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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

FABRICATION OF TANTALUM CAPSULES

FOR LAMPRE I REACTOR

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FABRICATION OF TANTALUM CAPSULES FOR LAMPRE I REACTOR

by

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ABSTRACT

The Los Alamos Scientific Laboratory has been concerned with the fabrication of tantalum primarily for use as a container for molten plutonium.

Two methods were developed for the forming of tantalum containers.

The first method was by deep drawing. This involved the rolling of a tantalum billet into sheet of the desired gage, cutting a circular blank, and deep drawing the tantalum blank through successive stages into the desired shape. The rolling schedule necessary to produce high quality sheet, with random orientation, suitable for deep drawing is discussed in detail. The correct type of lubrication, tool design, tool material, tantalum purity, and heat treatment are also discussed in detail.

The second method used for the fabrication of tantalum containers consisted of a combination of impact extrusion and ironing. This method involved the extrusion of the cast billet into rod, swaging the rod to a suitable diameter, and cutting it into slugs of the desired length. The slugs were then impact extruded into heavy walled containers. These starting containers were then taken through successive dies where the wall thickness was ironed down to the desired gage.

Extrusion, swaging, impact extrusion, and ironing techniques are discussed. The effect of lubrication, tool material, tool design, tantalum purity, and heat treatment, all play an important part in producing good tantalum containers by this method of fabrication.

Other methods of fabrication such as flow turning and spinning are briefly mentioned.

INTRODUCTION

Tantalum is one of the highly refractory metals becoming more and more important in reactor design. It is one of the few metals which can contain molten plutonium. This fact is very important in the design of the new family of molten plutonium reactors such as the LAMPRE I at Los Alamos.

Its increased use as a plutonium fuel container for fast reactors has necessitated an increased interest in the fabrication of containers and closed end tubing or thimbles for this type of application.

Although tantalum is quite widely used in other fields, this paper is concerned chiefly with the fabrication of tantalum for use as capsules, thimbles, containers, or crucibles for containing molten plutonium. It is more specifically concerned with those plutonium containers or thimbles for the LAMPRE I reactor.

The final size of this type of container was a closed end tube approximately 9 inches in length with an I.D. of approximately 0.375 inch and a wall thickness of 20 to 30 mils.

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CHAPTER 1

MATERIAL

All of the fabrication work on tantalum was done using two general types of starting material. This material was high purity tantalum either arc cast or electron beam melted and electron beam melted material containing approximately 1000 ppm of tungsten.

The high purity electron beam melted tantalum had a nominal composition as follows:

C	-	<25 ppm
H_2	-	<10
O_2	-	5-30 (Av 20)
N_2	-	15
Nb	-	<200
Mo	-	<30
W	-	<100

The high purity electron beam melted material containing tungsten had a nominal composition as follows:

C	-	<25 ppm
H_2	-	<20
O_2	-	10-30 (Av 20)
N_2	-	50-70 (Av 60)
Nb	-	<200
Mo	-	<50
W	-	200-1100 (Av 900)

All of these data presented in this paper are concerned with only the high purity material. The tantalum-tungsten alloy employed the same fabrication procedure as the unalloyed material with the exception of heat treatment. The addition of the tungsten helped to refine the grain structure and to raise the recrystallization temperature.

All of the starting material was received as an ingot either 1 1/2 or 3 3/4 inch diameter. The lengths varied from 12 inch maximum for the small diameter ingots up to 30 inch for the large diameter ingots.

CHAPTER 2

METHODS OF FABRICATION

The two chief methods of fabricating tantalum into thin walled containers were deep drawing and impact extrusion and ironing. These two methods will be discussed in considerable detail. Other methods which can be used for limited production are flow turning, spinning, casting and machining, explosive forming, marforming or rubber forming, and extrusion. None of these methods proved practical for large scale production of small diameter thin walled containers of the type necessary for plutonium containers.

2.1 Deep Drawing

The fabrication of a container by this method involves the rolling of sheet of a suitable gage for making the final container. After the sheet has been rolled it is annealed and blanked into a circular disc of the correct size. This blank is then cupped into a starting shape after which it is drawn through a succession of dies whereby the diameter is reduced progressively and the cup gradually lengthened.

Figure 1 shows the fabrication sequence from blank to a finished container.

