL.



2-High Bliss Mill

For hot breakdown of the as-cast plates at high alpha temperatures above the recrystallization range (500-625°C), a 2-High Bliss mill was used, Figure 11. This mill has work rolls 8 inches in diameter and 20 inches long. The mill is powered by a 25 horsepower DC motor. It was possible to vary roll speeds by means of a rheostat control. A tachometer was attached to the drive shaft to measure roll speeds directly in RPM. The mill was reversible and also had forward and reverse jog control, though this latter was found to be unnecessary. The work rolls on this mill were heated by rows of infra-red heat lamps placed in two semi-circles along the top half of the top work roll and along the bottom half of the bottom work roll, Figure 11. Roll temperatures up to 100°C could thus be obtained.

The rolls were initially raised and lowered by hand using the large hand wheel seen on the right side of the mill, Figure 11. Since this was a tedious operation, a one-half horsepower motor was geared to the hand wheel so that the rolls could be raised and lowered mechanically.

An electric resistance pot furnace, shown in Figures 11 and 12, having a cast iron pot 6 inches in diameter and 14 inches deep, was used to preheat the metal for rolling. A chromel-alumel thermocouple placed outside the furnace pot approximately one inch from the heating elements was connected to the controller seen on the top of the stand. A second chromel-alumel thermocouple, using a nickel protection tube closed at

FIG. 11 - 2-HIGH BLISS ROLLING MILL. THE STRIPED DISC VISIBLE IS PART OF EQUIPMENT SITUATED IN THE BACKGROUND.

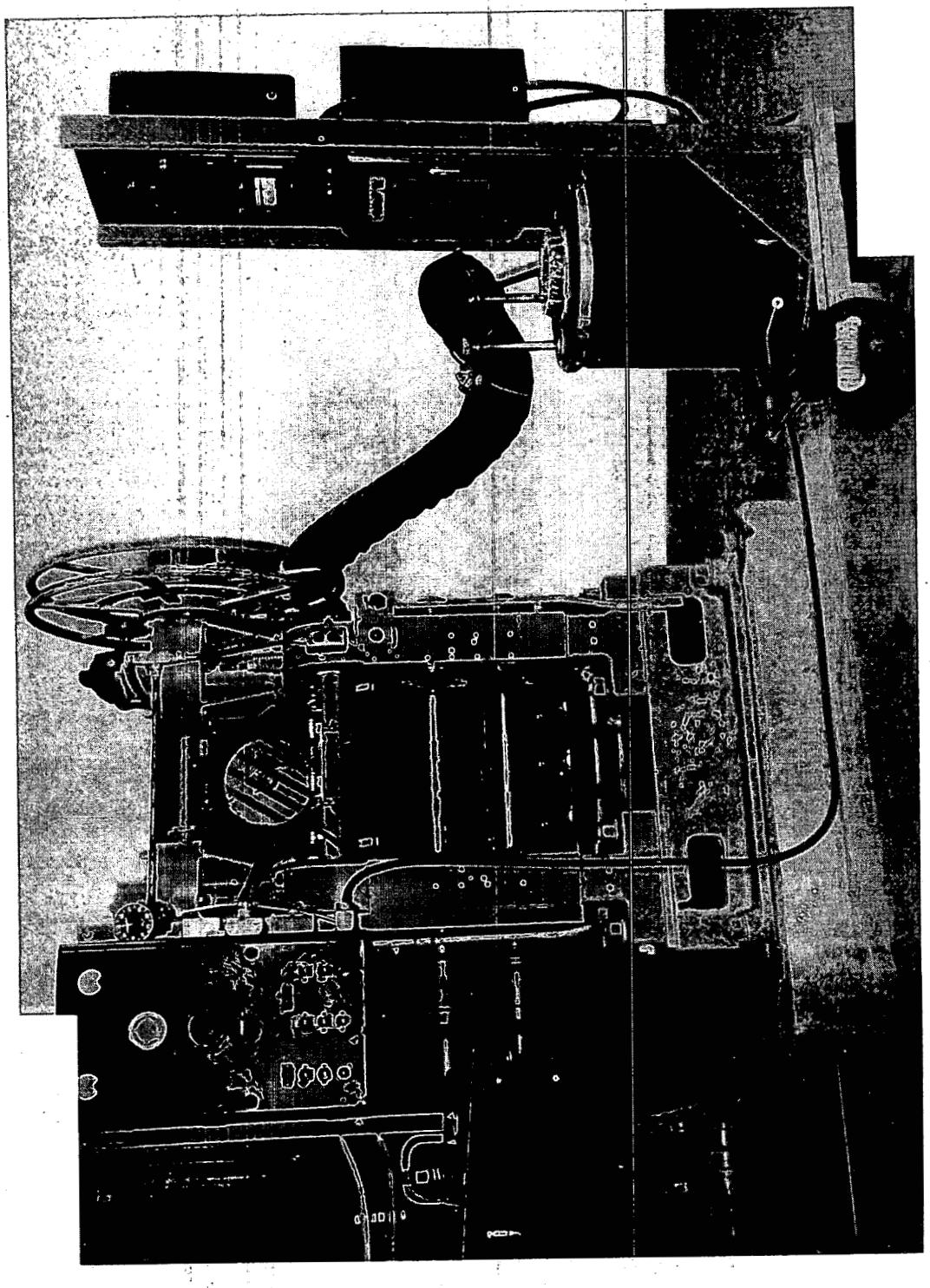


FIG. 12 - ELECTRIC POT FURNACE USED FOR HEATING SALT BATH.

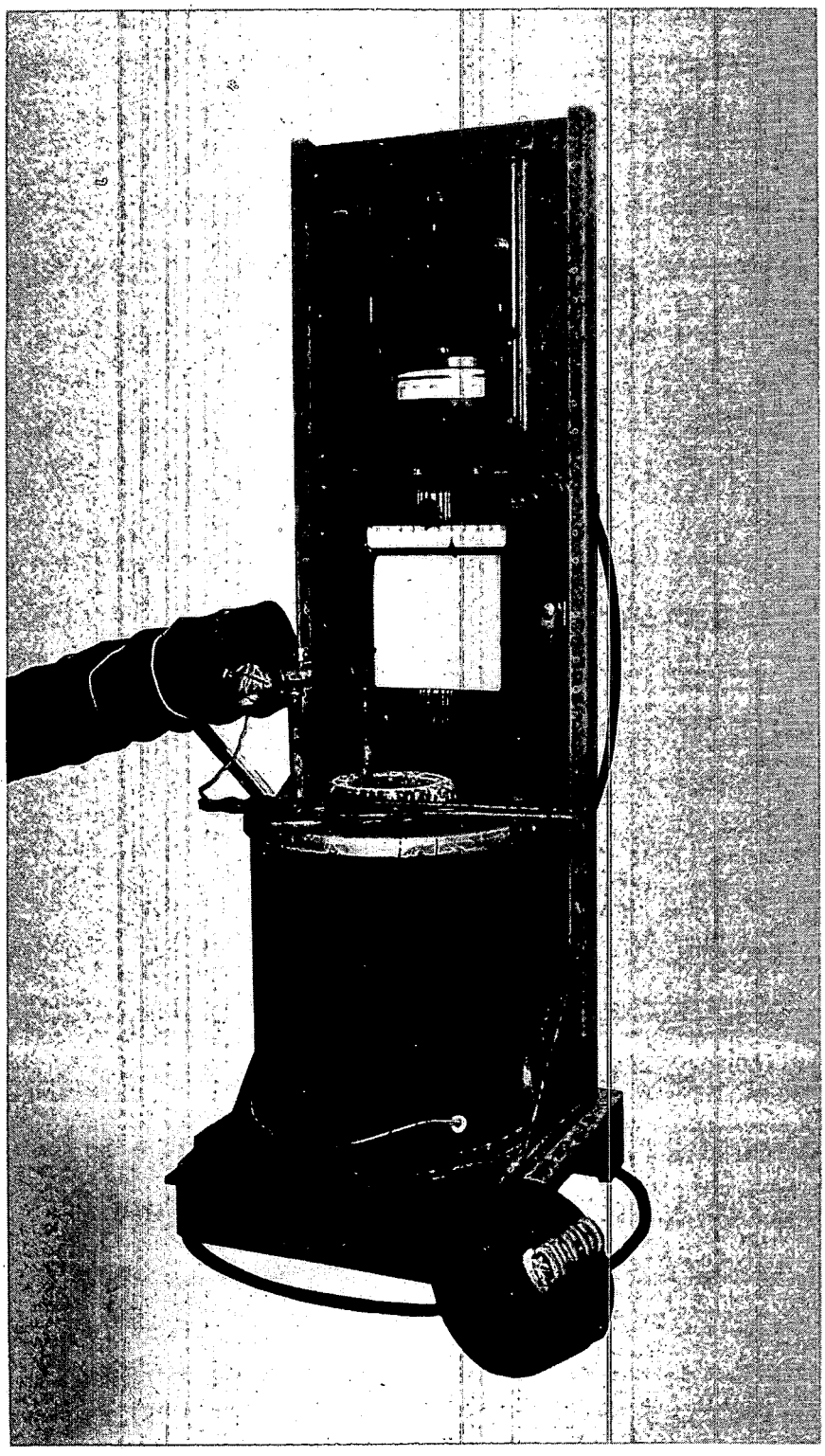
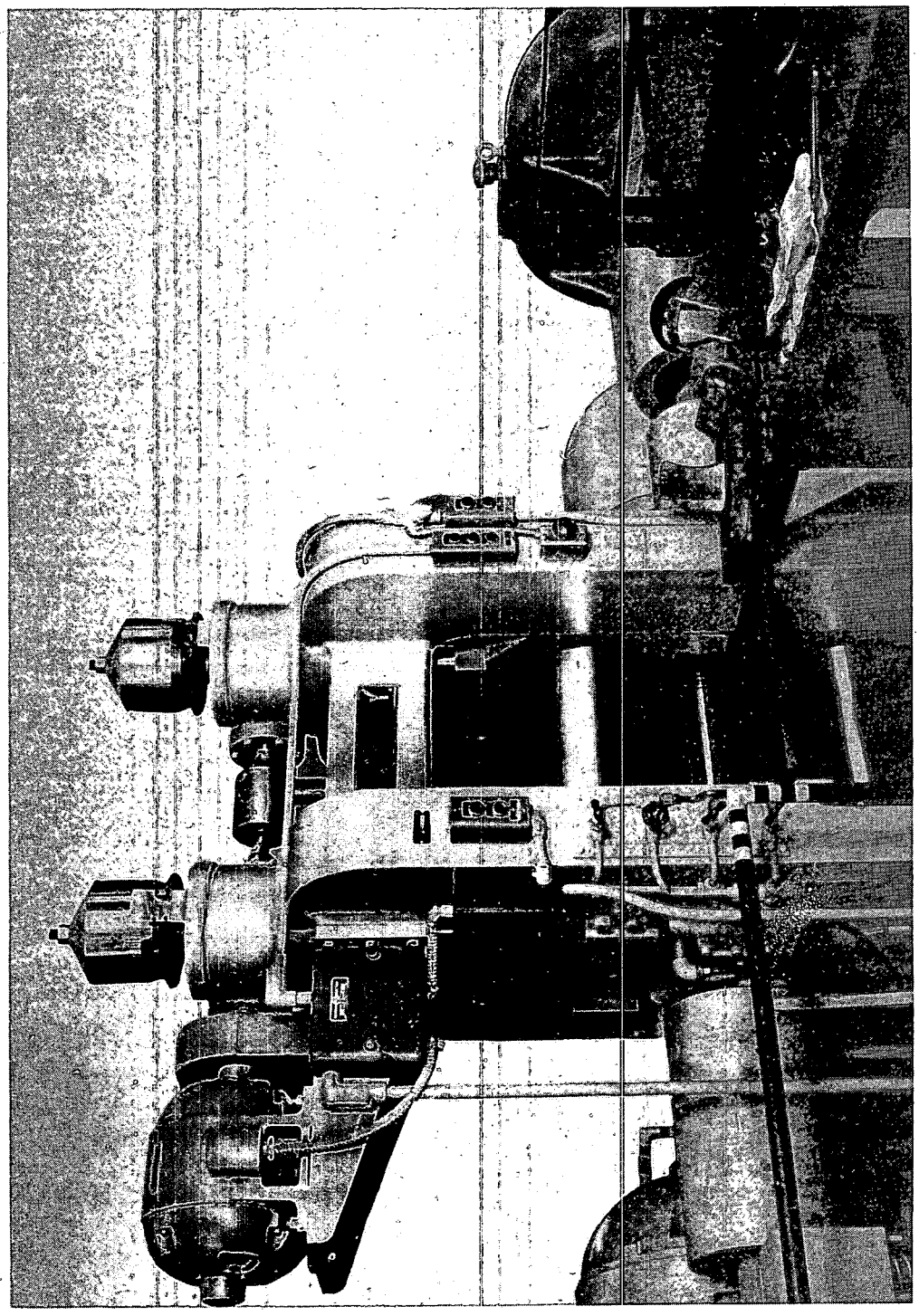


FIG. 13 - 4-HIGH LOEWY ROLLING MILL



the bottom end, was connected to a recorder seen on the stand below the controller. With the temperature control couple placed near the heating elements instead of directly in the bath, very little overshooting or undershooting was experienced and quite uniform control could be thus maintained (within $\pm$ 2°C at 600°C with no specimens in the bath).

With intermittent heating of metal during the course of rolling down to size, some lowering of bath temperature occurred. By an increase in the controller temperature, heat input to the furnace was increased and the bath temperature could be maintained reasonably well.

A length of eight inch flexible duct was run from the exhaust system to the stand and used to draw off any fumes from the bath.

No provision was made to mount an exhaust hood over the mill housing, though this would be desirable so as to withdraw fumes occurring during rolling operations, especially when rolling "25" metal.

Loewy 4-High Mill

For rolling and finishing of uranium sheet wider than approximately six inches, the small 4-High Standard mill could not be used. For the wider sheet, a large 4-High Loewy rolling mill was available, Figure 13. This mill had work rolls 3 3/4 inches in diameter, back-up rolls 18 inches in diameter, and a 20 inch roll face.

The work rolls were driven separately by two synchronized 75 horsepower, AC induction motors operating through separate gear boxes.

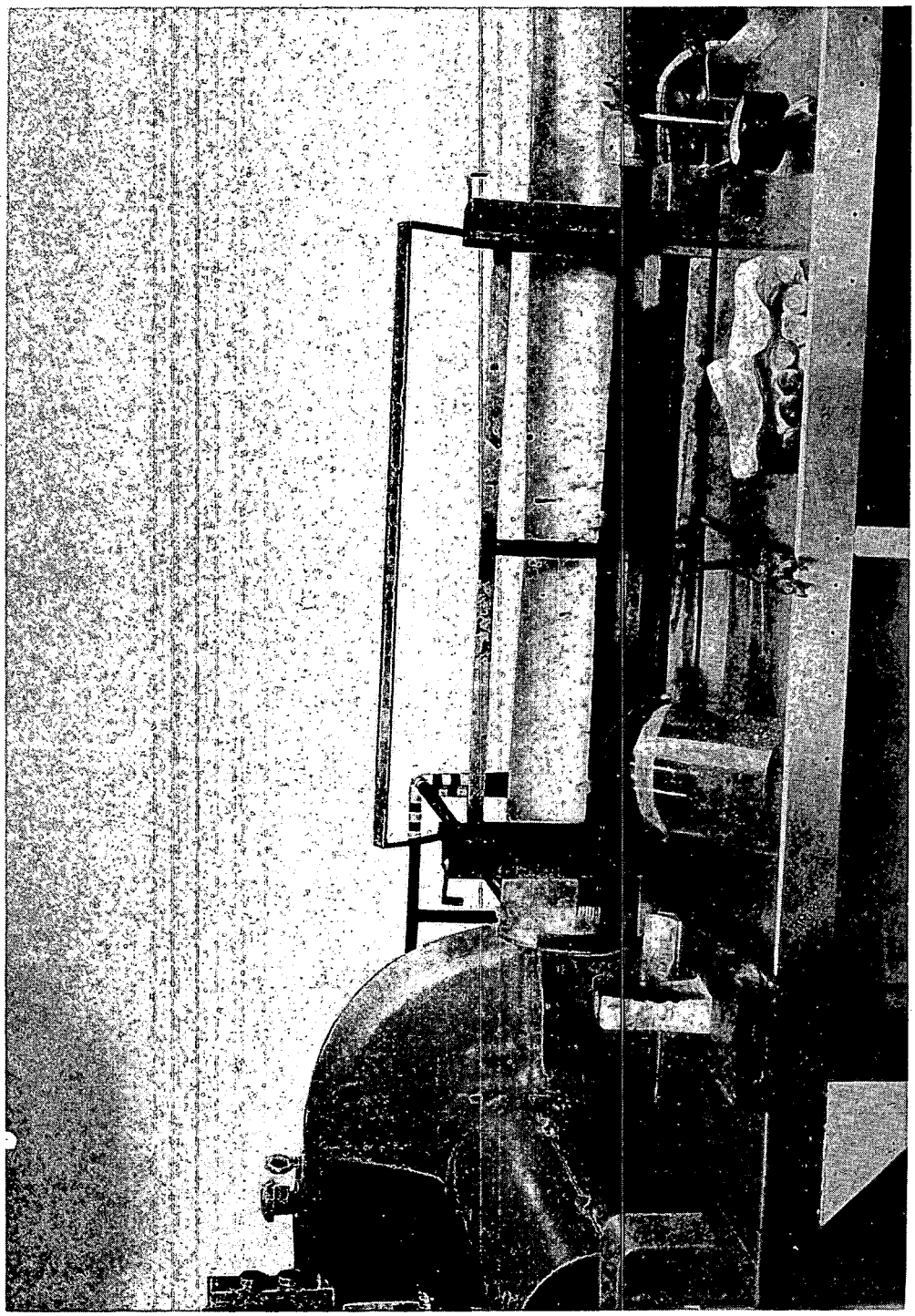
This arrangement gave a constant work roll speed of 100 RPM and a constant delivery speed of 96 feet/minute. Experience showed that this speed was too great when rolling cadmium, copper, and similar soft metals, causing tearing and wrinkling of the sheet. It was feared the mill would be too fast for rolling uranium sheet. This did not prove to be true, and uranium sheet was rolled quite satisfactorily at this speed. This mill and the Bliss 2-High mill were equipped with large "crash switches" on both the feed and delivery sides of the mill. These "crash switches" could be used to stop the rolls in an emergency, and proved valuable on several occasions.

An electric motor was used to raise and lower the top back-up roll, and the mill was provided with a hydraulic system operated by an electric motor and a compressor to snug the top work roll up against the back-up roll.

The mill could not be reversed, and did not have any exhaust provision over the mill housing, both of which would have been desirable features.

In connection with the 4-High Loewy mill, a stainless steel table on rollers was designed for mobility, Figure 14. It contained a deep-well tank, lines with Calrod immersion heaters for heating the silicone and later the Meltemp No. 7 oil used as a heating medium. A De Khotinsky thermo-regulator was used to control the temperature of the bath.

FIG. 14 - CLOSE-UP SHOWING WORKING SURFACE OF MOBILE STAINLESS STEEL TABLE WITH BUILT-IN HEATING TANK. USED WITH LOEWY 4-HIGH MILL. WOODEN "PUSHER" FOR FORCING METAL INTO THE ROLLS IS AT LEFT ON TABLE.



The table could be used as a feed table and provided a sizable working surface. As the table could be rolled up flush against the mill table, it enabled the operator to get the rolled sheet into the mill with a minimum of delay. This was advantageous as it prevented the sheet from losing too much heat before entering the rolls, especially on thicknesses below 0.050 inch, where the heat loss could reduce the metal temperature rapidly.

As a precaution in case of fire, fire extinguishers were situated convenient to both the Loewy and Bliss mills.

As can be seen in Figure 14, an arrangement for lowering and raising specimens in and out of the oil bath was designed. This mechanism was advantageous in working with large and heavy sheet specimens, and a considerable aid in removing specimens from the bath because the Meltemp No. 7 oil was so opaque that specimens immersed in it could not be seen when beneath the surface of the bath.

Heating Media for Warm Rolling

DC 550 Silicone Oil

Silicone oil was originally used in the warm rolling of U-235 stock at Los Alamos*, and this practice was continued for warm rolling in the beginning of the present rolling program. Due to the high cost of this oil, about $55 a gallon, and the fact that it had a closed cup flash point of only 314°C, it was decided to search for a cheaper and better medium for warm rolling.

*Report LA-720, Series B, dated 29 December, 1948, pg. 13.

Meltemp No. 7

Argonne Laboratories reported the use of Meltemp No. 7, a high flash point oil produced by Metal Lubricants Company of Chicago. This was tried and found superior to Silicone for the following reasons:

1. Its closed cup flash point of 347°C enabled warm rolling to be done in the range from 325 to 340°C, as compared to a top temperature of 300°C with silicone. The increased plasticity of uranium metal at the higher temperature range enabled larger sheet to be rolled and larger reductions to be attained with no apparent increase in roll pressure.
2. Meltemp No. 7 was approximately fifty times cheaper than Silicone and less care was required, since oil losses during rolling were not nearly as large an item of expense.
3. Meltemp No. 7 stuck to the work rolls and formed a tight, gummy film which appeared to increase the "bite" of the rolls and facilitate rolling.
4. The surface of uranium sheet produced was very smooth and even, although silicone was just as good in this respect.

Though the consistency of Meltemp No. 7 at room temperature made it more difficult to handle, and soiled clothes and shoes more than did silicone, it was nevertheless felt to be superior to silicone for general warm rolling usage.