Figure 2 shows the physical setup used in mounting the tools for forming the components shown in Figure 1.

The first stage or cupping stage is shown in the top sequence of views. This is the only operation where hold down pressure is used. A 4 inch diameter blank of tantalum sheet is placed over the die holder and formed into the starting cup. In the tooling setup shown in Figure 2, the dies are mounted on the top movable platen of the press while the punches are mounted on the stationery bottom platen of the press.

The lower sequence of photographs show the last redraw stage.

2.1.1 Rolling

Development work on the rolling of tantalum to produce sheet of deep drawing quality was done first on arc cast material. The billet was approximately 3 inches in diameter x 4 inches in length. The as-cast structure of the billet was broken up by upsetting on a 350 ton hydraulic press. The upsetting force was parallel to the longitudinal axis of the ingot. Upsetting in this direction seemed to produce the best final structure. A 50 to 60% reduction in height by upsetting followed by 75 to 85% reduction in thickness by rolling with subsequent intermediate anneals before final rolling and final annealing was necessary to completely break up the as-cast structure to gain complete

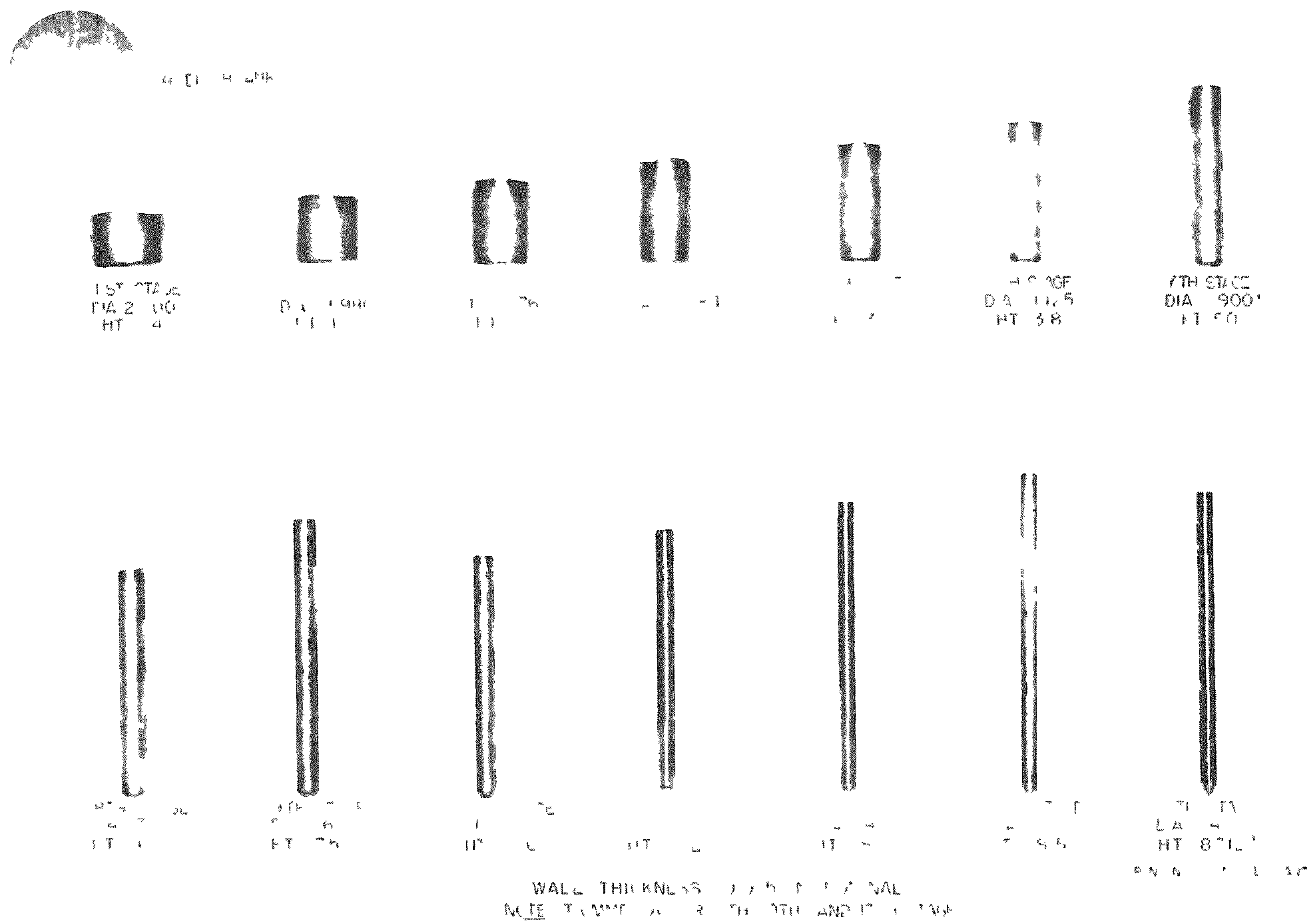


Fig. 1. Deep drawing stages for LAMPRE I -- pin type.

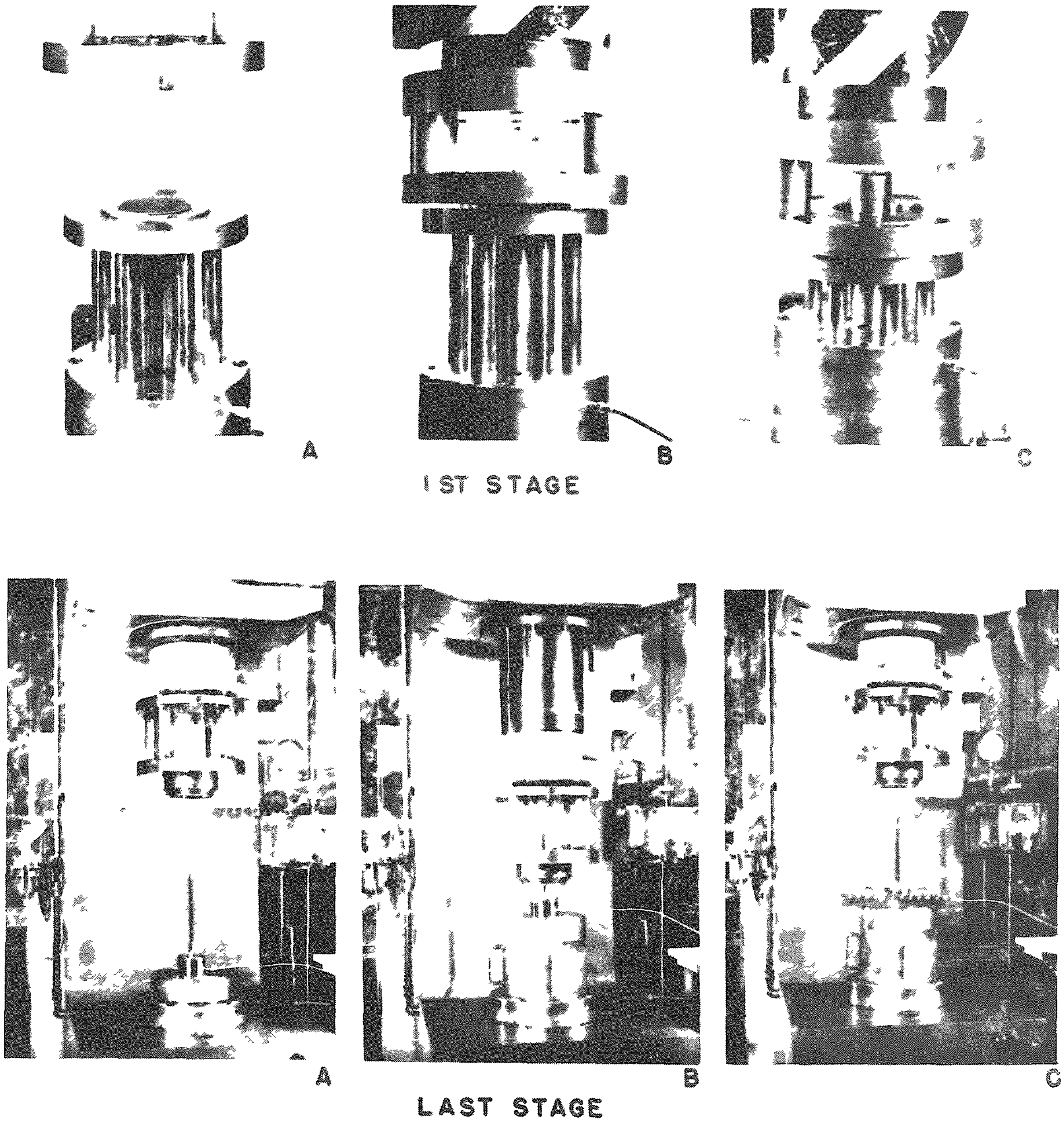


Fig. 2. Deep drawing of tantalum.