Heating Media for Hot Rolling

Selecting a satisfactory heating medium for rolling in the high alpha range around 600°C presented a more serious problem. Uranium oxidizes rapidly at these temperatures unless carefully protected, and an ideal medium for hot rolling must not only protect the surface of the uranium metal from oxidation, but must not adhere so tightly that it can not be stripped or washed off easily, must not alloy with the uranium metal, must not gum the working rolls, nor be excessively lubricating so the work rolls slip on the surface of the metal, and should not be too expensive, too toxic, or too dangerous to handle.

Inert Atmospheres

In the early part of the forging program at Los Alamos, specimens for forging were heated in an electric muffle furnace flushed with an atmosphere of argon. It was found very difficult to keep the atmosphere in the furnace sufficiently inert to prevent severe oxidation of unplated uranium samples. It was also found that moderate to heavy oxidation of plated samples occurred with the resultant loss of metal. The use of an inert atmosphere in which to preheat for rolling was not used.

Lead Bath

Heating in a bath of molten lead was tried, with generally unsatisfactory results.

Before actually using the lead bath to preheat samples for rolling, a series of preliminary experiments was run as part of the

forging program. Uranium slugs, 1 inch in diameter and 3 inches long (see illustration on right side of Figure 15), were heated in a lead bath at 600°C for fifteen minutes, with the following coatings:

1. Uncoated, oxidized Tu.
2. Tu freshly etched with nitric acid.
3. Coated with 0.003 inch Cu plate.
4. Coated with 0.003 inch Ag plate.
5. Coated with Aquadag.

As was to be expected, the Ag and Cu plated specimens showed very heavy adherence of lead. The specimen on the left of Figure 15 is the Ag plated specimen and shows extremely heavy adherence of Pb; that next to it is the Cu plated specimen, which also shows excessive Pb adherence. The third figure from the left shows the spotty lead adherence to a bare, uncoated uranium specimen.

In addition to sticking to the Cu and Ag coatings, the lead tended to diffuse through these coatings, and alloy with the uranium metal underneath, especially at cracks and minor discontinuities in the base uranium metal. This diffusion effect can be seen by examination of Figure 16, which shows the dark lead diffused through the copper plate into the uranium base metal (light area).

There was also appreciable adherence of Pb on the uncoated, oxidized sample and on the freshly etched sample of uranium, with the oxidized bar showing least adherence of lead to the uranium surface.

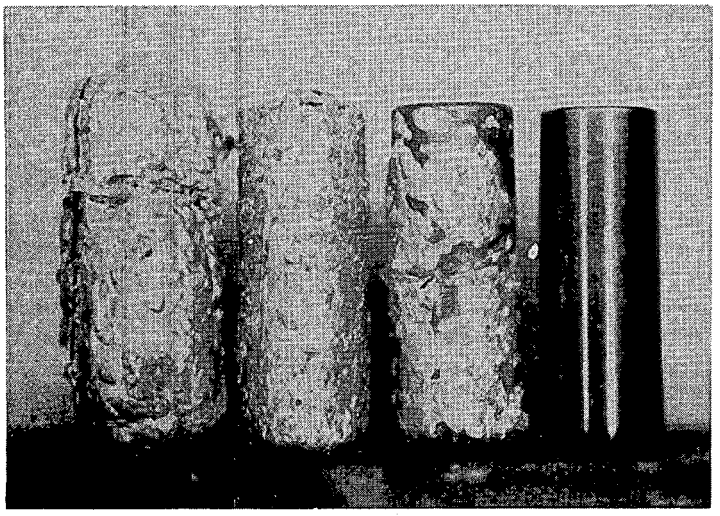


FIG. 15 - PLATE J82-D-1.

RIGHT - SAMPLE OF URANIUM SLUGS USED FOR LEAD ADHERENCE TESTS.

LEFT - SILVER PLATED URANIUM SPECIMEN SHOWING EXTREMELY HEAVY LEAD ADHERENCE.

SECOND FROM LEFT - COPPER PLATED URANIUM SPECIMEN SHOWING EXCESSIVE LEAD ADHERENCE.

THIRD FROM LEFT - UNPLATED, "BARE" URANIUM SPECIMEN SHOWING IRREGULAR LEAD ADHERENCE.

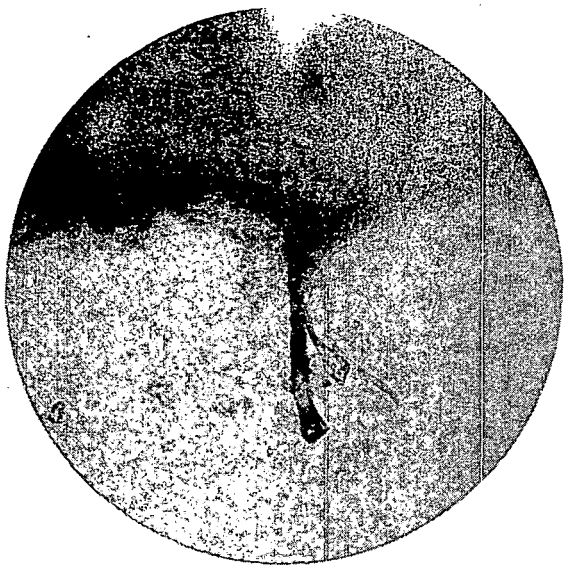


FIG. 16A - PLATE J85-C-1. CU PLATED URANIUM HEATED TO 600°C IN LEAD (0.003 INCH CU). SHOWS DIFFUSION OF LEAD (DARK AREA) THROUGH THE CU PLATE INTO URANIUM BASE METAL (LIGHT AREA). ETCHANT: 3 PARTS ACETIC ACID, 1 PART 9% HYDROGEN PEROXIDE. MAG. 250X.

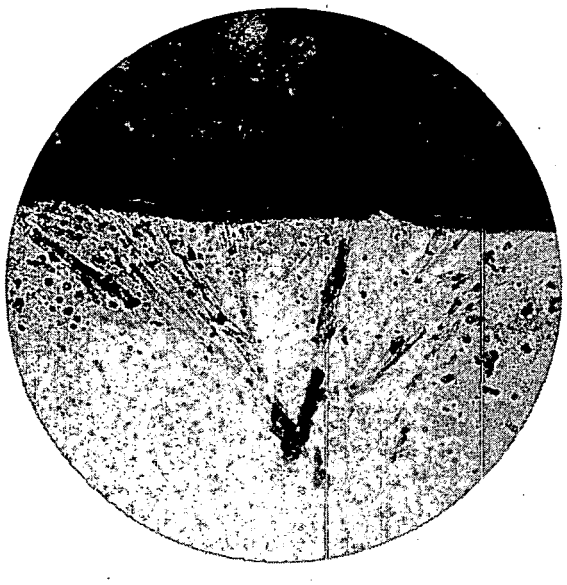


FIG. 16B - PLATE J85-D-1. URANIUM (LIGHT AREA) HEATED IN LEAD AT 600°C, SHOWING LEAD DIFFUSION INTO URANIUM METAL. ETCHANT: 3 PARTS ACETIC ACID; 1 PART 9% HYDROGEN PEROXIDE. MAG. 100X.

The uranium sample, uncoated but immersed in Aquadag prior to heating in the Pb bath, showed negligible adherence of lead, as long as the graphite coat was continuous. A continuous coat of graphite is difficult to attain, and any small area not coated permits adherence of Pb and Pb alloying.

In addition, Aquadag would be impractical to use in rolling of U-235 or high purity uranium, as there would be an opportunity for carbon contamination during hot rolling operations.

In addition to the above, lead is very difficult to strip from uranium in an acid bath, and large amounts of base metal are lost. This metal loss would be unsatisfactory from an accountability standpoint.

Nevertheless, two separate specimens of normal uranium were rolled using a molten lead bath as the preheat medium. Both samples were cut from as-cast plates of normal uranium. The surface was machined but not plated. The Bliss 2-High mill was used to roll both samples.

The first specimen, 1-6A, was 2 3/4 by 2 7/8 by 0.725 inches and was reduced to 136 mils in four passes at 600°C. It was very difficult to remove the Pb films adhering to the sheet after rolling.

The second specimen, 1-6B, of dimensions identical to 1-6A, was rolled down to 20 mils in twelve passes at 600°C. Rolling was generally unsatisfactory for the following reasons:

1. The stock was difficult to get into the rolls. The thin film of molten lead acted as a lubricant and reduced the bite of the rolls.

2. On heavy reductions, the rolls started to slip after the specimen had entered. Power and momentum were lost and the specimen stalled midway in the rolls and had to be backed out.

3. The severe edge cracking that occurred was thought to be caused by the Pb which occasionally covered only certain areas of the specimen. The rolls tended to slip where the specimen was covered with lead, but gripped and pulled where no lead was present. Such rolling action would result in uneven stresses on the sheet and could cause the severe edge cracking noticed.

4. The surface of the sheet after final rolling was rough and spongy and contained tightly adhering films of lead distributed unevenly over the surface, Figure 17.

Battelle Memorial Institute has reported metal losses ranging up to 16 percent in using Pb and Pb-Sn baths to preheat metal for rolling.[*] These losses are large from an accountability standpoint; however, rolling uranium which has an oxidized surface or is coated with Aquadag might be satisfactory for large pieces which are to be subsequently machined and where weight losses are not an important factor.

For the present rolling program, the use of Pb baths was considered inadvisable.

*Battelle Memorial Institute Progress Report for January, 1950, pg. 19

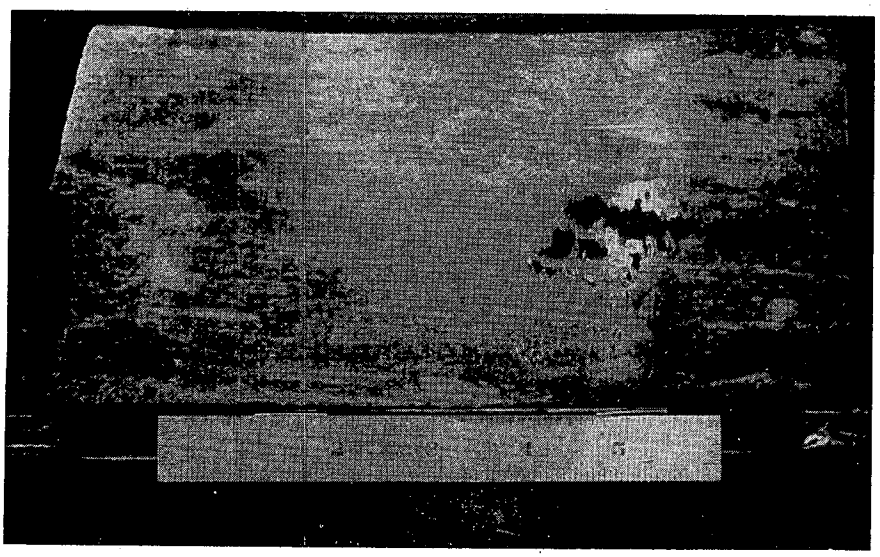


FIG. 17 - PLATE J93-A-2. PHOTOGRAPH OF URANIUM SHEET ROLLED FROM 725 MILS TO 29 MILS AT 630°C USING LEAD BATH AS A HEATING MEDIUM. THE LIGHT GRAY AND WHITE AREAS ARE TIGHTLY ADHERING FILMS OF LEAD WHICH HAVE DISTRIBUTED THEMSELVES UNEVENLY OVER THE URANIUM SHEET.

Chloride Salt Baths

The behavior of chloride salt baths for heating around 600°C was also investigated as part of the forging program.

KCl and LiCl - A eutectic mixture of Kcl and LiCl, with melting point at 360°C, was prepared and a uranium sample immersed in the melt at 600°C for fifteen minutes. Weight loss was 0.6 percent and a thin, black, nonadherent oxide coating was formed.

$BaCl_2$-LiCl in quantities to give the eutectic mixture was also tried as a salt bath. A uranium sample was heated in this bath for fifteen minutes at 600°C. The weight loss of metal was less than 0.1 percent. A specimen of uranium heated in the bath for two hours at 600°C showed evidence of a surface reaction. A tendency for the bath to boil was also considered as evidence of a reaction between the metal and salts.

KCl-NaCl-$BaCl_2$ - A combination of the three chlorides in the ratio 25% KCl, 20% NaCl, and 55% $BaCl_2$ was also investigated as a possible bath. This eutectic mixture caused the formation of a thin, tightly adhering coat on the surface of unplated uranium when the metal was immersed in the bath for fifteen minutes at 700°C.

The corrosive action of the chloride salts on rolls, tools, salt pot, etc.; the possibility of attack on uranium; and the question of toxicity of the chlorides caused this particular type of salt bath to be abandoned.

[redacted]

Li_2CO_3-K_2CO_3 - In the Knolls' Atomic Power Laboratory Report No. 179 for March, 1949, the use of a mixture of 35% Li_2CO_3 and 65% K_2CO_3 for heating uranium metal was described. This eutectic mixture has a melting point of 510°C and is not recommended for use above 700°C.

Preliminary tests in which copper plated uranium specimens were immersed in the above carbonate salt bath for fifteen minutes at 600°C showed no attack on the copper plate, as opposed to a severe attack of the copper when the specimens were treated in chloride baths.

Accountability tests showed that uranium specimens placed in the carbonate bath for fifteen to sixty minutes at 600°C lost less than 0.5 percent in weight with the average loss being about 0.2 percent. The metal loss was essentially oxide retained in the bath.

The uncoated uranium specimens, when removed from the bath, had a thin, tightly adhering, continuous film on the surface. This film was estimated to be about 0.0001 inch in thickness and considered to be an oxide. No direct analytical tests were made to prove the coating to be an oxide. However, chemical tests indicated that the coating was not a carbide; thus the assumption of an oxide coating seemed reasonable.

In view of the above experimental results using the carbonate bath, it was decided to use the bath as a preheating medium for uranium.

An as-cast uranium plate 6 by 3 by 0.800 inches was obtained and reduced to 60 mils in thickness. The results from the test, discussed below, were generally good.

[redacted]

1. The salt heated evenly without boiling or spattering up to 650°C.
2. Around 600 to 650°C, the molten salt was fluid enough so that most of it ran off the specimen when the specimen was removed from the bath.
3. The thin film of salt remaining on the specimen was usually spread evenly over the surface of the specimen, and did not tend to gather in pools.
4. The surface film of salt did not reduce the friction between the rolls and the stock, as did lead.
5. If heated work rolls are used, and the rolled specimen kept hot enough - no time delay from the bath to the rolls - the salt remains liquid on the surface of the specimen while passing through the rolls and does not mar or pit the sheet surface.
6. After rolling, the frozen salt can be easily removed from the sheet by washing in warm water.
7. When immersed in the salt bath, a thin, tight oxide coat is formed on the metal. This coat rolls down smoothly and evenly and does not flake or chip off. The final surface of the rolled sheet was excellent.
8. The carbonate salts are not toxic, give off very little fumes at 600°C, and are comparatively cheap.

As a result of the above observations, it was decided to use the mixture of 65% K_2CO_3 and 35% Li_2CO_3 for all hot rolling of uranium metal. In the future, references to "salt bath" in this report will refer to the eutectic carbonate mixture described above.

As is true of all salt baths, the carbonate salt bath must be handled with care, precautions being taken not to immerse moist or damp specimens, tongs, or foreign materials in the bath. Suitable protection of personnel should be in effect at all times.

The general procedure used in rolling uranium was to preheat all samples on the top surface of the furnace in order to dry thoroughly before immersing in the bath. Care must be taken not to allow water to drip or splash into the bath, and the bath should always be stirred gently and carefully.

When adding fresh salt to the bath, it is advisable to add the well stirred mixture of dry K_2CO_3 and Li_2CO_3 when the bath is cold, and allow the addition to dissolve as the bath comes up to temperature. If fresh salt must be added while the bath is hot, small amounts should be placed in the molten salt, and dissolved slowly before adding additional salt, as some spattering occurs when the addition is made.

Preparation of Stock for Rolling

Plating Materials

In working with uranium and especially with U-235, it had been customary to plate either silver or copper on the metal to prevent

excessive oxidation losses during rolling, forging, and other forming operations. A series of accountability tests was made as part of the forging program to determine the efficiency of these protective coatings.*

The tests showed that more uranium metal was lost in etching prior to plating and in stripping after plating, than was lost by rolling or forging uncoated specimens heated in the salt bath and afterwards stripping the thin oxide formed.

As part of these tests, uranium specimens were plated with 0.001 inch nickel, with 0.0003 inch nickel, and with 0.0003 inch nickel on top of which 0.003 inch copper was added. Diffusion of these specimens at 600°C, 700°C and cycling between the high alpha and the low gamma temperatures indicated that some diffusion of the plate metal into the uranium may have taken place, but the photomicrographs taken were very inconclusive. Forging results indicated that both copper and nickel plates, when heated in a drying over, adhered to the uranium base metal during forging just as well as did copper and nickel plates diffused at 600°C in vacuum for one hour prior to forging.

Rolling experiments with plated specimens were as follows:

1. Silver Plate - Extensive rolling operations on U-235 coated with silver showed rather poor adherence of the

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*The results of these tests will be incorporated in a report on the Forging Program.

silver plate.* Cast bars 8 by 2.375 by 0.250 inches were plated with 0.003 inch of silver and warm rolled at 250°C on the 4-High Standard mill to 0.040 inch in thickness. The silver plate peeled in patches. Annealing prior to rolling and/or a light skin pass to "set" the plate before rolling, did not improve adherence. From this experience, it was felt that less metal would be lost by rolling without the silver plate, although the greater part of metal losses due to stripping of the plate are recoverable in the stripping bath.

The adherence of the silver in patches caused uneven surface on the sheet. It was necessary to make one or two passes on the bare unplated metal after stripping the silver to secure sheet having a satisfactory surface.

2. Copper Plate - Copper plating was also found to be poor from the standpoint of adherence and protection. A cast bar of normal uranium 4 by 2.5 by 0.8 inches was plated with 0.003 inch copper. No anneal or diffusion of the plate prior to rolling was given. The metal was heated in the carbonate salt bath to 600°C and reduced on the Bliss 2-High mill with work rolls at 100°C from 0.800 inch

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*Preparation of U-235 sheet for G. E. Discs.

down to 0.029 inch, a 96 percent reduction in thickness. Almost complete separation and peeling of the copper plate from the uranium resulted during the reduction. Another specimen of normal uranium rod 0.625 inch in diameter and 2 inches long was plated with 0.003 inch of copper and upset in two planes by forging. The copper plate peeled extensively during forging. The forged section was heated to 620°C and rolled on the Bliss 2-High mill with heated work rolls. A reduction in thickness was made from 0.257 to 0.039 inch, after which practically no plate remained on the sheet.

A third experiment to study the protective effect of copper plate on uranium was carried out. An as-cast normal uranium bar 3 by 2 3/4 by 5/8 inches was plated with 0.002 inch of copper, heated to 625°C in the salt bath and rolled on the Bliss 2-High mill. In spite of a special etch given the uranium to acquire better adherence of the copper plate, the plate started peeling on the sixth pass at a thickness of 0.173 inch, and peeled off entirely on the seventh pass at 0.137 inch thickness.

3. Nickel and Copper Plate - As the electroplates of copper and silver did not adhere to uranium during rolling, it was thought that similar plates treated to diffuse the

plate into the uranium might adhere successfully. A specimen of as-cast, normal uranium 2 3/4 by 2 3/4 by 0.68 inches was given a coat of nickel approximately 0.0003 inch thick. A 0.003 inch thickness of copper was plated over the nickel and the bar heated in vacuum at 750°C for forty-eight hours to thoroughly diffuse the plate. The bar was then heated in salt to 610°C and reduced on the Bliss 2-High mill using cold work rolls, from 0.680 to 0.065 inch, a reduction of ninety percent in thickness. As can be seen in Figure 18, approximately fifty percent of the plate had peeled off. In some places where the plate did adhere, it appeared to stick very tightly and afford good protection, and there is a possibility that this plating and diffusion procedure can be improved to work successfully.

Unplated Surface

A series of accountability checks made on unplated specimens showed less metal loss than had been the experience with plated specimens. Hot rolling of some ten unplated, as-cast uranium samples showed that excellent sheet surface can be produced without a plated protective coat. Weight losses, as measured by weighing carefully before rolling and after rolling with thorough removal of adhering salt, were a maximum of 0.5 percent. Over the total number of samples rolled, the

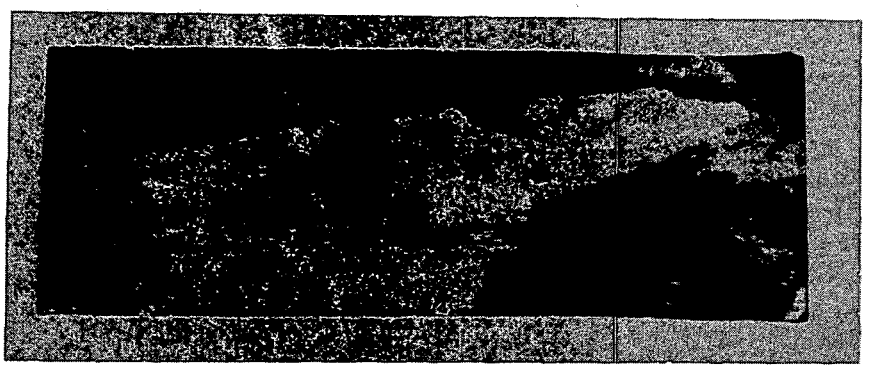

FIG. 18 - PLATE NO. J-117-1. SECTION OF ROLLED URANIUM SHEET. ORIGINAL SPECIMEN HAD 0.3 MIL NICKEL PLATE, THEN 3 MILS COPPER PLATE AND WAS DIFFUSION TREATED FOR 48 HOURS AT 750°C IN VACUUM. A 90% REDUCTION IN THICKNESS (HOT ROLLING AT 610°C) HAS CAUSED THE PLATE TO PEEL AWAY FROM THE URANIUM.

weight loss of metal averaged 0.2 percent per sample.