recrystallization on final annealing. Without an intermediate annealing treatment, a total reduction of 90% during rolling was not sufficient to give total recrystallization upon final annealing.

The rolling schedule of the upset billet consisted of rolling in two directions to develop a slab wide enough to give a 4 1/2 inch wide final sheet from which 4 inch diameter blanks could be obtained. This two direction rolling (each pass 90° to the previous pass) was carried out for an average of 80% to a thickness of 1/8 inch. At this thickness an intermediate anneal was given the sheet in a vacuum on the order of 0.1 micron for a period of 30 minutes at 1200°C.

The last few passes prior to the above intermediate anneal were usually in the same direction. After this first anneal the grain size of the rolled sheet was between 0.020 and 0.060 mm.

After annealing, the tantalum sheet was rolled to a final gage of 30 mils. Three different rolling schedules were investigated.

Schedule 1. Sheet rolled to final thickness all in one direction with this direction being the same as the last few passes before the intermediate anneal. Approximately 80% reduction in thickness was taken.

Schedule 2. Sheet was rolled exactly the same as in schedule 1 with one exception; namely, the rolling direction was 90° to the direction of the last few passes before the intermediate anneal.

Schedule 3. The sheet was cross rolled with approximately equal reductions taken in each direction. The first reduction was usually taken in the same direction as the last few passes before intermediate annealing.

In all three schedules the finished sheet was vacuum annealed for 30 minutes at 1200°C.

Tantalum blanks, from schedule 1 material, drawn into cups showed pronounced preferred orientation or directionality of grain structure as exhibited by four ears on the first stage deep drawn cup. This schedule also caused the tantalum to fold badly on the side walls during redrawing operations.

Blanks rolled by schedule 2 and drawn into cups still showed four ears but these were not as pronounced as in schedule 1. Some cups showed folding at various stages of redraw while others showed only slight folding.

Blanks made from sheet rolled by schedule 3 were drawn into cups with a complete lack of earing. The sheet made by this rolling schedule appeared to be randomly oriented. There was also no tendency for the material to fold on the side walls during redraw operations.

Sheet produced according to schedule 3 also showed nearly complete recrystallization upon annealing while sheet produced by schedules 1 and 2 exhibited only partial recrystallization.

Rolling schedule 3 was adopted for the fabrication of all sheet to be used for deep drawing.

To produce sheet of superior surface quality it was also found best to machine the surface of the billet after upsetting because of the rough grain structure on the upset slab which had a tendency to cause folds and seams during subsequent rolling operations.

Figure 3 shows some of the effects of material and rolling schedule on the directionality and earing of deep drawn tantalum cups.

Cup No. 1A was drawn from tantalum sheet made by powder metallurgy and not arc cast or electron beam melted. This deep drawn cup showed no directionality.

Cup No. 2A was drawn from sheet made by cutting a 1 inch thick section from a 3 inch diameter arc cast billet. This section was reduced 75% by rolling in four directions with each pass at 45° to the last pass. The resulting plate was then annealed at 1200°C for 30 minutes. Further rolling was continued in four directions again down to final gage. In this instance the total reduction after annealing was 88% and the final thickness was 0.030 inch. A final anneal of 30 minutes in a vacuum at 1200°C was given the sheet. This rolling schedule never broke up the cast structure completely and annealing only recrystallized the heavily worked areas. The drawn cup had five ears on the first stage draw. There was severe folding on subsequent redraws.