In view of these results, all hot and warm rolling of uranium, outside of the experiments cited above, has been done with unplated specimens.

Machined Surface

On several samples, the as-cast surface of the bar was machined prior to rolling to see if this would improve surface properties of the rolled sheet. Comparison of the surfaces of rolled sheet, the specimens having initially a machined surface or an "as-cast" surface, showed no noticeable difference in properties. It was therefore decided that machining of the as-cast surface prior to rolling was unnecessary.

Techniques Used in Hot Rolling of Uranium

The following techniques were evolved as being the most suitable methods for the satisfactory hot reduction of as-cast uranium billets, consistent with the equipment available for these experiments.

Prior Anneal

Prior annealing of the as-cast uranium was done to determine its effect upon hot rolling. In one instance, two samples of uranium, one normal and one high purity, each taken from identical sections of similar castings, were immersed in the salt bath at 600°C for forty minutes prior to rolling. In a second case, four small as-cast samples, one high purity and three normal uranium, from different sections of castings, were immersed in salt at 650°C for one-half hour before rolling.

In none of the above experiments was there a significant difference in response to rolling, as compared to uranium samples which received no heat treatment prior to rolling. Reduction per pass and total reductions were approximately the same, and samples behaved identically, regardless of prior annealing.

In effect, hot rolled samples were subject to intermittent annealing during the course of rolling, as each sample was heated to rolling temperature, passed through the rolls, and then immediately replaced in the salt bath to bring the somewhat cooled sample up to rolling temperature. Thus, there was no reason for a sample which had prior annealing to behave differently upon rolling than one which had no prior annealing.

Size and Shape of Specimens for Rolling

Specimen size was not critical, being determined only by the amount of sheet or rolled stock desired, by the capacity of the mill being used, and by limitations imposed by the size of the salt pot.

The Bliss 2-High mill was used for all hot rolling operations described in this report because of the large, 8 inch diameter work rolls and the large angle of nip or "bite". If thinner, as-cast uranium samples had been used, there is no reason to believe that the other mills available (the larger 4-High Loewy or the smaller 4-High Standard) would not have proven satisfactory for hot rolling operations.

As-cast specimens 6 by 3 by 0.800 inches, 3 by 3 by 0.800 inches, and 3 by 1.5 by 0.800 inches were rolled satisfactorily, as were even larger forged specimens up to one inch in thickness.

Only flat stock was rolled, as only straight rolls were available. The geometry of the specimens, other than always being flat, was not considered as a variable, for square bars, rectangular bars, and bars with convex edges all rolled with equal ease. No difference in rollability was apparent as a function of shape.

Mill capacity and its relation to billet size will be described in detail in the chapter on reductions per pass and total reduction.

Time to Temperature

The rate of heating of uranium metal in molten salt was investigated as part of the forging program. A one-quarter inch hole was tapped in a cylinder of uranium 3 inches long and 1 inch in diameter. A chromel-alumel thermocouple was inserted through the center of a uranium plug screwed into the hole. The assembly was placed in the bath of molten salt at 600°C, and curves of time versus temperature recorded. Results showed that four to five minutes were required for the center of the 1 inch diameter uranium cylinder to arrive at the 600°C temperature.

Although five minutes was considered to be adequate time for heating in the salt bath prior to rolling of specimens up to 1 inch in

thickness, it was general practice to preheat at least ten and often fifteen minutes before rolling to assure thorough heating.

Rolling Temperatures

Specimens were rolled hot at temperatures ranging from 550°C to 640°C. Although the plasticity of uranium is considered to increase over this range of temperature, qualitatively no appreciable increase in ease of rolling or ease of deformation could be noticed. As equipment for determining roll pressures was not available, no quantitative figures to compare temperature effect upon ease of rolling can be presented.

It is felt that rolling temperature is not critical, so long as the temperature is in the high alpha range (550-620°C). Rolling temperature might be more important where production is needed. In that case, heavy reductions per pass might be desirable and it would be necessary to study the effect of temperature upon deformability.

A listing of roll pressures (Battelle Memorial Institute Progress Report for December, 1949) shows a decrease in closed-pass roll pressures of 10 to 60 percent in going from 500 to 600°C rolling temperature, with the average reduction in roll pressures being about 30 percent.

The large variations in roll pressures due to geometry tend to show that precise rolling temperatures would not be important, as long as one stays well above the recrystallization temperature, as the variations in roll pressures due to small temperature variations would

be less than those due to geometry of the stock and the reduction per pass taken.

A 600°C salt bath temperature was therefore used as a convenient, median figure and all heating was done at this standardized temperature to eliminate the temperature factor as a variable in hot rolling.

Time Between Extraction from the Salt Bath and Insertion into Rolls

The time elapsed between removal of the specimen from the salt bath and start of rolling was not thought critical, due to the fact that fairly large billets were used which retained their heat for an appreciable period of time. To test this assumption, a cooling curve for a 6 by 3 by 0.800 inch uranium sample was obtained. A chromel-alumel thermocouple was inserted through a copper plug into the interior of the plate of uranium and the hole sealed with an insulating ceramic material. The specimen was heated to 600°C in salt and allowed to cool in air. A temperature versus time curve for the cooling was obtained on a Speedomax recorder. This specimen cooled from 537°C to 382°C in two minutes, twenty-five seconds, a temperature drop of approximately 65°C per minute for the 0.800 inch thickness used. It was nevertheless considered wise to get the rolling stock into the rolls with a minimum of elapsed time, usually within ten seconds after extraction from the bath, which included two to three seconds holding time over the salt bath to allow excess molten salt to drain off.

This rate of cooling was more important in warm rolling of thin gauge stock. In several instances where material could not be fed into the rolls within a reasonable length of time (approximately thirty seconds or less), it was felt that too much heat was lost, since it became very difficult to force the specimen into the rolls, and the piece was reheated before rolling. The time limit to the delay from salt pot to rolling could be roughly determined as follows:

The freezing point of the carbonate salts used, 510°C, provided an excellent qualitative gauge of the surface temperature of the specimens. As long as the salt on the surface of the stock remained completely molten, it was considered to be hot enough for rolling. Once the salt started to freeze on the surface, the metal was considered too cold for rolling and was returned to the bath for reheating.

Roll Temperatures

As previously mentioned, the Bliss 2-High mill was equipped with banks of infra-red heat lamps to heat the work rolls, and work roll temperatures up to 100°C could thus be attained.

Specimens were rolled at various work roll temperatures starting at room temperature, approximately 23°C, up to 100°C.

It was noticed that the work roll temperature was not critical in hot rolling. Sheet surface and edges and ease of reduction were similar whether cold work rolls or heated work rolls were used. It was found, however, that when heated work rolls were used, the temperature of the

surface of the metal after passing through the rolls was higher by almost exactly the temperature difference between the heated and cold work rolls, as compared to stock rolled with cold work rolls.

It was concluded that, although heated work rolls were desirable in preventing a temperature drop during rolling, and possibly facilitated ease of reduction slightly for this reason, they were not necessary for hot rolling.

Actually, if large amounts of uranium metal were to be hot rolled on a production basis at temperatures around 600°C, the rolls might become heated from continuous contact with the hot metal, and some provision for cooling the work rolls might have to be made to prevent a temperature rise of uranium metal during deformation into the brittle beta region (around 660°C), where the metal would crack up during rolling.

Consideration had been given to a method for circulating hot oil through the hollow centers of the work rolls on the Bliss mill to obtain roll temperatures in excess of 100°C. The experiments indicated that temperatures higher than 100°C would have no particular advantage, so plans to heat the work rolls by interior means were abandoned.

Roll Speeds

Since the Bliss mill used for hot rolling was equipped with a rheostat control which enabled the operator to vary roll speeds, the effect of such varied roll speeds on hot rolling of uranium was studied.

Very slow speeds of 5 RPM were first tried, and caused the mill to stall on moderately heavy reductions per pass. Roll speeds of 20, 25, 30, 35, on up to 60 RPM were then tried, and though all were successful as far as reducing the metal was concerned, a roll speed of approximately 30 RPM was found most satisfactory. At this speed, the 30 HP motor had enough momentum to pull the hot metal through the rolls at reductions of up to 12 or 15 percent per pass, and the speed of the rolls was still not so great as to present a hazard to the safety of the operator.

Roll speeds were not thought to be a critical factor, though on a production setup, roll speeds would have to be determined by the strength of the mill available, the reduction per pass desired, and the speed of the hot breakdown process necessary for profitable operation.

Rolling Direction

Rolling was always done on the flat sides of the billet, but in alternately longitudinal and transverse directions where possible.

The dimensions of the pot used for the salt bath, 12 inches deep and 6 inches in diameter, limited the amount of transverse rolling that could be done on the piece to about four or five passes. After that, the piece was too wide to fit into the salt bath.

Tensile test specimens taken parallel to, at 45 degrees to, and perpendicular to the direction of rolling, showed consistently similar results for sheet cross-rolled only two or three times, as well

as on sheet cross-rolled five or six times. Cupping tests based on a modified Olsen test showed that two or three cross-rolls were sufficient to produce sheet without detectable directional properties. This procedure - cross-rolling for only as long as the piece would conveniently fit in the salt bath - was adopted as standard procedure. All remaining passes were then made in the longitudinal direction.

Reduction per Pass

In the Battelle Memorial Institute Progress Report for February, 1950, hot reductions of 10, 20, 30, 40 and 50 percent per pass on small samples of uranium metal were reported. Although reductions up to 50 percent or more per pass should be possible on large billets by hot rolling, metallographic studies, which will be presented in later chapters of this report, indicate that heavy reductions per pass are not necessary to produce a finished, recrystallized uranium sheet having uniform, equi-axed grains. It thus appears that heavy single reductions might have an economic advantage, but are not necessary to produce finished sheet having satistactory properties.

On the Bliss mill used for hot rolling in these experiments, the 30 HP driving motor limited the reduction per pass on a 3 inch width of metal from a maximum of 10-12 percent reduction per pass at 0.800 to 1.00 inch in thickness, up to 20-25 percent reduction per pass at 0.250 to 0.400 inch in thickness. On one occasion, 73 percent reduction was obtained in a single pass.

In the early work, it was thought advisable to obtain maximum reductions per pass in the hope that more thorough breakdown of the as-cast structure would be accomplished. However, metallographic examination of sheet produced in later experiments showed that completely recrystallized metal containing no apparent traces of the as-cast structure could be obtained with comparatively moderate reductions per pass (in the neighborhood of 10 to 20 percent), as long as the total reduction was sufficient.

Thus, although it was earlier decided to devise some means of mechanically pushing the hot metal into the rolls to obtain maximum reductions, it was finally agreed that the use of wooden pushers, one of which can be seen in Figure 14, was satisfactory, in that sufficient deformation per pass could be thus obtained, and the roll table would be left clear of any mechanical contrivance.

Tapering of the rolling stock in order to facilitate feeding into the rolls was also found unnecessary.

Total Reduction Hot

Preliminary recrystallization studies (Battelle Memorial Institute Progress Report for February, 1950) state that recrystallization occurred after hot reductions of 40 to 50 percent.

Thus far, no experiments have been done to determine the minimum percent of hot reduction required to attain recrystallization in the worked metal.

In the present work, the interest was not pointed toward structural studies of the worked metal, but was essentially directed toward the development of methods to obtain rolled sheet having good drawing and forming characteristics. The main factors to be investigated were: procedures to effect the hot breakdown of the as-cast structure, and the effects of cold work and annealing temperatures on the properties of the finished sheet.

With these considerations in mind, the total reduction in hot rolling was not found to be a critical variable. Total reductions from 97 percent down to 65 percent by hot rolling gave sheet whose structure, after cold rolling, did not appear to have directional properties nor traces of the as-cast structure.

Actually, the total reduction hot would be determined by other considerations, such as the size of the work rolls used for hot breakdown. Experience indicates that the larger the work rolls used in hot breakdown, the more difficult is the reduction at small thicknesses, since the efficiency of large work rolls is small. Using the 8 inch diameter work rolls of the Bliss 2-High mill, it was found difficult to reduce hot below approximately 0.050 inch thickness, giving a limiting minimum ratio of sheet thickness to roll diameter of about 1:100 for hot reduction, or 1:200 at the most.

The total reduction hot would also be determined by the diameter of the work rolls used in cold rolling, since if very small

work rolls were used, heavy gauge sheet would be difficult to enter into the rolls, the angle of nip or amount of "bite" being small on such rolls. If larger work rolls were available, hot rolling could be stopped earlier, and the heavier gauge sheet cold rolled. Of course, this would make reduction more difficult if very thin gauge sheet was required.

If sheet of good formability and physical characteristics is needed, the most important consideration in where to stop hot rolling would be the amount of cold work required by the sheet before the final thickness is reached. To elaborate, metallographic studies show that at least 60-70 percent cold work and preferably more, is desirable to obtain sheet that can be easily and fully recrystallized to a given grain size by control of annealing time and temperature. Thus, if sheet 0.040 inch thick were required, it would be necessary to stop hot rolling at 0.100 inch or above to obtain the necessary reduction by cold work needed for recrystallization. However, if sheet 0.010 inch thick were needed, hot rolling down to 0.035 inch would be satisfactory (and much faster than cold reduction from 0.100 down to 0.035 inch) and a sufficient amount of cold work introduced into the sheet to obtain complete recrystallization by cold rolling from 0.035 inch to 0.010 inch.

Finishing Temperatures

The above techniques, comprising hot rolling at 600°C with work rolls heated around 80°C resulted in finishing temperatures (as measured on the surface of the rolled sheet immediately after each pass

with a Pyrocon instrument) ranging from 500 to 550°C for specimens over 0.400 inch in thickness down to 350-400°C for specimens around 0.200 inch thick. The interior of the rolling stock can be presumed to be somewhat higher in temperature than the surface. As was expected, the thicker specimens, whose surface area to weight ratio was small, lost less heat than did thinner specimens where this ratio was larger.

Handling time in getting the piece back into the bath after each pass for reheating was considered to be unimportant, as long as done with reasonable dispatch. Thick specimens lost comparatively little heat between passes, and thus did not require much time for reheating. Thinner specimens, which lost more heat between passes, did not require as much time to reheat to temperature, since they came up to temperature more rapidly than thick sections. Thus, the one minute interval required to change roll settings and record data was considered ample time for reheating to bath temperature between passes.

The actual rise in temperature of the metal during rolling caused by internal friction resulting from the work of deformation was masked by radiation losses at the surface. An experiment to determine temperature rise during rolling was performed. A chromel-alumel thermocouple was inserted through a copper plug into the interior of a sample of normal uranium 0.800 by 3 by 6 inches and the hole plugged with insulating ceramic material. After heating to 600°C in salt, the ends of the thermocouple leads were connected to a Speedomax electronic

recorder, and the piece rolled on the 2-High Bliss mill with work rolls heated to 70°C and roll speed of 25 RPM.

On the first pass in which 5 percent hot reduction was effected, the temperature rise at the interior of the piece was 6°C. During the second pass, the temperature rose 7°C while the piece was being reduced 5½ percent. The figures check similar data reported by Battelle Memorial Institute*.

An interesting aspect of the temperature rise observed during rolling was the fact that the temperature rose and returned to the ambient temperature within one-tenth of one second. This time interval corresponds to the time during which the rolls exerted pressure on the surface area above the thermocouple.

Techniques Used in Warm Rolling of Uranium

All the warm rolling and warm finishing of hot rolled uranium metal below its recrystallization range was done on either the 4-High Standard or the 4-High Loewy rolling mill previously described. The small Standard mill was used for sheet up to 6 inches wide and, where sheet of greater width was required, the larger Loewy mill was used. In all other respects, the two mills were quite similar in performance and efficiency.

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*Battelle Memorial Institute Progress Report for February, 1950, pg. 21

[redacted]

Intermediate Annealing

As might be expected, intermediate annealing or heat treatment after hot rolling should present no advantage, since the metal is in the recrystallized condition after hot rolling is completed. Several experiments, in which intermediate annealing of hot rolled specimens in salt at 600°C for one-half hour was carried out, confirmed this fact. Hot rolled specimens which received no intermediate anneal were found to warm roll as easily and efficiently as those specimens which had received an annealing treatment. Intermediate annealing was thus abandoned as a step between hot and warm rolling.

However, as an intermediate step in warm rolling at thicknesses of 0.020 inch and less, intermediate annealing was found to be definitely desirable and in some instances necessary.

In warm rolling of light gauge sheet, the uranium metal becomes increasingly work hardened until a point is reached at which the deformation pressure required to further reduce the sheet is equal to actual roll pressures available, and the rolls start yielding elastically as the sheet passes through, and spring back with very little or no reduction accomplished. With the 2.5 inch diameter work rolls of the Standard mill, this point was usually reached at a sheet thickness around 0.015 to 0.020 inch thickness. Beyond this thickness, a large number of passes (20 to 30) was required to further reduce sheet down to 0.010 or 0.005 inch. The following experiments show the value of intermediate anneals at these thin gauges.

[redacted]

Two samples of normal uranium, warm rolled 70 percent down to 0.020 inch, required nine and eleven passes respectively for further reduction to 0.010 inch with no intermediate anneal. A similar sample of normal uranium, warm rolled 70 percent to 0.020 inch, was given an intermediate anneal at 550°C for one-half hour in the salt bath, and was then reduced to 0.010 inch on the same Standard mill in only three passes.

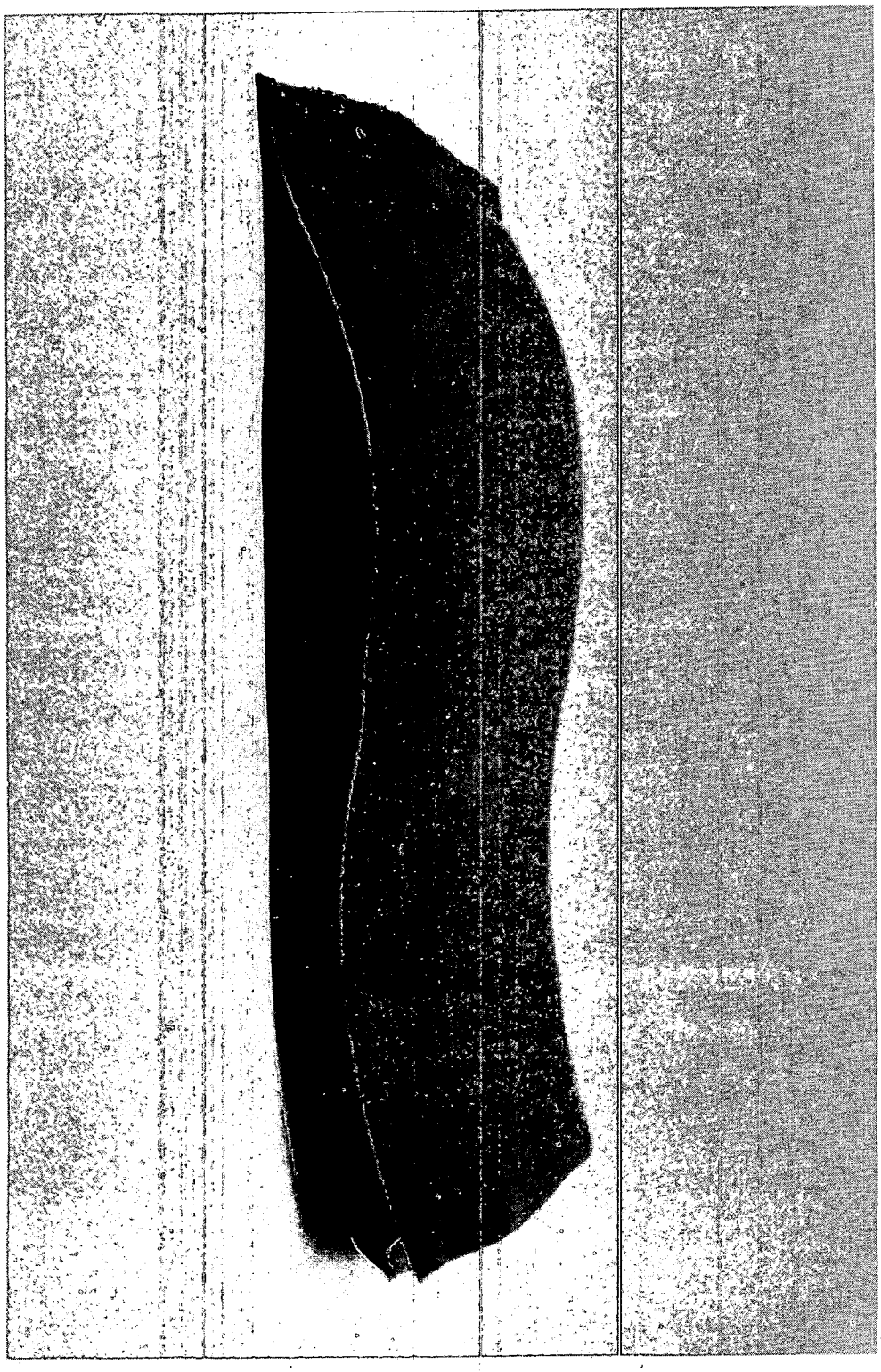
An 85 percent cold worked specimen required twenty-three passes to secure a reduction from 0.010 to 0.005 inch without anneal, while a specimen with intermediate annealing was reduced from 0.010 to 0.005 inch in six passes.

A method of obtaining very thin sheet is pack rolling of two or more sheets of uranium. Figure 19 shows the results of pack rolling two sheets of 0.005 inch uranium, which were spot welded by electrical resistance in ten places along the leading edge, annealed in vacuum at 600°C for 30 minutes, and then warm rolled at 335°C down to 0.003 inches in twelve passes, reheating between each pass.

Although this experiment was successful, other similar experiments in which sheets, spot welded and lap welded on the leading edges, were reduced down to 0.001 inch proved generally unsuccessful. The packed sheets tore and wrinkled badly, and it was necessary to shear after each pass to keep the side edges of the sheets lined up. This caused excessive metal losses.

Another series of experiments was attempted where a thin sheet of copper was inserted between each pair of uranium sheets, and the assembly then pack rolled at 300°C. This technique was also unsuccessful, with the uranium sheet again undergoing severe wrinkling, and surface and edge cracking.

FIG. 19 - TWO 0.005 INCH URANIUM SHEETS SPOT WELDED ON LEADING EDGE, ANNEALED, AND WARM ROLLED AT 335°C DOWN TO 0.003 INCH.



The following experiment was also attempted. Sheets of uranium of varying thickness, 0.010, 0.012, 0.015 and 0.020 inch, were firmly sealed inside a stainless steel sheet envelope, closed at the front and back edges, and open at the sides. The assembly was then rolled at 300°C. The first few passes were satisfactory, but after that the stainless envelope curled severely and the uranium sheet was no longer held firmly in position satisfactory for rolling. The uranium sheet then tore and wrinkled, and rolling was generally unsatisfactory.

Good results were obtained in an experiment where a sheet of U-235 metal 0.001 inch thick was required. Starting with a small section of casting 0.250 x 2 x 6 inches, the metal was rolled directly from the as-cast state down to 0.005 inch sheet from a Meltemp bath at 300°C. It was thought that heating such thin sheet would be of no value because it would be instantly chilled to roll temperature upon entering the rolls. It was therefore decided to roll the 0.005 inch sheet at room temperature on the 4-High Standard mill using work rolls heated to 100°C.

No intermediate anneals were used, and the metal edge cracked slightly during rolling so that shearing was necessary every 25 to 30 passes. At one point, the sides were sheared by hand following the general contour of the cracks so that the two sides of the specimens were both convex in contour instead of straight and parallel as formerly. After that, it was possible to take approximately 400 additional passes with absolutely no further edge cracking, and the metal reduced to 0.001

inch thickness with good surface and no pin-point holes in evidence.

It can thus be seen that, should thin uranium sheet or foil be required in large amounts, one of the special mills for rolling thin sheet (such as the Sendzimir, the Steckel or the Y-mill) would be desirable. If none of these mills were available, intermediate annealing at approximately every 0.005 inch reduction below the efficient operation of the mill used would be advisable.

Preparation of Sheet for Warm Rolling

Other than removing salt adhering to the sheet from the hot rolling operations, using warm water and a steel or brass wire brush and then drying thoroughly, no particular prepration of hot rolled sheet for warm rolling was found necessary.

The thin, tightly adhering oxide film formed on the surface of the uranium metal by the salt bath remained continuous throughout the hot rolling and warm rolling procedures and afforded excellent protection from oxidation and mechanical losses. Overall loss of metal during the entire rolling operation remained under 0.5 percent, as mentioned before.

On several occasions after hot rolling, the oxide film was stripped in a nitric acid bath in order to examine the sheet surface. The hot rolled surface at 12X, 24X and 48X magnification was always found good, free from macro and micro cracks, and was quite suitable for warm rolling. After good quality sheet was produced consistently by hot and warm rolling, the practice of examining the sheet surface under the

viewing scope after hot rolling was considered unnecessary.

Rolling Temperatures

In preparation for warm rolling, the sheet was heated in a Meltemp No. 7 oil bath at temperatures ranging from 300° to 340°C, the higher temperatures usually being considered preferable. Sheet from 0.100 up to 0.200 inch in thickness was heated in the oil bath for approximately five minutes, which was considered sufficient time to reach bath temperature. The sheet was reheated between passes.

Thinner gauge stock from 0.100 down to 0.010 inch was given less time in the bath and usually reheated between passes for one minute or even less, depending on thickness. It was considered that thin sheet around 0.010 inch would reach bath temperature within a matter of a few seconds.

Handling Time

Handling time is more important in warm rolling than in hot rolling. At thin gauges the sheet tends to lose heat quite rapidly in air after extraction from the oil bath, and prompt insertion into the rolls was made part of standard procedure.

The mobile heating table shown in Figure 14 helped in speedy handling, as it could be wheeled up to the feed table of the 4-High Loewy mill and the sheet could then be handled with a minimum of elapsed time.

Handling time to return the sheet to the oil bath for reheating after each pass was not critical, since the thin sheet heated to temperature

quickly, and little time would be lost, such as might occur in hot rolling where a heavy section, once cooled off, would require five minutes or more to again reach rolling temperature.

Sufficient time was taken after each pass to record finishing temperatures of the sheet, measure its thickness and the increase in length and width, and check surface and edges carefully.

Work Roll Temperatures

Evidence in respect to the effect of heated work rolls on the surface texture of warm rolled sheet is inconclusive thus far. Preliminary experiments on sheet warm rolled on the Standard 4-High mill with cold work rolls resulted in fine surface cracks, many of the cracks hair-like and visible only under the viewing scope at 12X magnification or better. After several such observations, the rolls on the 4-High Standard mill were equipped with electrical resistance strip heaters, which enabled work roll temperatures up to 100°C to be attained.

With the work rolls thus heated, thin gauge sheet of both normal and high purity uranium was produced which possessed excellent surface qualities of smoothness and freedom from macro and micro cracks under the viewing scope. It was therefore believed that heating the work rolls prevented chilling of the surface of sheet being warm rolled and thus prevented formation of the fine cracks previously noted.

However, several dozens of similar rolling experiments on the 4-High Loewy mill, which had no provision for heating the work rolls,

also produced sheet of good surface and edge qualities down to 0.003 inch in thickness.

It may be, therefore, that heated work rolls are beneficial in warm rolling, certainly as far as keeping surface temperatures higher and increasing ease of reduction are concerned. But as far as the final goal is concerned, that of producing sheet of good surface quality and good forming characteristics, heated work rolls do not appear to be necessary.

Roll Speeds

Both of the 4-High mills on which warm rolling was done were equipped with constant speed AC motor drives, and it was not possible to vary roll speed to determine its effect. Both mills gave fairly high delivery speeds, 40.5 and 98 feet per minute, and though the latter speed was feared to be too fast, uranium sheet rolled easily and well without undue tearing, wrinkling, or edge cracking, and final sheet properties were satisfactory. Whether or not slower roll speeds would give greater ease of deformation or result in more uniform deformation and better sheet properties is questionable. From present experience, slower roll speeds do not appear to offer any advantage.

Rolling Direction

As uranium has directional properties associated with its crystallography, it was considered advisable in early warm rolling experiments to cross-roll the sheet when possible. However, it was

later shown that uranium sheet warm rolled in one direction did not show any directionality after annealing as indicated by perfect C-shaped breaks in the Olsen cup test and by excellent deep drawing performance, as shown in Figure 36. On the basis of these results, and since rolling in one direction avoided the "dishing" and wrinkling which occurred occasionally on cross-rolling, it was decided to adopt a standard procedure of rolling sheet in one direction only.

Reduction per Pass

Reduction per pass in warm rolling is often limited by the size of the mill and roll pressures available. However, even where large roll pressures are available, or on rolling sheet that is not very wide where roll pressures can greatly exceed deformation pressures required, it was found most desirable to take comparatively small reductions per unit pass; around 10 percent per pass on material of 0.100 inch thickness and up to 15 or 18 percent per pass on thinner sheet. The necessity for taking small reductions per pass was borne out by several experiments in which heavier reductions - in one instance 32 percent and in another 40 percent - were taken. In both instances, the metal deformed with explosive violence, the surface became corrugated like an old-fashioned washboard, and severe edge cracking occurred.

It was presumed that in heavy reductions the metal cannot flow forward toward the free surface fast enough, and piles up on itself. Excessive roll pressures also caused metal to flow to the sides more

than normally, with resultant edge cracking.

A similar phenomenon occurred when hot rolling an as-cast bar of normal uranium directly down to sheet on the Bliss 2-High mill. In hot reducing from 0.045 to 0.029 inch in one pass, a reduction of 36 percent, the metal deformed with explosive violence. The appearance of the sheet is shown in Figure 20.

In most instances during warm rolling, sufficient roll pressures were not available to attain the reduction per pass of 10 to 15 percent desired. Where roll pressures much in excess of those required are available, deformation per pass should nevertheless be limited to 10 or 15 percent to avoid the phenomena described above.

Total Reduction

Unlike hot rolling, the total reduction accomplished during warm rolling is an important factor. Recrystallization studies, which will be given in succeeding paragraphs, show that a definite minimum amount of reduction is necessary to secure complete recrystallization upon annealing.

Depending on the individual metal, impurities, etc., this minimum amount of cold work varies from roughly 75 percent required for complete recrystallization at 500°C down to approximately 60 percent required at 600 and 650°C.

Thus, knowing the final thickness of sheet required, hot rolling must be stopped at a thickness great enough to allow at least

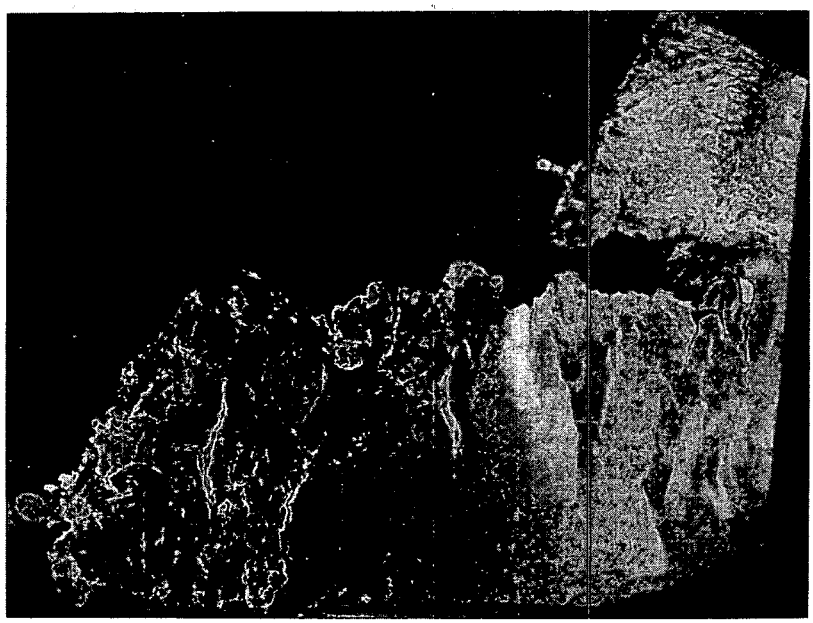


FIG. 20 - EXAMPLES OF URANIUM SHEET ROLLED WITH EXCESSIVE DEFORMATION PER PASS. NOTE FAILURE IN EVENLY SPACED, WASHBOARD PATTERN. MAGNIFICATION APPROXIMATELY 2X.

60 to 75 percent or more warm reduction, if recrystallization and maximum physical properties of the final sheet are important. Of course, if sheet of low properties or nuclear properties only is required, the above figures on total reduction need not apply.

Summary of Techniques in Rolling

To summarize briefly, the easiest and best procedure thus far developed for hot breakdown and warm finishing of uranium metal to sheet is as follows:

Hot Breakdown of As Cast Ingot or Forged Bar

1. Roll convenient size bar or billet. No prior anneal or surface preparation necessary for as cast ingots prepared in smooth graphite plate molds. Metal cast in cylindrical or corrugated molds and forged to plate suitable for rolling should be carefully inspected for laps, folds, and similar imperfections which may be imparted by poor forging technique.

2. Use a heating medium to provide rolling temperatures around 600°C . $Li_2 CO_3$ - K_2CO_3 (35-65) found best thus far.

3. Roll temperatures - may be at room temperature, or heated if thought desirable to obtain higher ambient rolling temperatures and somewhat greater ease of reduction.

4. Roll speeds - not critical. Depend on mill available, size of billets rolled, and specific requirements.

5. Rolling direction - two or three transverse passes are

believed necessary to avoid directionality in finished sheet. More not required.

6. Reduction per pass - 10 to 15 percent sufficient. Larger reduction is satisfactory if mill capacity is available.

7. Total reduction - breakdown hot as far as feasible depending on roll efficiency, but leaving sheet thick enough to allow 60 to 75 percent warm reduction, so complete and uniform recrystallization can be obtained.

(No intermediate anneal between hot and warm rolling required. Merely wash salt from surface or clean off other heating media used. No other surface preparation necessary).

Warm Rolling

1. Roll at a temperature as high as possible below recrystallication range, around 300-350°C, in oil or similar medium for ease of reduction and good surface and edge properties. Warm rolling around 225°C is also satisfactory, but more difficult, and gives higher tensile properties.

2. Roll temperatures - may be cold or heated, as desired. Heated rolls preferable for thin gauge sheet to prevent surface cracks occurring when thin gauge sheet is suddenly chilled by cold work rolls.

3. Roll speeds - apparently not critical, may be fast or slow as desired.

4. Rolling direction - continuous longitudinal passes satisfactory. Transverse warm rolling not necessary, as cross rolling during

hot breakdown appears to eliminate directionality in finished sheet.

5. Reduction per pass - do not exceed 10 to 20 percent per unit pass to avoid washboarding of surface and edge cracking.

6. Total Reduction - at least 60 and preferably 70 to 80 percent warm reduction necessary if controlled recrystallization and maximum properties for fabrication are required.

7. Intermediate annealing - may be necessary at thin gauges after sheet becomes work hardened and deformation pressures exceed available roll pressures.

8. Handling time - process sheet from heating bath to rolls as rapidly as is convenient to prevent loss of heat enroute.

Several dozens of tests using the above techniques have consistently produced good quality sheet free from surface and edge defects, and possessing no directional properties as evidenced by good deep drawing characteristics, perfect C-shaped breaks in the Olsen cup test, and substantiated by x-ray crystallographic studies.

Annealing time and temperature have been standardized at 575°C for twenty minutes in the salt bath of 65% Li_2CO_3 and 35% $K2CO_3$. This annealing procedure consistently gives uniform, equiaxed grains whose average diameter is 15-20 microns. This grain size appears to give the best combination of strength and ductility for deep drawing, cupping, stretching, and other means of fabrication.

Properties of Rolled Uranium Sheet

Using the techniques outlined above, rolled uranium sheet possessed good surface (Figure 21) and edge properties. Other properties and characteristics are noted.

Hardness

The hardness of both normal and high purity uranium in the as-cast state was found to average 57 Rockwell "A". This value was confirmed by upwards of one hundred hardness readings on forty-eight separate specimens.

As was to be expected, the hardness of uranium increased with increasing amounts of cold work. Figure 22 shows the relationship between the two variables, starting with 57 Rockwell "A" in the as cast condition and increasing to a maximum of 73 Rockwell "A" at 92 percent warm reduction.

The hardness of uranium drops off in typical fashion after annealing warm rolled specimens. Figure 23 shows a series of hardness readings taken on two samples of normal uranium. One sample was hot rolled 90 percent, then warm rolled 70 percent; the other sample was hot rolled 93 percent, then warm rolled 60 percent. Separate specimens of each rolled sheet were then annealed at various temperatures for a period of thirty minutes.

Starting with an as-rolled hardness value of 66 R_A for the specimen warm rolled 70 percent, the course of recrystallization and

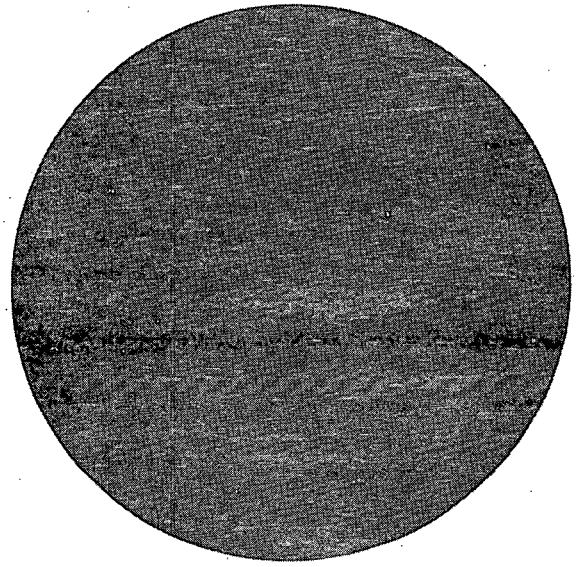
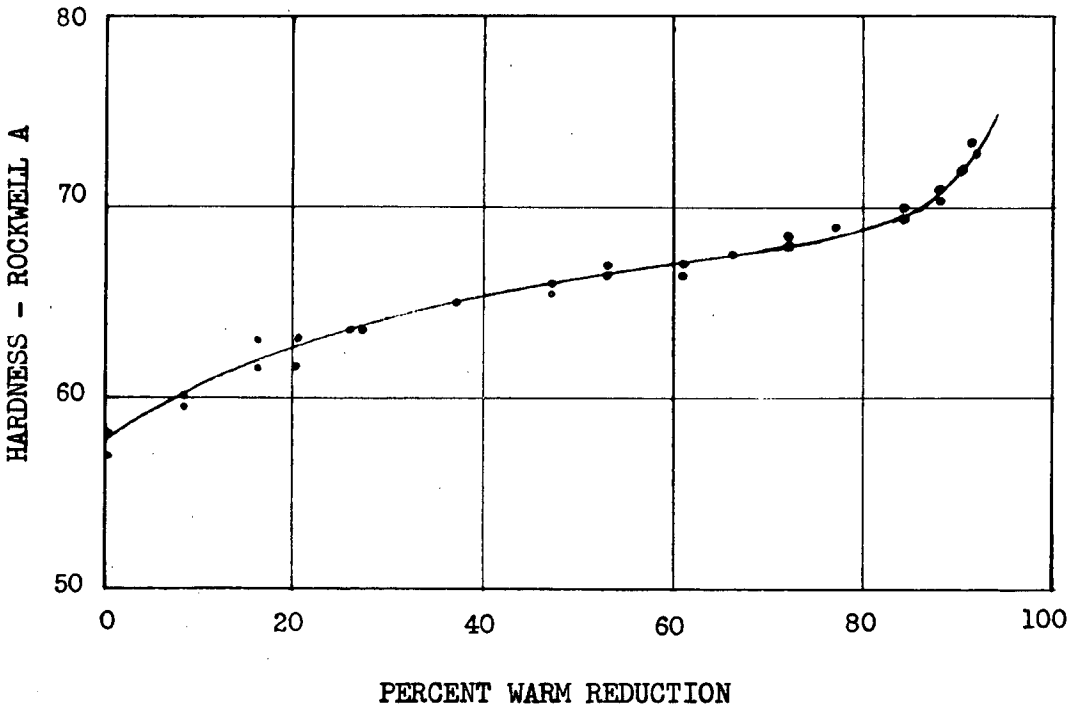


FIG. 21 - SURFACE OF NORMAL TUBALLOY SHEET 3-6A-2 AT 10X. WARM ROLLED 97% WITH WARM WORK ROLLS. SURFACE IS SMOOTH, WITH COMPLETE FREEDOM FROM CRACKS, SEAMS, RIDGES, AND OTHER IMPERFECTIONS.

Fig. 22. HARDNESS OF NORMAL URANIUM SAMPLES AS A FUNCTION OF PERCENT REDUCTION BY WARM ROLLING AT 300°C. SAMPLES ALPHA-ANNEALED PRIOR TO WARM ROLLING. ALL READINGS TAKEN PERPENDICULAR TO PLANE OF ROLLING.



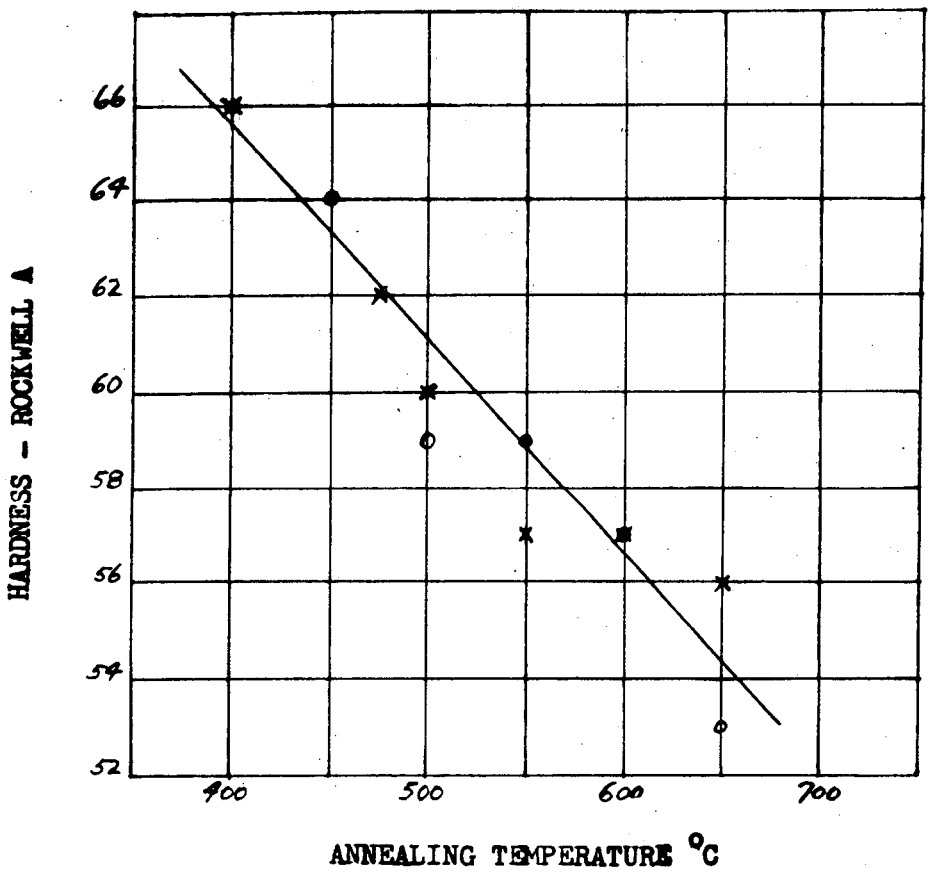


Fig. 23. NORMAL URANIUM SAMPLES AS-CAST, HOT ROLLED AND WARM ROLLED AS INDICATED, THEN ANNEALED IN VACUUM FOR 30 MINUTES AND FURNACE COOLED

o - Hot Rolled 90%, Warm Rolled 70%

x - Hot Rolled 93%, Warm Rolled 60%

grain growth is evidenced by the consistent lowering of hardness values as higher annealing temperatures were employed, finally reaching 53 R_A after annealing for thirty minutes at 650°C in vacuum. The hardness values for the specimen warm rolled only 60 percent, show an almost identical pattern, starting at 66 R_A for the as-rolled specimen and decreasing to 56 R_A for a specimen annealed in vacuum at 650°C for thirty minutes.