Cup No. 3A was deep drawn from sheet made from an arc cast billet. A 3 inch diameter billet section was cut through its center parallel to

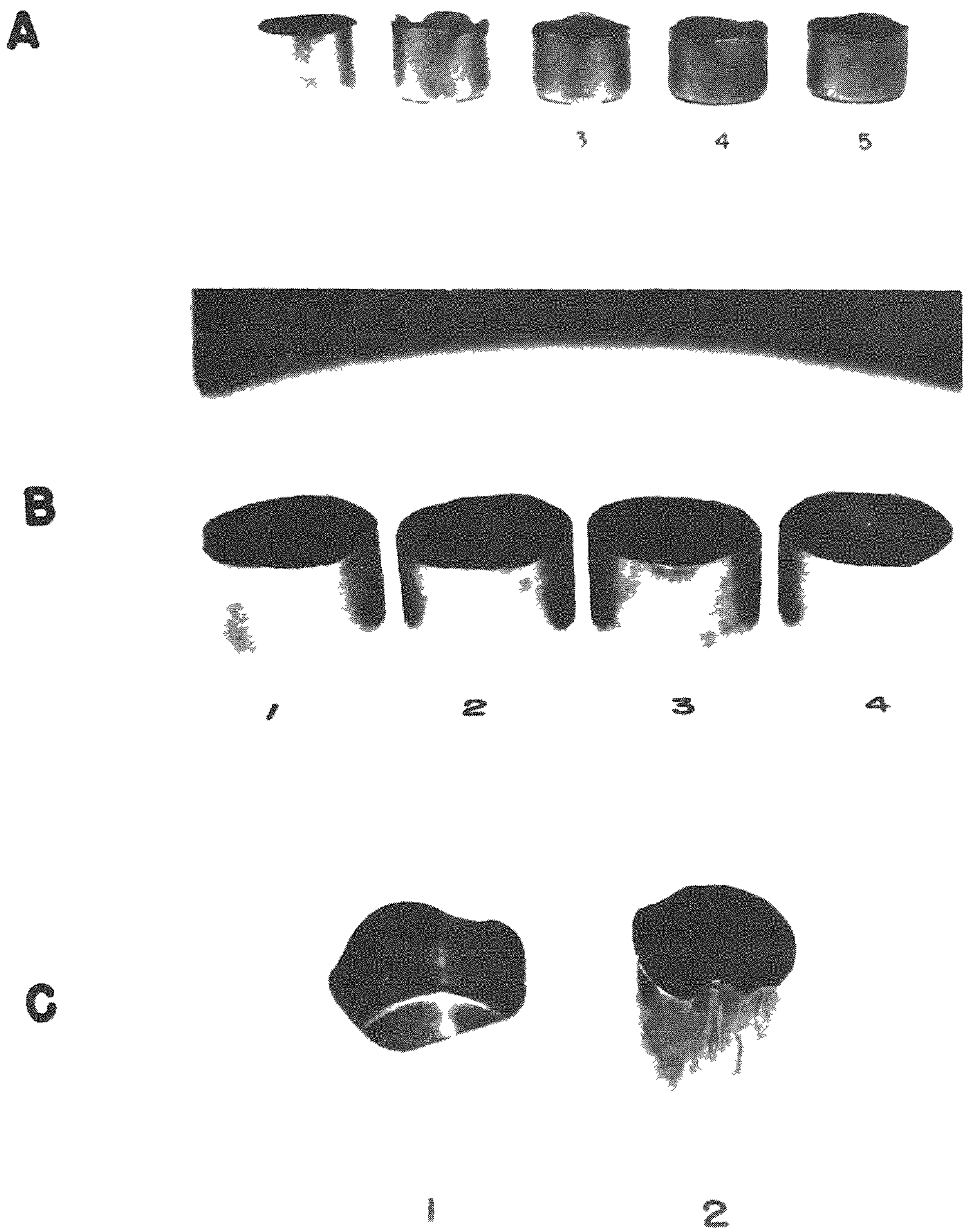


Fig. 3. Effect of rolling and annealing on deep drawing of tantalum.

the longitudinal axis and this half moon section rolled in two directions. Each pass was 90° to the previous pass. A total reduction of 93% was made to a thickness of 0.105 inch. The sheet was given an intermediate anneal in vacuum for 30 minutes at 1250°C. One piece of the 105 mil sheet was rolled to 0.030 inch, approximately 72% reduction, with the rolling at 90° to the last rolling direction before the intermediate anneal. The final sheet was vacuum annealed at 1300°C for 30 minutes which gave complete recrystallization. The cup as drawn shows four pronounced ears.