The hardness of hot rolled uranium did not increase materially after 20 percent hot reduction had been exceeded. This hardness effect was probably associated with recrystallization and softening of the metal at the temperature concerned. Table II below gives the hardnesses of a normal uranium specimen at various percentages of hot reduction. Rolling was done at 600°C from a salt bath, and the specimen was heated for periods up to 10 minutes between each pass. The heating time was reduced as the specimen became thinner. The hot rolling caused the metal to be in a partially recrystallized state between each pass. That recrystallization occurred is substantiated by the hardness readings, Table II, which remain fairly uniform up to 95 percent hot reduction. Figure 24 is a photomicrograph of the specimen taken at 74 percent hot reduction and shows that the metal was essentially recrystallized, being strained but little in the direction of rolling.

The hardness valuesbelow were taken after quenching into water within 30 seconds after each pass. Each value represents an

Fig. 24 250X
Plate 265-1-1. Electrolytic polish.
Normal uranium under polarized light.
Hot rolled 74% at 600°C. Quenched into
water within 30 seconds after each pass.

average of 10 or more hardness readings all taken on the flat side of the uranium sample normal to the plane of rolling:

Table II

Hardness as Related to Amount of Hot Reduction

Percent Hot Reduction	Hardness Rockwell "A"
0 (As Cast)	54.0
4	54.5
11	58.2
16	59.1
23	57.7
27	59.5
39	58.2
47	58.1
54	58.8
60	59.4
67	59.9
74	59.2
80	58.4
85	60.0
88	59.5
92	60.5
95	60.7

Carbides and Carbide Distribution

As can be seen from all photomicrographs in the following sections, and in particular from Figure 25, all uranium metal, both normal and high purity, contains carbides as inclusions in a matrix of uranium metal. A sample of uranium was mounted under a microscope and a series of hardness readings taken on the carbides imbedded in the surface by the use of a standard Tukon hardness tester. The hardness readings obtained averaged from 800 to 900 Vickers, as compared with 57 Rockwell "A" or approximately 200 Vickers for the uranium matrix. That these carbides are brittle, as well as very hard, can be seen in Figure 25, where careful examination shows the two large patches of carbides visible to be "cracked" or broken up by the rolling process.

The size and distribution of carbides in normal versus high purity uranium metal is worthy of note. The only difference metallurgically or chemically between normal and high purity uranium is the percent of carbon in the analysis; normal uranium containing around 630 parts per million of carbon, while high purity uranium averages about 100 parts per million of carbon. The carbides formed as a result of the presence of carbon tend to fracture and become elongated in the direction of rolling. Figures 26 and 27 show the difference in the number and size of the carbides present in two samples of normal and high purity uranium. The samples were taken from identical sections of similar shaped cast billets and rolled under identical conditions.

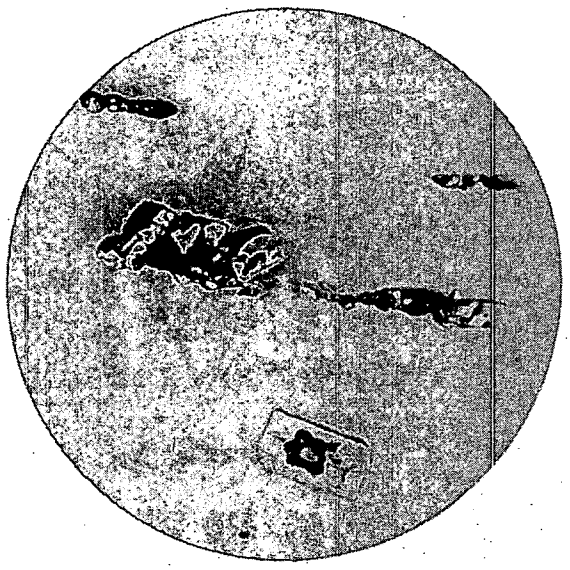


FIG. 25 - NORMAL URANIUM SHEET HOT ROLLED IN SALT (600°C) 680 TO 49 MILS, 92% REDUCTION, WARM ROLLED IN SILICONE (280°C) 49 TO 20 MILS, 60% REDUCTION, ANNEALED 30 MINUTES AT 650 C. MAGNIFICATION 1000X. UNETCHED SURFACE SHOWS "CRACKED CARBIDES".

Figure 26A shows a transverse section of normal uranium hot rolled 90 percent and warm rolled 72 percent. The photomicrograph is that of a typical cold worked structure elongated in the rolling direction. Another normal uranium sample, Figure 26B, was hot rolled 92 percent and warm rolled only 8 percent. This sample was recrystallized during hot rolling and the 8 percent cold reduction has not yet distorted or broken up the uniform, equi-axed grains formed during recrystallization. A photograph of the carbide distribution in the normal uranium samples is shown in Figure 26C. Average carbide size is No. 5*, considerably larger than the No. 8 average carbide size of the high purity sample shown in Figure 27A. But the number of carbides present, Figure 26C, is of the same order of magnitude as the number of carbides in the high purity metal, Figure 27B, thus giving a significantly larger volume of carbides per unit mass of uranium in the normal sample as opposed to the high purity sample.

The same condition is also illustrated in Figure 28, which shows micrographs of normal and high purity samples taken from similar positions in the cast ingot and warm rolled at 300°C under identical conditions to a 97 percent reduction.

The micrograph of the normal specimen, Figure 28A, shows considerably larger carbide inclusions than are shown in the high purity specimen, Figure 28B. Both of these pictures were taken parallel to

*For discussion on origin of carbide sizes, see Appendix A.

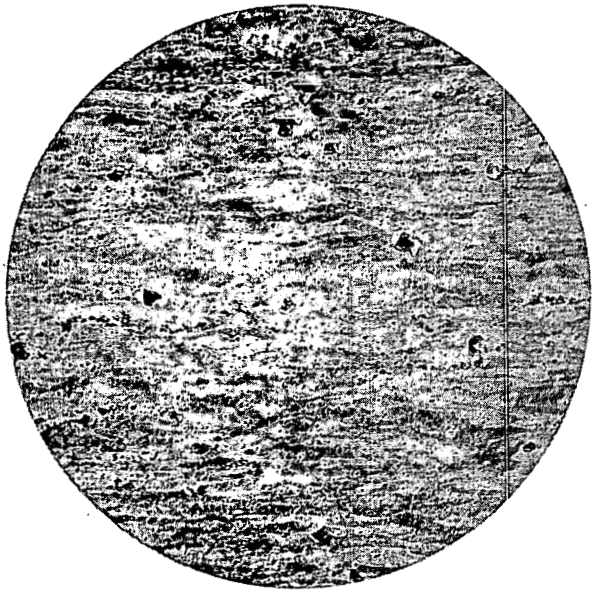


FIG. 26A - PLATE J116-2-1. MAG. 250X. TRANSVERSE SECTION. NO ETCH; POLARIZED LIGHT. SAMPLE 3-2B-3, NORMAL PURITY URANIUM. HOT BREAKDOWN 90%, WARM FINISH 72%, NOT ANNEALED.

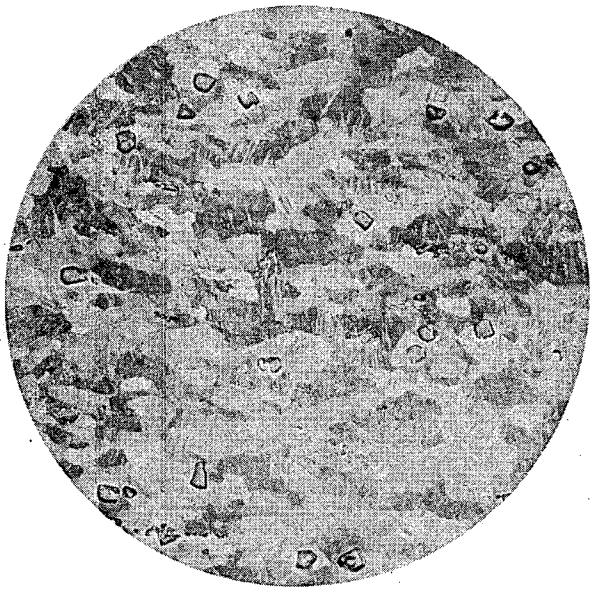


FIG. 26B - PLATE NO. J164-2-1. MAG. 250X.
LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT.
3-6B-1, NORMAL PURITY U HOT ROLLED 92%, WARM
ROLLED 8%. GRAINS AVERAGE 0.010 MM BY 0.025 MM.

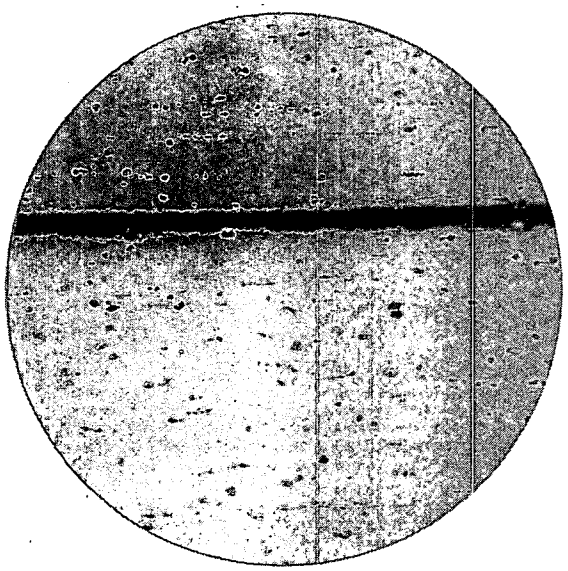


FIG. 26C - PLATE J116-2-2. MAG. 100X. TRANSVERSE SECTION. NO ETCH; BRIGHT FIELD. 3-2B-3. AVERAGE CARBIDE SIZE #5. TWO FLAT SPECIMENS MOUNTED TOGETHER WITH DIVIDING LINE RUNNING HORIZONTALLY ACROSS MICROGRAPH.

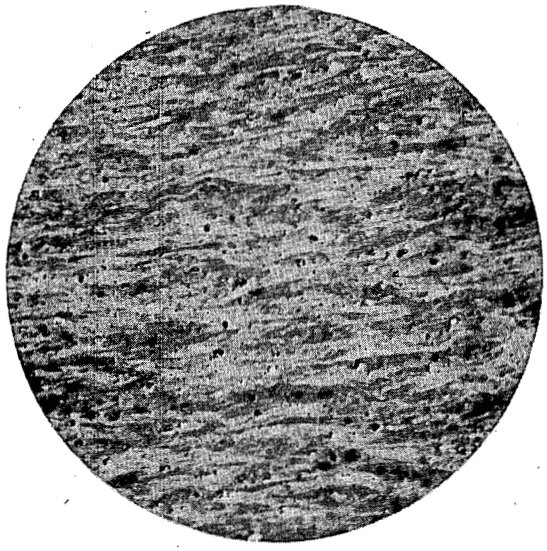


FIG. 27A - PLATE J116-1-2. MAG. 250X. TRANSVERSE SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3, HOT BREAKDOWN 90%, WARM FINISH 72%, NOT ANNEALED. HIGH PURITY URANIUM.

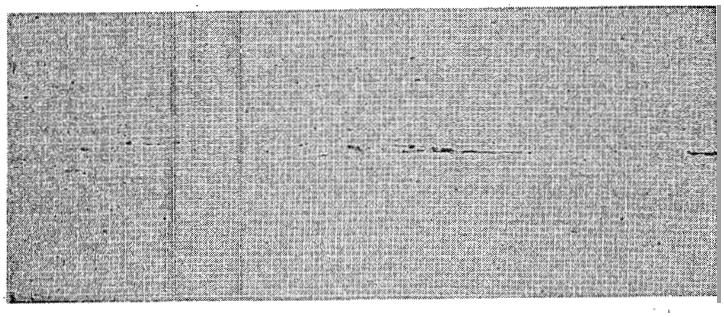


FIG. 27B - PLATE J116-1-1. MAG. 100X. TRANSVERSE SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3. AVERAGE CARBIDE SIZE #8.

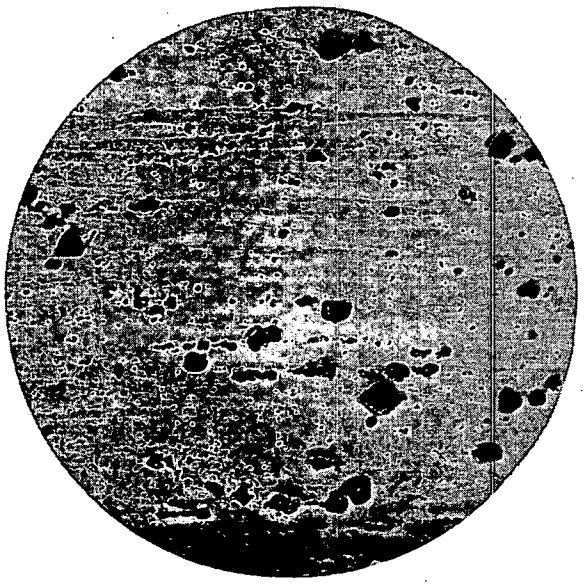


FIG. 28A - PLATE NO. J128-2-2. SAMPLE 3-6A-2. NORMAL PURITY URANIUM WARM ROLLED 97%, 300°C, AS ROLLED. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. NOTE THE GREATER SIZE AND VOLUME OF CARBIDE INCLUSIONS, AS COMPARED WITH THE SIMILAR HIGH PURITY SAMPLE BELOW. MAGNIFICATION 250X.

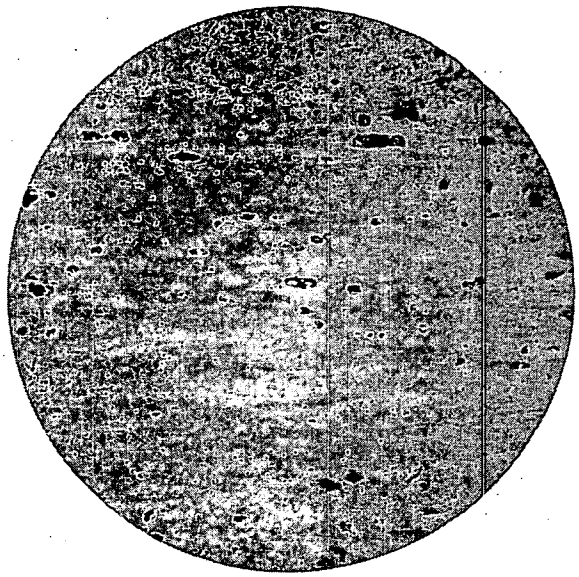


FIG. 28B - PLATE NO. J128-4-2. SAMPLE 8-7C-2. HIGH PURITY URANIUM, WARM ROLLED 97%, 300°C, AS ROLLED. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. MAGNIFICATION 250X.

the direction of rolling and the carbides can be seen elongated and strung out parallel to the direction of rolling. The difference in the size of carbides between normal and high purity uranium is similarly true for all comparisons made thus far in these experiments.

In some cases, the difference in carbide size or number and distribution was not as striking as in the micrographs selected above. In a few series, very little difference in carbide size could be noted between the normal and high purity metal. This size similarity might be ascribed to the fact that the region examined was not truly representative of the sample; perhaps an area of unusually high density of carbides on the high purity sample, or conversely, an area of unusually low density of carbides in the normal uranium sample.

As far as could be determined, the amount of carbides normally present in normal and high purity samples did not affect rolling procedure, surface condition, or other gross properties of the samples checked.

Recrystallization and Annealing of Rolled Uranium Samples

It was thought that the size or distribution of carbides might, however, influence the recrystallization properties of uranium in that the rate of recrystallization or grain size may be affected by the presence of impurities.

Theoretically, one might suppose that the larger number of carbides often associated with high purity uranium, even though smaller

in size, should afford a greater number of sites for nucleation, and that recrystallization would start earlier and progress further in high purity samples. Although one series of micrographs could be used to support this hypothesis, other series showed no difference in recrystallization properties of normal versus high purity uranium, and some actually showed normal uranium recrystallizing faster than similarly treated high purity samples.

This latter behavior can also be explained by the assumption that the larger sized carbides found in normal uranium cause greater strain in the surrounding uranium matrix and result in high energy areas which trigger nucleation.

Figures 29 and 30 show the recrystallization behavior of normal and high purity uranium both rolled and annealed together under identical conditions at different annealing temperatures.

Figure 29 is a series of photomicrographs of a normal uranium specimen hot rolled 90 percent, warm rolled 72 percent, and sections annealed at 500, 550, 600, 650, and 700°C in vacuum for thirty minutes. Figure 29A shows recrystallization well started, though the banded, worked structure is still in evidence. After annealing at 550°C, Figure 29B, recrystallization is almost complete, and the grains have grown slightly . At 600°C for thirty minutes, Figure 29C, recrystallization is complete and the grains have grown till the average diameter is 0.015 mm. A section annealed at 650°C, Figure 29D, shows grain growth reaching an average grain diameter of 0.020 mm. The section annealed at 700°C for

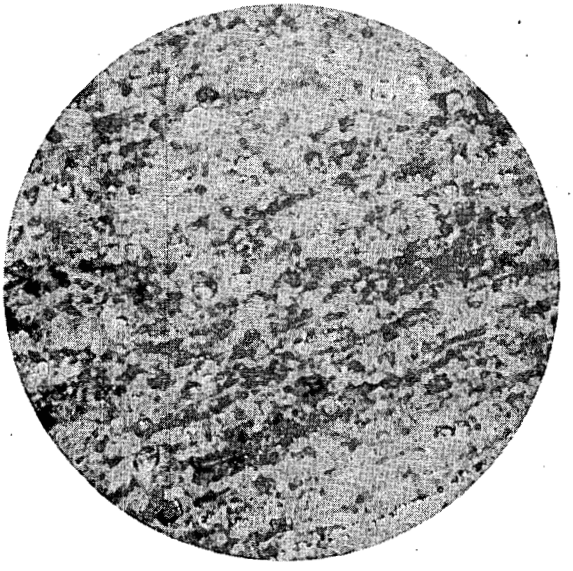


FIG. 29A - PLATE J127-12-1. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 3-2B-3E. NORMAL PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 500° FOR ½ HOUR. RECRYSTALLIZATION WELL STARTED. AVERAGE GRAIN DIAMETER 0.0075 MM.

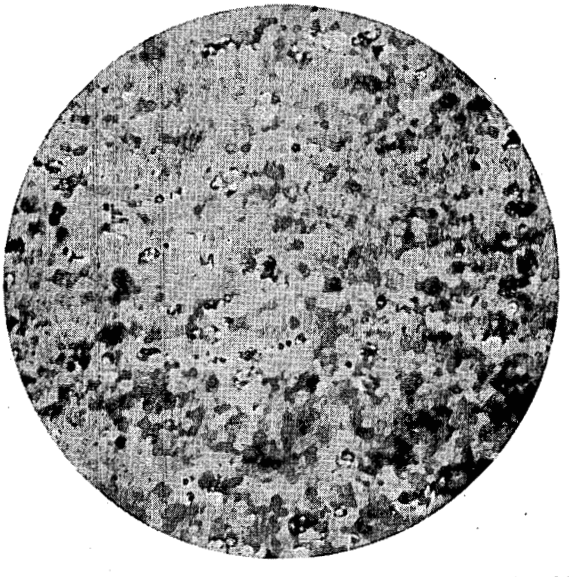


FIG. 29B - PLATE J127-10-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 3-2B-3D. NORMAL PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 550°C FOR ½ HOUR. RECRYSTALLIZATION ALMOST COMPLETE. AVERAGE GRAIN DIAMETER 0.0075 MM.

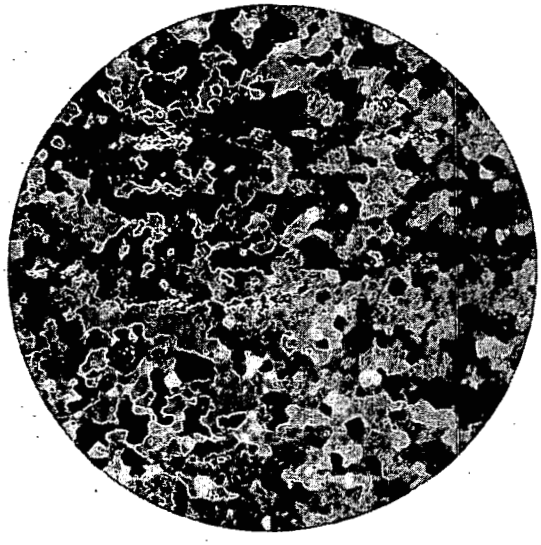


FIG. 29C - PLATE J135-1-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 3-2B-3C, NORMAL PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR ½ HOUR. AVERAGE GRAIN DIAMETER 0.015 MM.

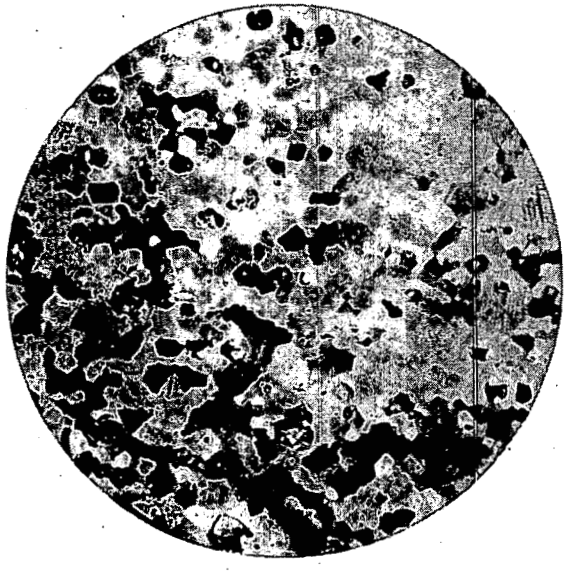


FIG. 29D - PLATE J127-8-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 3-2B-3B, NORMAL PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 650°C FOR ½ HOUR. RECRYSTALLIZATION COMPLETE. AVERAGE GRAIN DIAMETER 0.020 MM.

one-half hour, Figure 29E, shows the enormous grains that occur, presumably due to heating into the beta range.

For purposes of comparison, high purity metal was treated in precisely the same manner as the normal tuballoy described in the paragraph immediately above. Annealed at 500°C for one-half hour, Figure 30A, no recrystallization is evident in the high purity sample, though recrystallization was well under way in its corresponding normal sample. At 550°C, Figure 30B, recrystallization is almost complete, and the grains have grown to a size of 0.0075 mm, and same size as in the corresponding normal sample.

At 600 and 650°C, Figures 30C and 30D respectively, recrystallization is complete, but grain growth is not as great as in the corresponding normal samples, the grain sizes being 0.010 mm at 650°C, as compared to 0.020 mm for the normal metal. These results do not conform to theory, for grain growth should increase as the annealing temperature is raised, and the high purity sample exhibits the same grain size at the 600 and 650°C annealing temperatures. There may be some internal effect which influenced the grain size and which is not presently understood. Upon annealing a sample at 700°C, large grains were formed, Figure 30E, similar to those of the normal sample.

From a comparison of grain size with annealing temperature for both normal and high purity samples, it appears that grain size increases linearly with annealing temperature, and one is provided with a very valuable technique, that of controlling grain size by control of annealing

FIG. 29E - PLATE J149-1-3. MAG. 250X. SECTIONED ALONG A RADIUS. NO ETCH; POLARIZED LIGHT. 3-2B-3A, NORMAL U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 700°C FOR ½ HOUR. AV. GRAIN DIAMETER 0.200 MM.

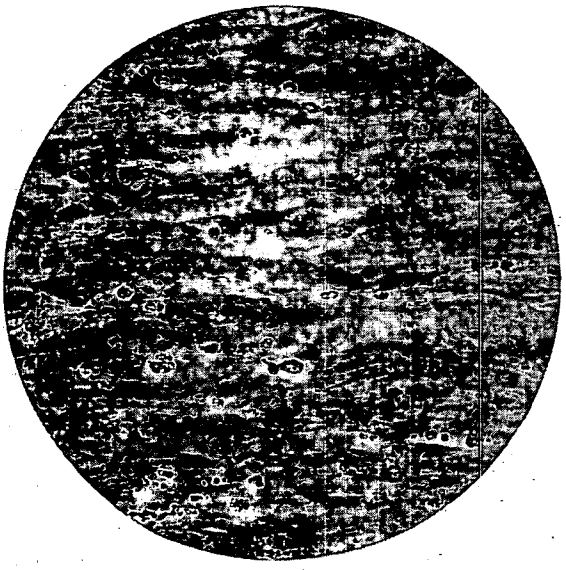


FIG. 30A - PLATE J127-5-2. MAG. 250X. TRANSVERSE SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3E, HIGH PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 500°C FOR ½ HOUR. NO RECRYSTALLIZATION.

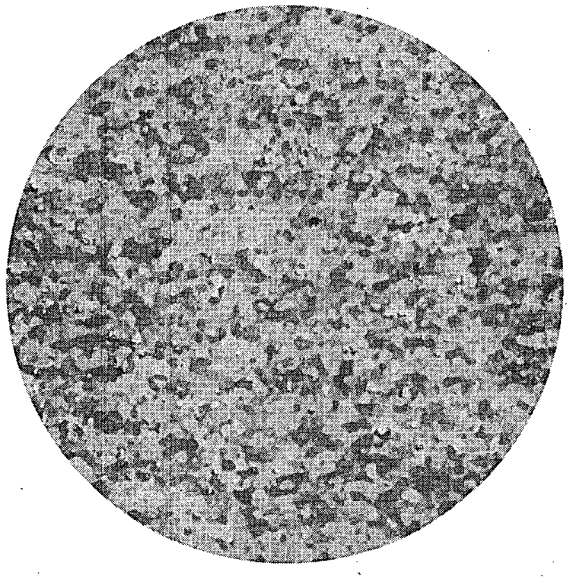


FIG. 30B - PIATE J127-4-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3D. HIGH PURITY U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 550°C FOR ½ HOUR. RECRYSTALLIZATION ALMOST COMPLETE. AVERAGE GRAIN DIAMETER 0.0075 MM.

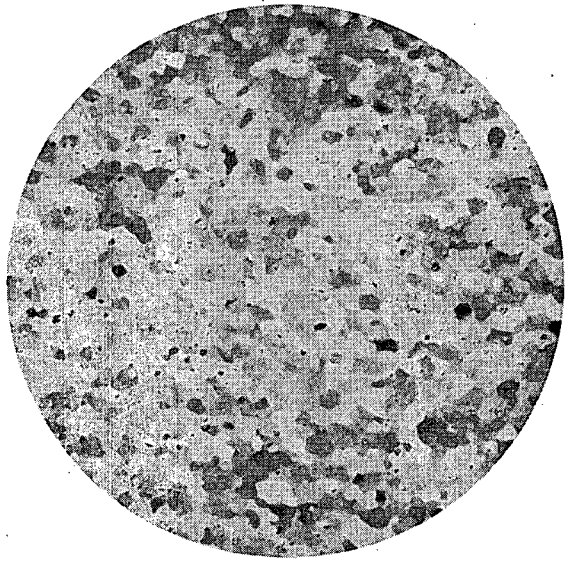


FIG. 30C - PLATE J116-3-1. MAG. 250X. TRANSVERSE SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3C. HOT BREAKDOWN 90%, WARM FINISH 72%, HI-PURITY U ANNEALED 600°C ½ HOUR. AVERAGE GRAIN DIAMETER 0.010 MM.

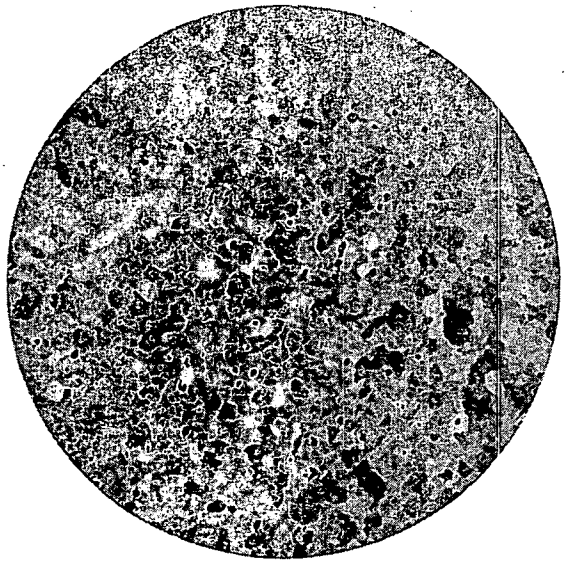


FIG. 30D - PLATE J127-2-1. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3B. HIGH PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 650°C FOR ½ HOUR. RECRYSTALLIZATION COMPLETE. AVERAGE GRAIN DIAMETER 0.010 MM.

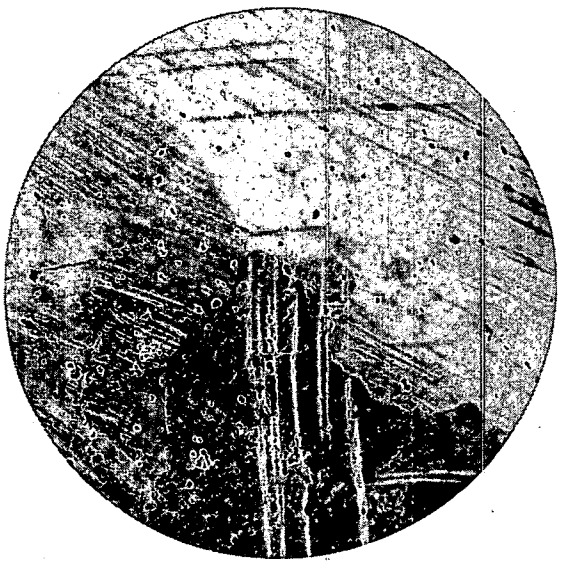


FIG. 30E - PLATE J149-2-2. MAG. 250X. SECTIONED ALONG A RADIUS. NO ETCH; POLARIZED LIGHT. 8-2B-3A. HIGH PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 700°C FOR ½ HOUR. AVERAGE GRAIN DIAMETER 0.300 MM.

temperature. Thus, if a sufficient amount of cold work is given uranium (with 60 percent being a rough minimum), it is possible to secure a desired grain size by proper choice of annealing time and temperature.

A similar series of micrographs showing recrystallization is shown in Figure 31. These pictures were taken of normal uranium samples hot rolled 92 percent, then warm rolled 60 percent. The first micrograph, Figure 31A, shows the structure after hot rolling 92 percent at 600°C. Some recrystallization has occurred, but it is by no means complete, and evidence of the worked structure remains. The second micrograph, Figure 31B, shows the severely worked structure remaining after 60 percent warm rolling was done. Annealing at 400°C for thirty minutes caused no recrystallization, Figure 31C, nor did annealing at 475°C, Figure 31D, or at 500°C, Figure 31E, for thirty minutes. It was not until 550°C had been reached, Figure 31F, that recrystallization occurred, showing how a decrease in the amount of cold work displaced recrystallization to higher annealing temperatures. At 600 and 650°C, Figures 31G and 31H respectively, recrystallization is complete, and grain growth has been under way, resulting in an average grain diameter of 0.035 mm at 650°C.

When a sample has been annealed at 700°C for thirty minutes, Figure 31J, the huge recrystallized grains previously noticed have again been formed.

FIG. 31A - PLATE 105-1-1. MAGNIFICATION 250X. SECTION PARALLEL TO ROLLED SURFACE. STANDARD URANIUM ETCH. SAMPLE 1-7-B-9, NORMAL PURITY URANIUM HOT ROLLED (600°C) 92% REDUCTION. RECRYSTALLIZATION STARTED. EVIDENCE OF WORKED STRUCTURE REMAINS.

FIG. 31B - PLATE 106-5-2. MAG. 250X. SECTION PARALLEL TO ROLLED SURFACE. POLARIZED LIGHT. 1-7B-8, NORMAL PURITY U HOT ROLLED (600°C) 92% REDUCTION. WARM ROLLED (280°C) 60% REDUCTION. SEVERELY WORKED STRUCTURE.

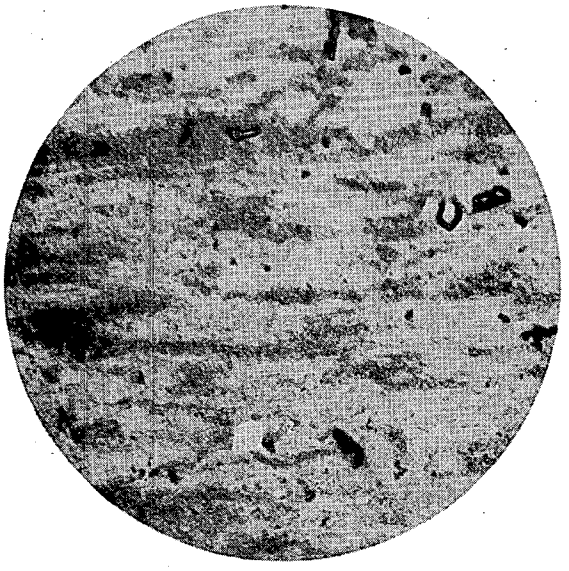


FIG. 31C - PLATE 106-4-1. MAG. 250X. SECTION PARALLEL TO ROLLED SURFACE. STANDARD U ETCH. 1-7B-7, NORMAL PURITY U HOT ROLLED (600°C) 92% REDUCTION. WARM ROLLED (280°C) 60% REDUCTION. ANNEALED AT 400°C FOR 30 MINUTES.

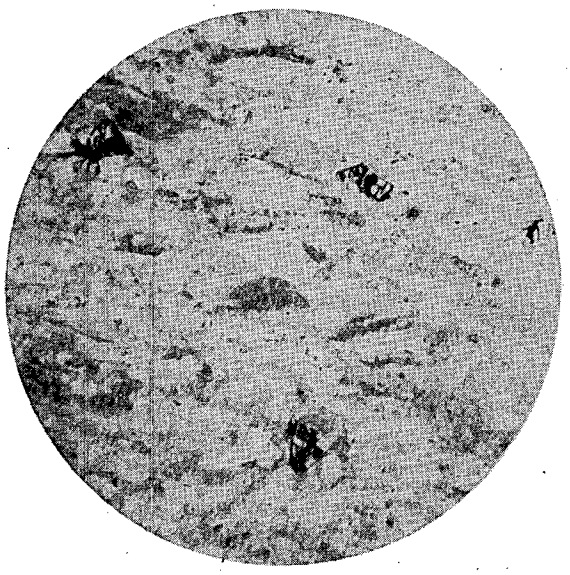


FIG. 31D - PLATE 105-7-1. MAG. 250X. SECTION PARALLEL TO ROLLED SURFACE. STANDARD U ETCHANT. 1-7B-6. NORMAL PURITY U HOT ROLLED (600°C) 92% REDUCTION. WARM ROLLED (280°C) 60% REDUCTION; ANNEALED AT 475°C FOR 30 MINUTES. AVERAGE GRAIN DIAMETER 0.025 MM.

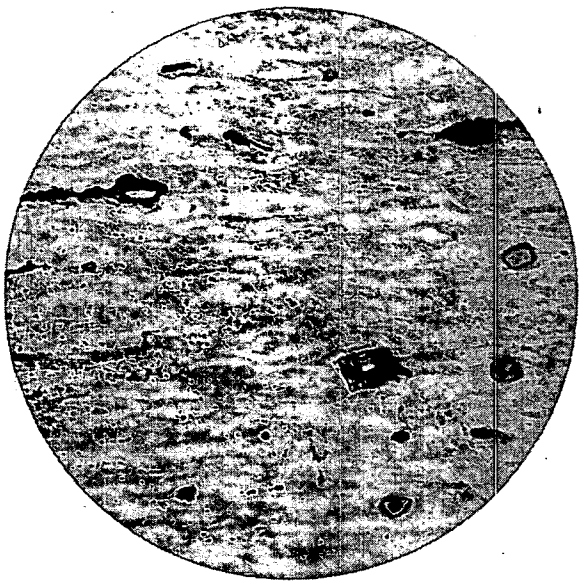


FIG. 31E - PLATE 105-5-3. MAG. 250X. TRANSVERSE SECTION; STANDARD U ETCH. 1-7B-5, NORMAL PURITY U HOT ROLLED (600°C) 92% RED. WARM ROLLED (280°C) 60% RED. ANNEALED AT 500°C FOR 30 MIN. FIELD SHOWING AREA OF PRACTICALLY NO RECRYSTALLIZATION.

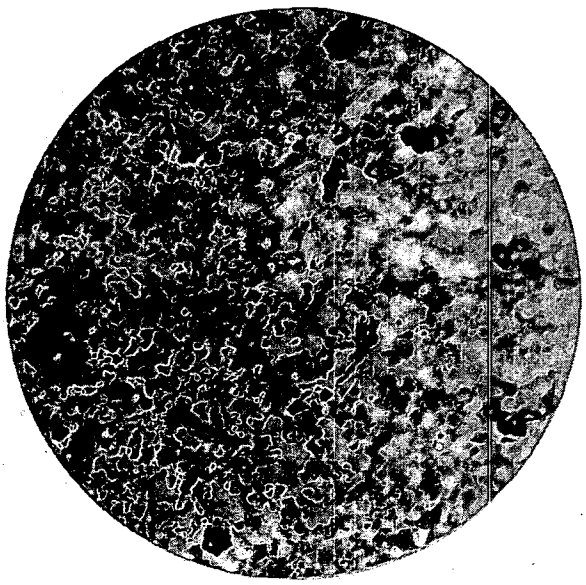


FIG. 31F - PLATE 106-1-2. MAG. 250X. SECTION PARALLEL TO WORKED SURFACE. STANDARD U ETCH. 1-7B-4, NORMAL PURITY U HOT ROLLED (600°C) 92% RED. WARM ROLLED (280°C) 60% RED. ANNEALED AT 550°C 30 MIN. GRAIN SIZE 0.010 MM. COMPLETE RECRYSTALLIZATION.

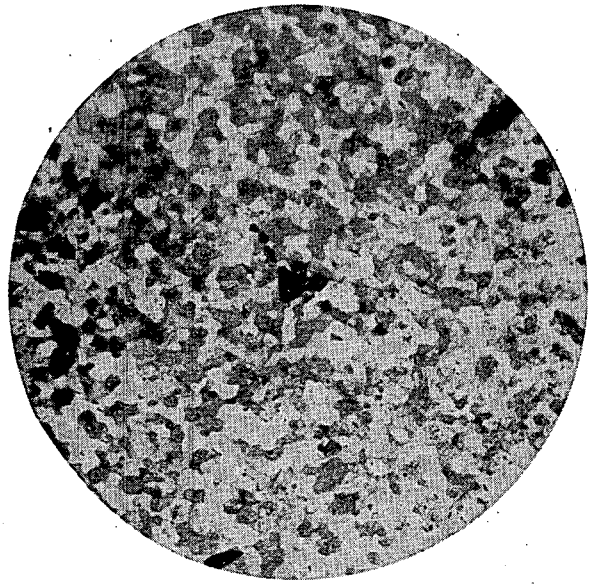


FIG. 31G - PLATE 105-6-2. MAG. 250X. SECTION PARALLEL TO ROLLED SURFACE. STANDARD U ETCH. 1-7B-3. NORMAL PURITY U HOT ROLLED (600°C) 92% RED, WARM ROLLED (280°C) 60% RED. ANNEALED AT 600°C FOR 30 MIN. GRAIN SIZE 0.015 MM. COMPLETE RECRYSTALLIZATION.

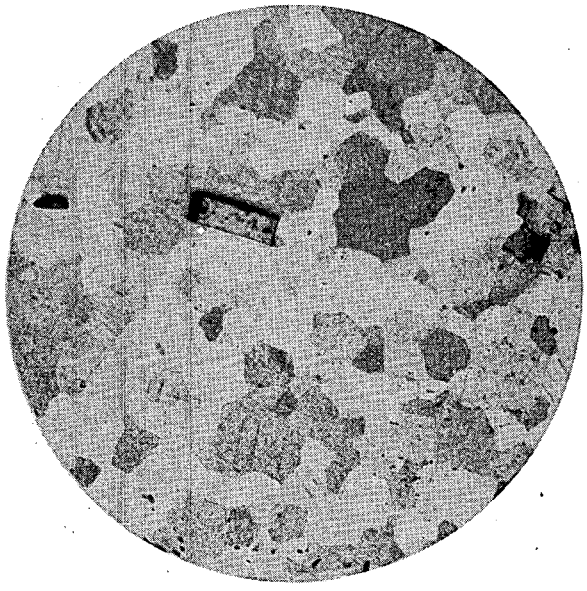


FIG. 31H - PLATE 105-2-2. MAG. 250X. SECTION PARALLEL TO ROLLED SURFACE. STANDARD U ETCH. 1-7B-2, NORMAL PURITY U HOT ROLLED (600°C) 92% RED, WARM ROLLED (280°C) 60% RED. ANNEALED AT 650°C FOR 30 MIN. GRAIN SIZE 0.035 MM.

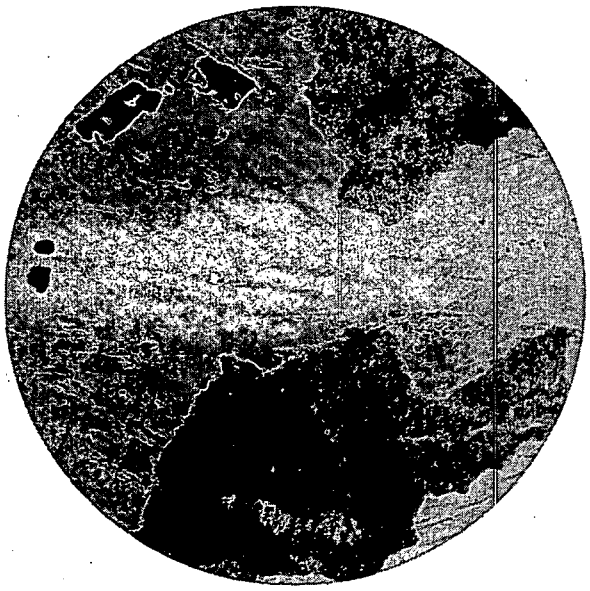


FIG. 31J - PLATE 106-2-1. MAG. 250X. SECTION PARALLEL TO ROLLED SURFACE. STANDARD U ETCH. 1-7B-1, NORMAL PURITY U HOT ROLLED (600°C) 92% RED, WARM ROLLED (280°C) 60% RED. ANNEALED AT 700°C FOR 30 MIN. AV. GRAIN DIA. GREATER THAN 0.200 MM. AV. CARBIDE SIZE #4.