Cup No. 4A was deep drawn from sheet rolled exactly like cup No. 3 with the exception that the final blank was annealed in vacuum for 30 minutes at 1400°C. The grain size after annealing was an average of 0.050 mm with some grains as large as .100 mm. Earing was the same as in 3 but the cup surface was much rougher, especially on the radius.

Cup No. 5A was drawn from sheet made using the same schedule as Cup No. 3 with the exception that the final anneal was in vacuum for 30 minutes at 1350°C. The grain size was the same as for Cup No. 4. Earing was about the same as for Cup No. 4 but the surface was not quite so rough.

Cups No. 3A, 4A, and 5A, however, did draw through all stages without folding.

Figure 3, view B, illustrates cups deep drawn from sheet rolled according to schedule 3 but each cup was given a final anneal at a different temperature. The tantalum used was high purity electron beam melted material.

Cup No. 1B was annealed in vacuum for 30 minutes at 1300°C. It was fully recrystallized with large grains between .100 and .150 mm in size. The drawn cup exhibited a rough "orange peel" surface.

Cup No. 2B was drawn from sheet annealed at 1150°C for 30 minutes. It was fully recrystallized with a grain size of 0.050 to 0.100 mm. The surface of the drawn cup was somewhat smoother than cup No. 1 but was still quite rough.

Cup No. 3B was drawn from sheet vacuum annealed at 1100°C for 30 minutes. It showed approximately 90% recrystallization with a grain size between 0.050 and 0.080 mm. The surface was still rough but somewhat smoother than cup No. 2.

Cup No. 4B was drawn from sheet vacuum annealed for 30 minutes at 1000°C. Recrystallization was about 85% complete showing a grain size between 0.050 and 0.060 mm. This cup had the smoothest finish.

Cups No. 3B and No. 4B are types which have a structure which will fold during some stage on the redraw.

Figure 3C illustrates preferred orientation in sheet by rolling in one direction and the folding that occurs on this type of material during redrawing.

Figure 4 illustrates graphically the effect of hardness vs reduction before and after annealing. At about 40% reduction, the hardness levels off at approximately 115 DPH.