It can again be observed how uniform, equi-axed grains of controlled size can be obtained by regulating the amount of cold work in the metal and the annealing temperature.

Annealing Time

Although most of the annealing experiments were performed in which the annealing time was held constant at thirty minutes and the temperature varied, it was decided to determine if annealing time was a variable and what its importance was. Samples of high purity uranium, hot rolled 90 percent and warm rolled 72 percent, were annealed in the salt bath at 600°C for various times ranging from 1 second, 10, 100, 1000, 1800, to 5400 seconds, after which the samples were quenched in water to arrest recrystallization at the time desired for study.

Figure 32A shows no recrystallization at 1 second, and Figure 32B complete recrystallization at 10 seconds, with grain growth continuing up to 1800 seconds, Figures 32C, 32D and 32E. No additional grain growth is evidenced at 5400 seconds, the last time interval studied, Figure 32F.

Hardness readings taken on samples annealed at the different times stated showed a gradual decrease which paralleled the increase of grain size as one progressed to longer annealing times. The results are tabulated in graph form in Figures 33 and 34.

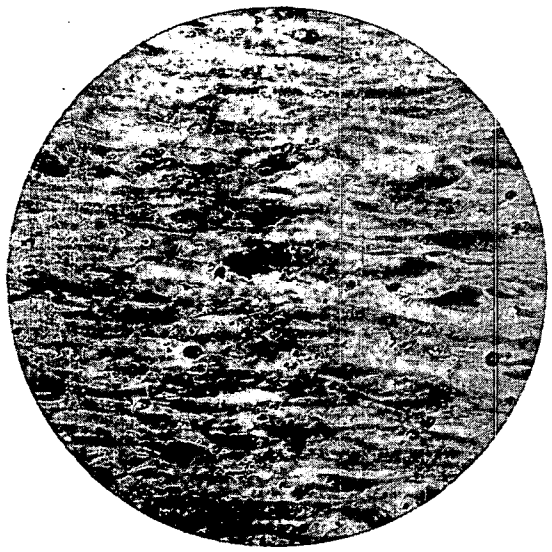


FIG. 32A - PLATE J139-1-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3Z. HIGH PURITY URANIUM HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR 1 SECOND. HARDNESS ROCKWELL B 103. NO RECRYSTALLIZATION.

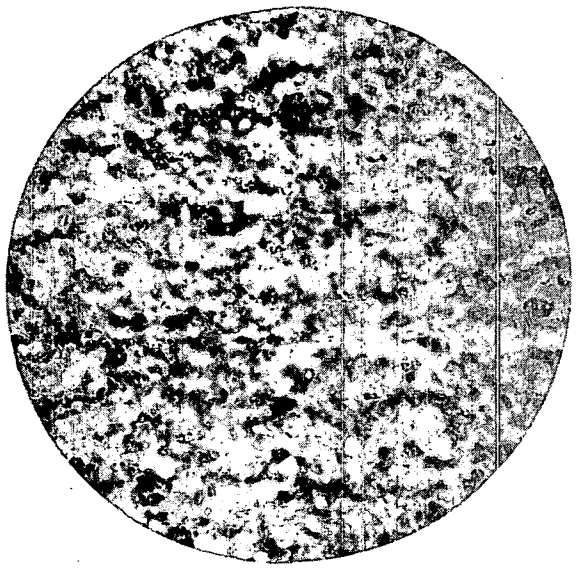


FIG. 32B - PLATE J139-2-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3Y. HI-PURITY U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR 10 SECONDS. HARDNESS ROCKWELL B 80. AVERAGE GRAIN DIAMETER 0.010 MM.

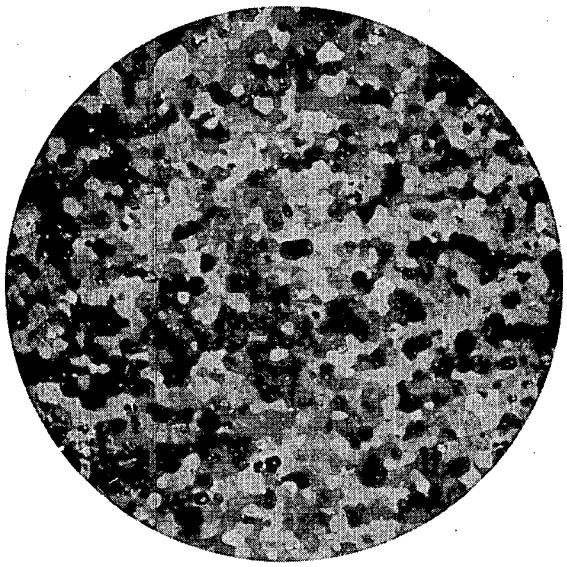


FIG. 32C - PLATE J139-3-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3X. HI-PURITY U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR 100 SECONDS. HARDNESS ROCKWELL B 78. AV. GRAIN DIA. 0.0125 MM.

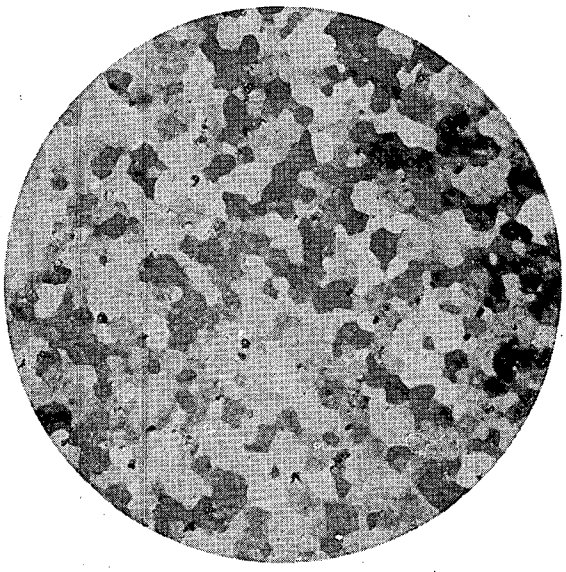


FIG. 32D - PLATE J139-4-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3U, HIGH PURITY U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR 1000 SECONDS. AVERAGE GRAIN DIAMETER 0.015 MM.

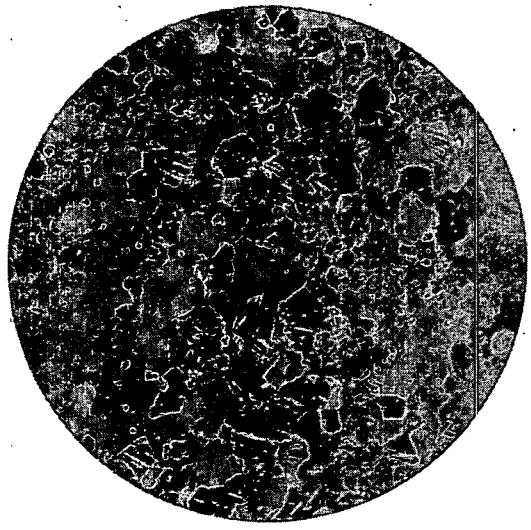


FIG. 32E - PLATE J157-2. MAG. 250X. LONGITUDINAL SECTION; NO ETCH; POLARIZED LIGHT. 8-2B-3. HI-PURITY U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR 1800 SECONDS, QUENCHED. HARDNESS ROCKWELL B 74. AV. GRAIN DIAMETER 0.020 MM. (TWINS THOUGHT DUE TO MECHANICAL POLISHING).

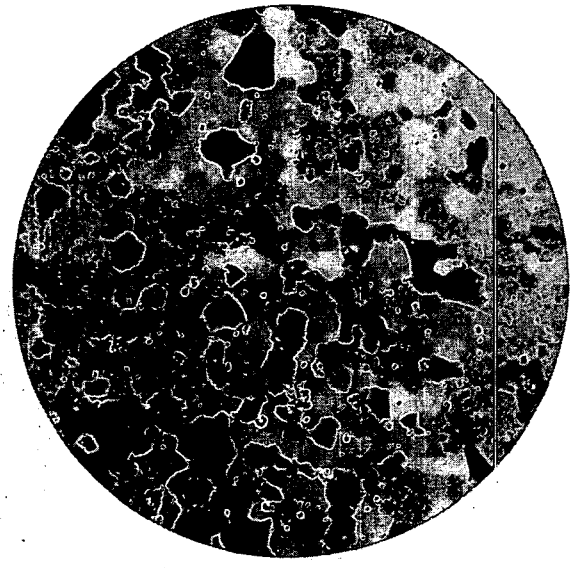


FIG. 32F - PLATE J139-5-2. MAG. 250X. LONGITUDINAL SECTION. NO ETCH; POLARIZED LIGHT. 8-2B-3V. HI-PURITY U HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED AT 600°C FOR 5400 SECONDS. HARDNESS ROCKWELL B 29. AV. GRAIN DIAMETER 0.020 MM.

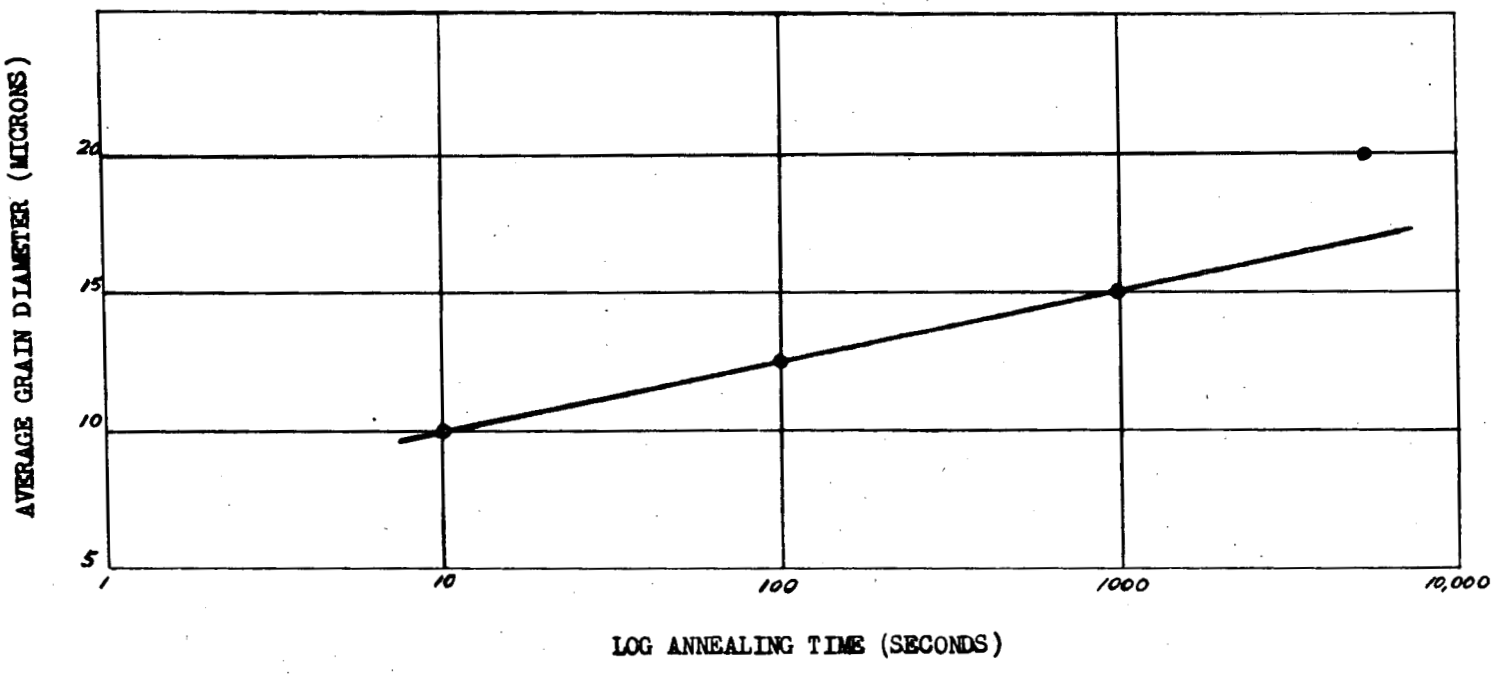


FIG. 33. HIGH PURITY URANIUM SPECIMENS AS-CAST, HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED IN SALT AT 600°C AND QUENCHED INTO WATER AT ROOM TEMPERATURE TO ARREST GRAIN GROWTH.

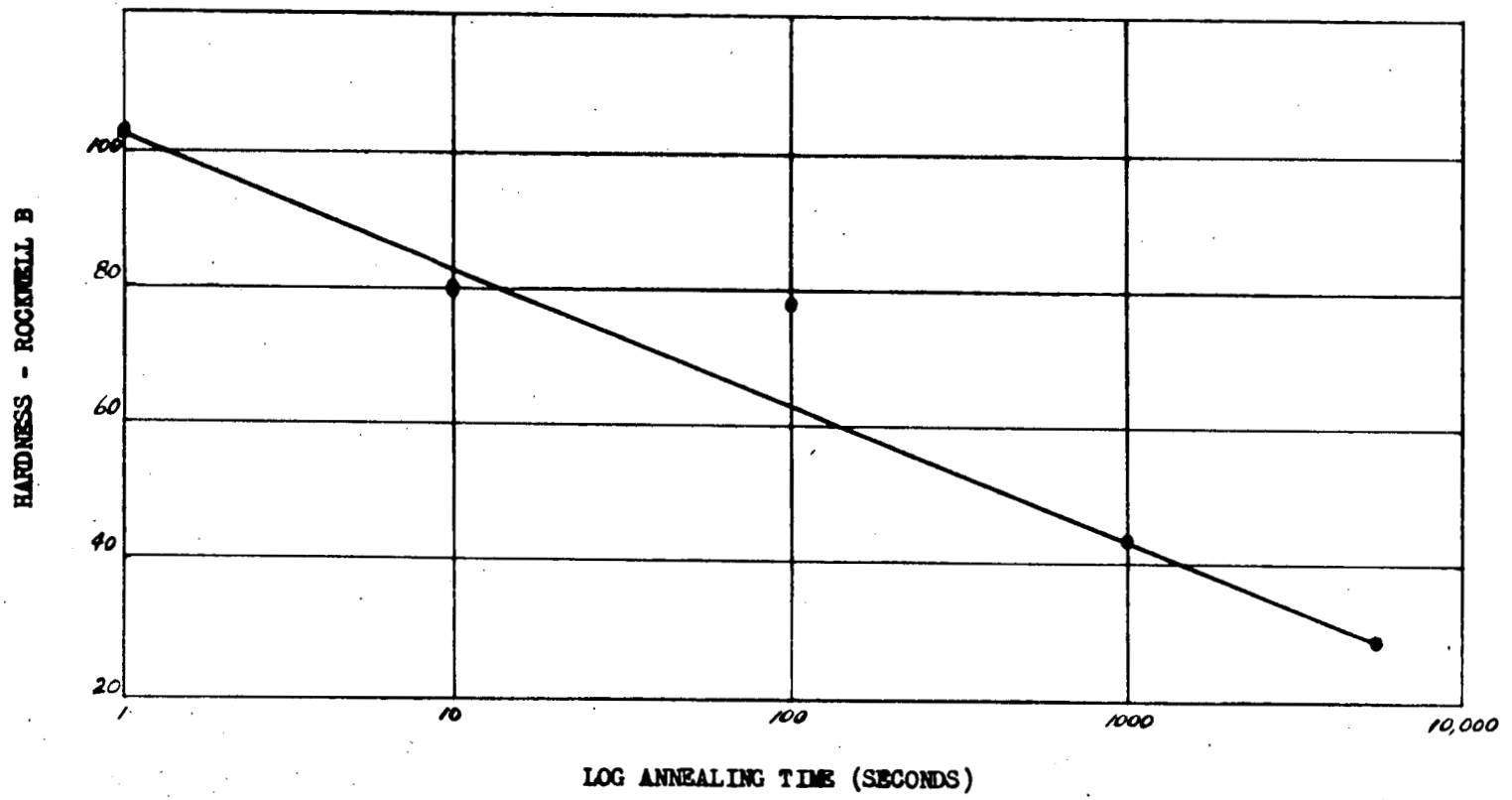


FIG. 34. HIGH PURITY URANIUM SPECIMENS AS-CAST, HOT ROLLED 90%, WARM ROLLED 72%, ANNEALED IN SALT AT 600°C AND QUENCHED INTO WATER AT ROOM TEMPERATURE.

TENSILE TESTS ON ROLLED SHEET

Sample	Prior Treatment	Reduction	Anneal	Average Tensile psi	Average Yield 0.2%	Percent Elong. in 2"	Modulus x 10^6	No. of Samples
(1. Hot Rolled Sheet)								
N	As Cast	78% at 625°	As Rolled	111,300	41,200	27.5	22.3	4
N	As Cast	90% at 625°	As Rolled	128,000	51,500	-	17.5	1
N	As Cast	90% at 625°	550°C - ½ Hr.	113,000	41,600	17.9	25.0	1
N	As Cast	92% at 640°	As Rolled	123,000	47,700	20	17.5	2
N	As Cast	92% at 640°	600° - ½ Hr.	114,500	42,300	17.8	21.7	2
N	As Cast	96% at 610°	As Rolled	99,200	45,000	19.5	18.7	2
N	As Cast	96% at 610°	600° - ½ Hr.	93,000	-	8.5	-	1
(2. Warm Rolled Sheet)								
N	As Cast	96% at 150°	As Rolled	Results very erratic.		No reliability		3
	Cylindrical Mold		600° - 20 Min.	70,000	38,000	-	24.5	3
N	As Cast	96% at 225°	As Rolled	232,000	125,000	5	22.0	3
	Cylindrical Mold		600° - 20 Min.	120,000	59,000	-	20.5	2
N	As Cast	96% at 325°	As Rolled	215,000	121,000	5.5	21.9	4
	Cylindrical Mold		600° - 20 Min.	115,000	65,000	-	23.2	5

Note: All temperatures are centigrade.

TENSILE TESTS ON ROLLED SHEET (CONT'D)

Sample	Prior Treatment	Reduction	Anneal	Average Tensile psi	Average Yield 0.2%	Percent Elong. in 2"	Modulus x 10^6	No. of Samples
(2. Warm Rolled Sheet (Cont'd)								
N	As Cast Cylindrical Mold	96% at 225° +20% at Room Temperature	As Rolled	226,600	125,000	6	23.3	4
			600° - 20 Min.	104,000	42,000	5	27.2	3
N	As Cast Cylindrical Mold	96% at 325° plus 20% at Room Temperature	As Rolled	209,500	116,500	9	25.9	2
			600° - 20 Min.	104,000	43,500	-	20.6	2
HP	As Cast	97% at 300°	As Rolled	218,000	129,000	8	23.5	2
(3. Hot Rolled Plus Warm Rolled Sheet)								
One N	As Cast	90% at 610° plus 72% at 285°	As Rolled	162,000	102,500	28	21.5	2
and one HP Spec.	As Cast	90% at 610° plus 72% at 285°	As Rolled (45° to Roll. Dir.)	150,000	101,500	30	22.8	2
in Each Pair	As Cast	90% at 610° plus 72% at 285°	As Rolled (90° to Roll. Dir.)	157,000	102,300	33.5	17.0	2

TENSILE TESTS ON ROLLED SHEET (CONT'D)

Sample	Prior Treatment	Reduction	Anneal	Average Tensile psi	Average Yield 0.2%	Percent Elong. in 2"	Modulus x 10^6	No. of Samples
(3. Hot Rolled Plus Warm Rolled Sheet (Cont'd)								
N	As Cast	72% at 625° plus 87% at 325°	As Rolled	206,000	125,000	5.8	22.5	5
			AR then scrubbed with H_2	202,000	126,000	4.3	25.0	5
			AR and polished	201,000	129,000	5.5	30.8	3
N	As Cast	72% at 625° plus 87% at 325°	575° - 20 Min.	128,400	53,000	4.5	24.8	5
			575° - 20 Min. then scrubbed with H_2	125,200	57,700	4	28.2	5
N	As Cast	83% at 600°C plus 93% at 320°	As Rolled	206,000	115,000	-	26.5	2
HP	As Cast	83% at 600°C plus 93% at 320°	As Rolled	202,000	116,000	7	23.6	3
HP	Cylindrical ingot plus forged in 3 planes	76% at 610° plus 91% at 330°	As Rolled	208,000				
N	As Cast	79% at 600° plus 88% at 330°	As Rolled	199,800	121,000	6.5		23
			575° - 20 Min.	115,400	59,000	6.5		30
N	As Cast	75% at 640° plus 90% at 340°	575° - 20 Min.	127,000	65,000	5.5		51

(NOTES: All tensile data were obtained from 0.020 inch thick sheet specimens, machined according to specifications for standard tensile sheet specimens*, and were pulled on a Baldwin-Southwark-Emery tensile testing machine.

All tensile specimens were taken parallel to the rolling direction, except where otherwise noted.

All as cast metal was prepared in plate molds previously described, except where otherwise noted.

All warm rolled sheet specimens listed in the second section above were rolled directly from cylindrical ingots severely forged in three planes.)

From the above data, the following observations can be made.

1. Hot rolled sheet has tensile properties approximately those of warm rolled sheet that has been annealed. This would indicate considerable recrystallization after hot rolling. The decrease in ultimate and yield strengths for sheet having the larger percentages of hot reduction is indicative of increasing grain growth after recrystallization. Quite noticeably are the high elongations of the hot rolled specimens as compared to elongations of the warm rolled specimens.

- -

*ASM Metals Handbook, Cleveland 1948, Page 87, Figure 2.

2. Warm rolled sheet shows an almost linear increase in ultimate and yield strenghts starting around 160,000 psi ultimate for uranium metal warm rolled 70%, and increasing to upwards of 200,000 psi ultimate around 90% warm reduction. Percent of hot reduction prior to warm rolling does not appear to affect final tensile properties.

3. Temperature of warm rolling significantly affects tensile properties.

Rolling in the neighborhood of 325°C gives high tensile and yield properties, and good elongations.

Rolling around 225°C gives highest tensile and yield properties (up to 230,000 psi ultimate as rolled) with slightly lower elongations. Surface and edges of sheet produced at 225°C are excellent (as good as that of sheet rolled at 325°C), but rolling is more difficult.

Rolling below 225°C (and on down to room temperature) is not practical for production of any considerable quantity of uranium sheet. Rolling is difficult and arduous, and reductions must be limited to 1 or 2 percent per pass if severe surface and edge cracking, warping and buckling of sheet are to be avoided. Sheet produced at these low temperatures is unevenly stressed and non-uniform as is shown by very erratic and inconsistent tensile data, and further fabrication would be difficult and impractical.

Compared with the above, sheet rolled at 225°C and above is consistently uniform, with no noticeable directional properties after annealing. Even the as-rolled sheet has low directional properties. Comparison of tensile data taken parallel, at 45°, and at 90° to the rolling direction on both normal and high purity as-rolled uranium sheet reduced 72% at 285°C, shows fairly consistent results in all 3 directions. As larger percentages of warm reduction were made, the difference in strength taken parallel to the rolling direction became greater.

4. Variation in carbon content of the metal used for these experiments, 0.01% average for high purity uranium and 0.06% average for normal uranium makes no appreciable difference in tensile properties As can be seen from the micrographs in this report, the similarity of tensile results is probably due to the fact that the carbides are discontinuous, and distributed evenly and in random fashion through the uranium matrix. The large sized carbides associated with normal uranium apparently have little or no effect on tensile properties, although investigation has indicated that cupping performance and probably drawability are adversely affected*.

5. Mold shape does not appear to affect final tensile properties. Comparison of data in sections 2 and 3 above on sheet rolled from both cylindrical and plate ingots shows no significant difference in results.

*Unpublished research on deep drawing program, Los Alamos.

6. Forging does not increase tensile properties appreciably, though a slight increase can be noted.

7. Hydrogen embrittlement results in no significant decrease in tensile properties. Five specimens reported in section 3 were electrolytically scrubbed with H_2 gas for 30 minutes and pulled within 10 minutes after extraction from the embrittling bath. Average ultimate strength for all specimens was 202,000 psi, as compared with 206,000 psi on 5 specimens pulled as standards. Yield strengths were the same, although the elongation of the H_2 embrittled specimens averaged 4.3% as compared to 5.8% for the standard specimens.

8. Anodic polishing of tensile specimens also showed no appreciable change in tensile properties compared with standard specimens on which the thin, tightly adhering oxide coat was allowed to remain. Ultimate strength was lower, 201,000 psi compared to 206,000 for the standards. But yield strenghts were slightly higher, 129,000 versus 125,000 for the standards, and elongations were essentially the same, 5.5% versus 5.8% for the standards.

9. The high elongations obtained on the hot rolled sheet, and on the first three series of hot and warm rolled sheet reported in section 3 are in surprising contrast to the usual elongations of 4 to 8 percent reported for both as-rolled and annealed sheet. This behavior is not fully understood at present, and additional data is being gathered in an effort to explain this phenomenon.

APPENDIX A

CARBIDES IN URANIUM

C. Gordon Hoffman

[redacted]

APPENDIX A

Carbides in Uranium

The carbide size standard (Figure 1) used in this and subsequent work on uranium is patterned after a system developed for steel.

The original system was based on the equation:

$$A = \frac{1}{2^N}$$ at a magnification of 1000.

where A = the area in square centimeters

and N = the carbide size number.

Assuming the carbides to be spheres, there was calculated the probability of cutting the spheres at various distances from the center by the polished plane, which results in circles of smaller areas than represent the true size of the spherical carbide. The proper proportion of the various sizes of carbides was incorporated in the resulting chart, Figure 1.

However, since the carbides in uranium are much larger than those in steel, the chart has to be used at 100X. Everything remains the same except that the actual area of a section through a size 0 uranium carbide is 0.01 mm^2, as against 0.001 mm^2 for a size 0 iron carbide.

It will be noted from the equation that the cross sectional area of each carbide size is one-half that of the preceding size.

The chief impurity in uranium, from a metallographic point of view, is UC which has a cubic lattice, and which is visible under the

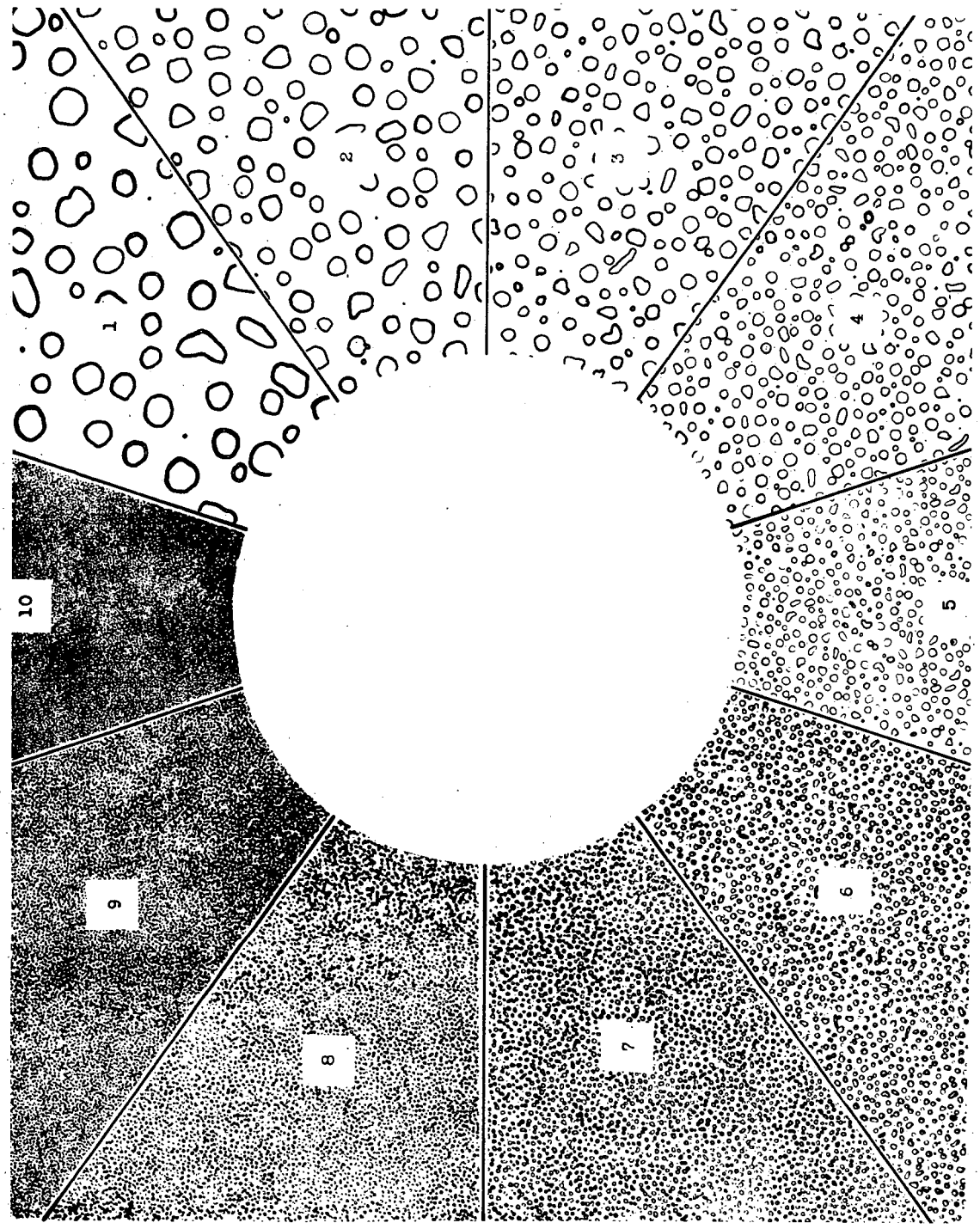


FIG. 1 CARBIDE SIZE STANDARDS

microscope primarily as rectangular particles of somewhat lower reflectivity than the matrix of uranium. Carbon is soluble to a negligible extent in alpha uranium at room temperature, precipitating out as UC when present in amounts of 0.006 percent or less. As mentioned in the foregoing report, carbon is present in normal uranium in amounts of from 0.06 percent to 0.08 percent and in "high purity" uranium to a lesser extent, at times as low as 0.003 percent, but usually about 0.015-0.02 percent.

At 100 diameters 0.06 percent carbon in uranium, as UC, appears in carbide size and number not unlike a spheroidized SAE1060 steel at about 1000X, though of course the shape is usually rectangular (Figures 2A and 2B).

Uranium carbide (UC) has a hardness of approximately 850 D.P.H., hence it is not at all surprising that it should also be extremely brittle, and such is the case. The following photomicrographs show UC as found in cast uranium (Figures 2A and 2B); hot forged at about 600°C (Figures 3A and 3B); hot rolled at about 600°C (Figures 4A and 4B); and in "warm rolled" uranium rolled at about 300°C (Figures 5A and 5B).

These photomicrographs show that the uranium has ample plasticity at about 600°C to flow around the UC particles without exerting much stress upon them, though some fracturing is found in the "hot rolled" material. However, during hot rolling the temperature drops much below the 600°C and may approach "warm rolling" in character.

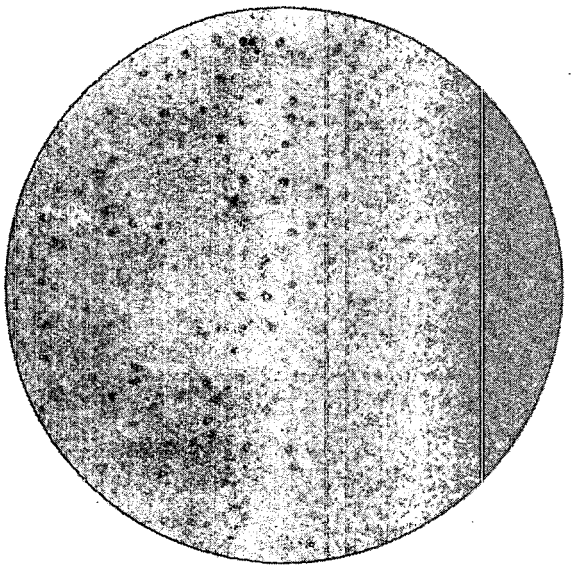


FIG. 2A - Plate 169-4-2 Magnification: 100X
Electrolytic polish: CH_3COOH & CrO_3. Not etched.
"Normal" uranium as-cast carbides are size #5.

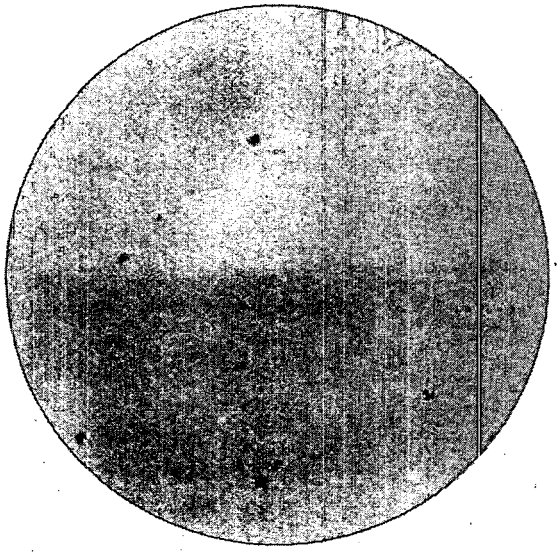


FIG. 2B - Plate 169-2-1 Magnification: 100X
Electrolytic polish: CH_3COOH & CrO_3. Not etched.
"High purity" uranium as-cast carbide size #5.

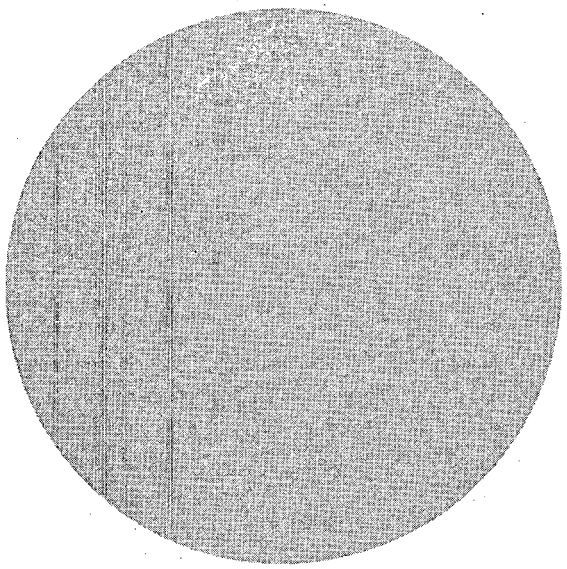


FIG. 3A - Plate 78A-3 Magnification: 100X
Electrolytic polish: CH_3COOH & CrO_3. Not etched.
"Normal" uranium hot forged (600°C) 75%. Carbides (size 5) not broken up or strung out.

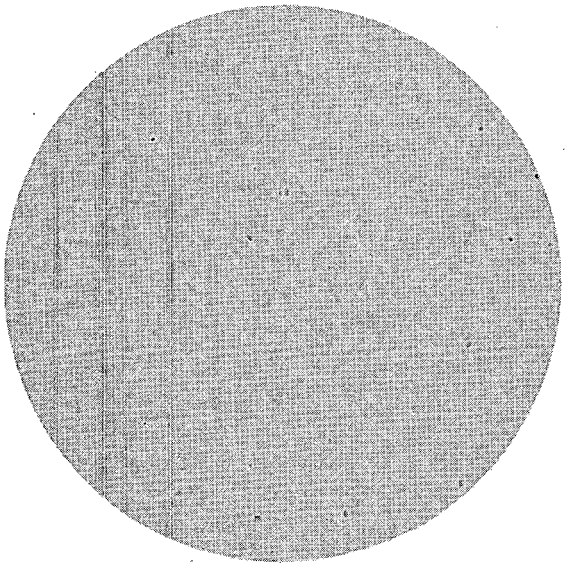


FIG. 3B - Plate 126-3-3 Magnification: 100X
Electrolytic polish: CH_3COOH & CrO_3. Not etched.
"High purity" uranium hot forged (600°C) 75%. Carbides (size 6-7) not yet broken up or strung out.

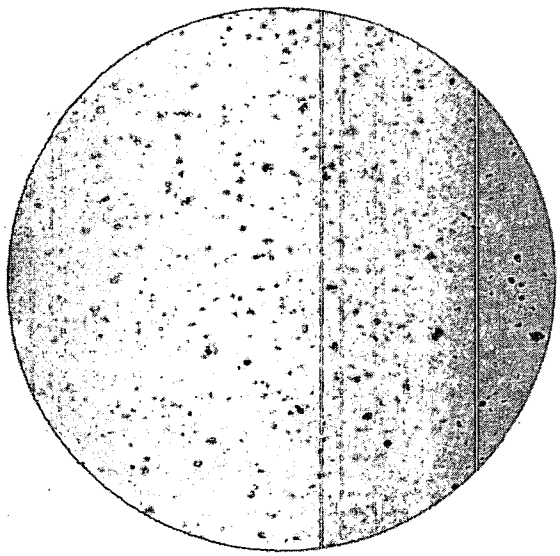


FIG. 4A - Plate 113-3-2 Magnification: 100X
Electrolytic Polish: CH_3COOH & CrO_3. Not etched.
"Normal" uranium hot rolled (600°C) 90%. Carbides (size 5) not broken up much or strung out.

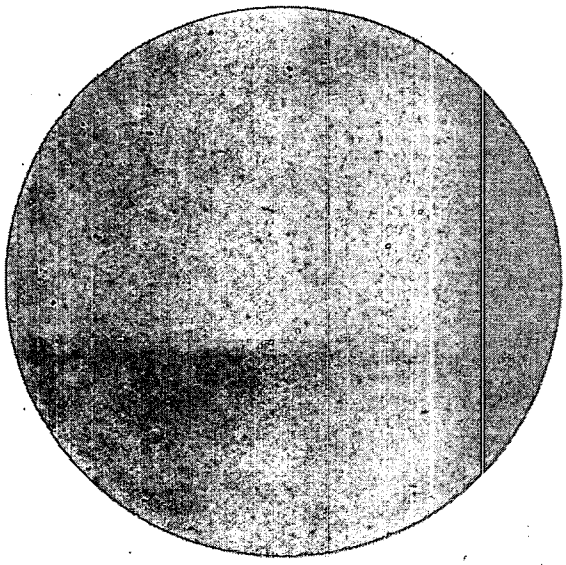


FIG. 4B - Plate 113-6-2 Magnification: 100X
Electrolytic Polish: CH_3COOH & CrO_3. Not etched.
"High purity" uranium hot rolled (600°C) 90%. Carbides (size 7) not broken up or strung out much.

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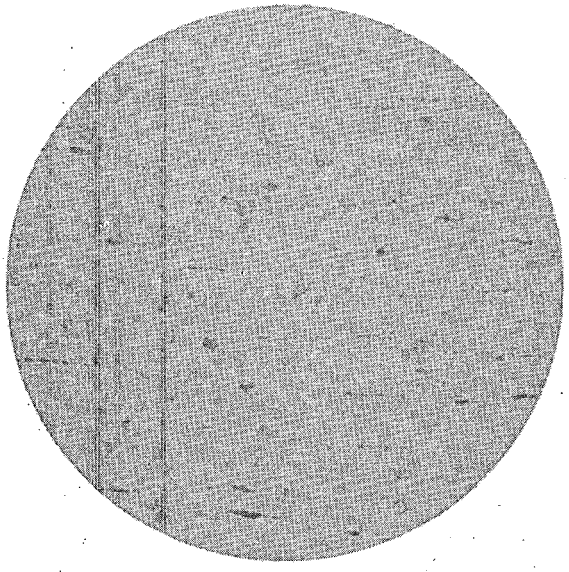

FIG. 5A - Plate 135-1-1 Magnification: 100X
Electrolytic polish: CH_3COOH & CrO_3. Not etched.
"Normal" uranium warm rolled (300°C) 93%. Carbides (size 4) broken up and strung out in matrix.

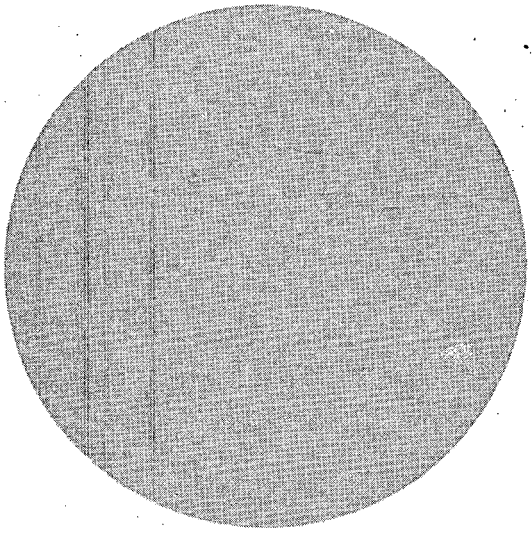

FIG. 5B - Plate 135-8-1 Magnification: 100X
Electrolytic polish: CH_3COOH & CrO_3. Not etched.
"High purity" uranium warm rolled (300°C) 93%.
Carbides (size 7-8) broken up and strung out.

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At temperatures in the neighborhood of 300°C, uranium in being worked exerts sufficient stress upon the brittle carbides to fracture them and upon further elongation by rolling carries the fragments with it, resulting in long stringers, as shown in Figures 5A and 5B.

This process is pictured in Figures 6A, 6B and 6C. These pictures show the starting of fracture (6A), the general breaking up (6B), and carrying along of the fragments into long strings (6C).

These strings consist of long voids which contain fragments of carbide at intervals (6C). The void is partially closed up after passage of the carbide fragment, but remains as a discontinuity and therefore a point of weakness. These stringers of carbides can and do cause definite weakness in the uranium and detract greatly from the ductility and drawability of the metal. This has been discussed in prior reports.

The role of carbides and carbide fragments as nucleation points for recrystallization has also been discussed in previous reports. The accompanying photomicrographs indicate, and it seems probable, that there are fewer carbides in the "high purity" uranium than in "normal" uranium, though at times there seem to be about the same number of carbides but somewhat smaller.

The fact that "normal" uranium usually begins to recrystallize at a lower temperature than does the "high purity" uranium is readily explained by nucleation for recrystallization by the carbides and by points of high strain created by fracturing and stringing out of the

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carbide fragments.

Much work remains to be done on carbides in uranium and their effect on working the metal, both as to the metallographic study as compared with fabrication studies, and in preparation of very low carbon uranium. The solubility of carbon in alpha uranium at room temperatures will be pursued to lower carbon contents as such metal becomes available.

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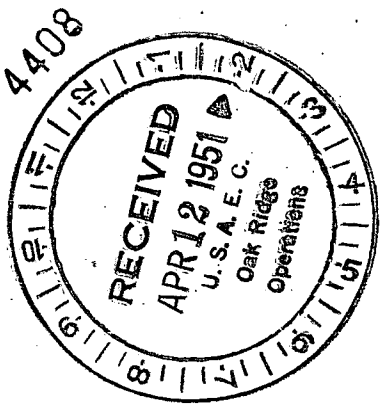

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