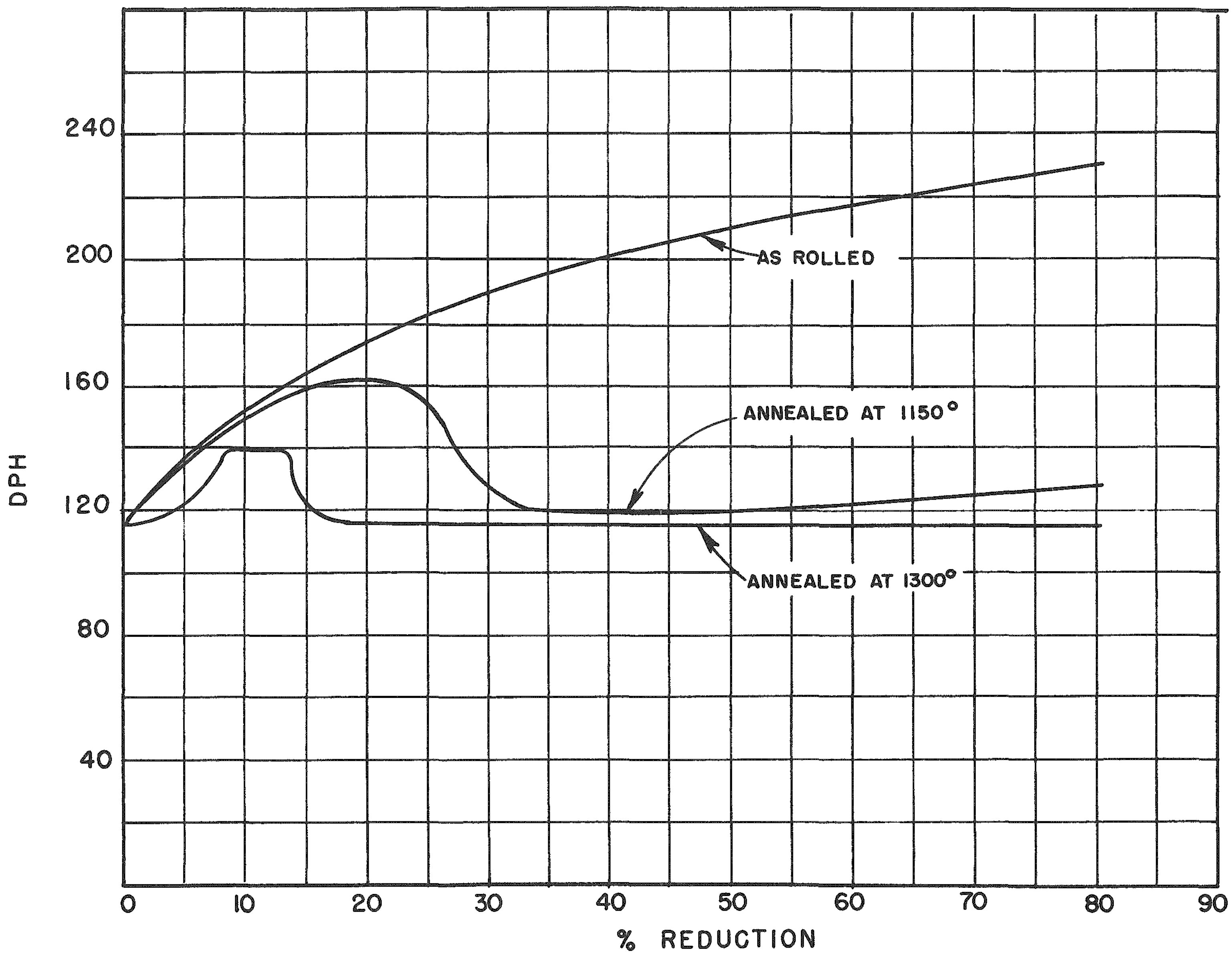


Fig. 4. Hardness vs per cent reduction -- arc cast tantalum.

2.1.2 Tool Design

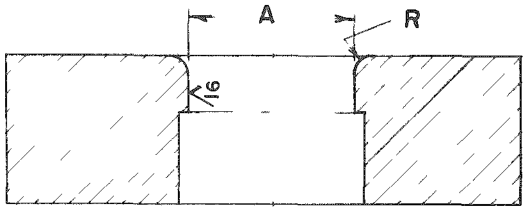
The tool design for the deep drawing of tantalum is typical of that used for mild steel. The same general rules apply. However, the die material is of utmost importance. Dies cannot be made of steel as severe galling will take place. Satisfactory drawing can be done using aluminum-bronze alloy dies and hardened steel punches which have been chrome plated. Even this combination will sometimes cause galling on the inside of the cup or tube. If the tantalum blank was anodized previous to drawing, galling and pickup can almost be eliminated when using steel chrome plated punches and aluminum-bronze dies. The best combination is the use of both punches and dies made from aluminum-bronze.

An overlay of aluminum-bronze metal on mild steel will work satisfactorily if the punch is large enough in diameter to prevent warpage. Aluminum-bronze can also be sprayed on a mild steel surface.

The typical analysis of the aluminum-bronze used by LASL for tool making was as follows:

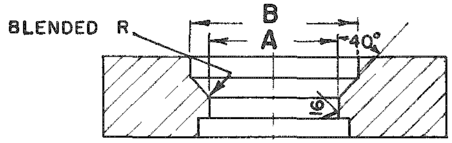
Al	=	14 to 15 w/o
Fe	=	4 to 6 w/o
Others	=	0.5 max w/o
Cu	=	Balance

Figure 5 shows the cross section of the tools used for making the plutonium containers for the LAMPRE I reactor. The fabrication sequence was shown in Figure 1.



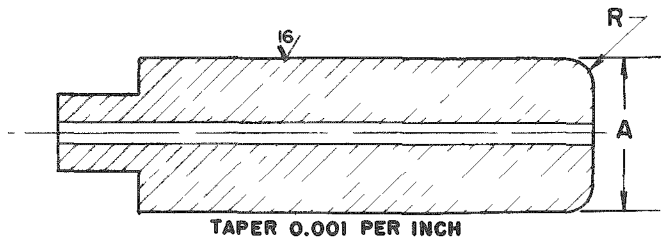
1 ST STAGE DIE

A	R
2.200	4-7 X SHT. T



REDRAWING DIES

STAGE	A	B
2	1.980	2.205
3	1.760	1.985
4	1.585	1.765
5	1.410	1.590
6	1.125	1.415
7	.900	1.130
8	.720	.905
9	.612	.725
10	.550	.617
11	.495	.555
12	.450	.500
13	.428	.455



DRAWING PUNCHES

STAGE	A
1	2.144
2	1.924
3	1.704
4	1.528
5	1.354
6	1.069
7	.844
8	.664
9	.556
10	.494
11	.439
12	.394
13	.372

Fig. 5. LAMPRE I deep drawing tools.

First stage cupping dies have a lead in radius of 4 to 7 times the sheet thickness. This ratio of sheet thickness to die radius is not as critical as that required for drawing such metals as copper but is still a factor in design.

A short draw land should be used. Land lengths from 1/8 to 1/4 inch in length were used to draw the LAMPRE I capsules.

A lead in angle of 40° was found to be optimum for redraw dies. This was especially true on drawing the larger diameters. Steeper lead in angles apparently did not work the side walls enough to obtain the strength necessary to resist folding of the wall.

Again, as in cupping dies, the land length for redraw dies should be as short as possible. No ratio of tube diameter to land length was established, however, a land length of 1/8 inch worked very well.

Reductions in diameter of 5 to 30% per redraw were made successfully. However, 20% was about optimum for most types of tantalum.

Cumulative reductions up to 90% can be made successfully with no intermediate anneal or stress relieving treatment. For most of LASL fabrication, 70 to 75% reductions in diameter was the cut off point for an intermediate anneal.

Tantalum can be drawn satisfactorily during the cupping stage with a clearance of up to 15% of the metal thickness to the point where a 20% reduction in metal thickness by ironing of the sidewalls of the cup will occur. Die clearances of 20% of the metal thickness will cause wrinkles on the cup flange.

Up to 25% ironing or reduction in thickness of sidewalls can be done during redraw stages.

Table I indicates the data for the redraw stages used for making the components shown previously in Figure 1.

TABLE I

DATA ON REDRAWING TANTALUM

Stage	% Reduction in Diameter	Cup Dia.	Cross Sectional Area, in.^{2}	Drawing Pressure, lbs.
2	10	1.980	0.154	5700
3	11	1.760	0.136	4150
4	10	1.584	0.123	4000
5	11	1.410	0.108	3080
6	20	1.125	0.086	2670
7	20	0.900	0.069	2460
8	20	0.720	0.054	2300
9	15	0.612	0.046	1500
10	10	0.550	0.040	1080
11	10	0.495	0.037	920
12	9	0.450	0.033	770
13	5	0.428	0.032	770

2.1.3 Annealing of Tantalum

Tantalum must be annealed at a fairly high temperature in an excellent vacuum. Pressure on the order of 1 micron or less is recommended. Above 1 micron pressure, high purity tantalum will absorb oxygen causing the material to become brittle and difficult to fabricate.

Arc cast material, cold worked approximately 90%, completely recrystallized when annealed for 30 minutes at 1300 to 1350°C. The resulting grain size was 0.040 to 0.060 mm.

Electron beam melted material, cold worked 90% and annealed at 1100 to 1150°C for 30 minutes was 90 to 100% recrystallized with a resulting grain size of 0.040 to 0.100 mm.

Upon annealing this same material at 1300 to 1350°C for 30 minutes, the grain size was 0.150 mm. This material showed complete recrystallization.

The initial grain size of the cast ingot has quite an effect on the final grain size of cold worked and annealed tantalum sheet. If the initial grain size of the casting is under approximately 5 mm in size, one can expect an easily obtainable grain size of 40 to 50 microns. However, if the grain size of the starting billet is over 5 mm in size, the final grain size of the annealed sheet will be 50 to 100 microns.

Additions of small amounts of tungsten or molybdenum on the order of 500 parts per million, will refine the as-cast grain size, making it easier to control the grain size during subsequent fabricating operations.

Tantalum which is 90 to 95% recrystallized will deep draw but there is a tendency for the material to fold or form seams on subsequent redraw stages. If the material is 100% recrystallized, folding is not experienced.

It is also possible to draw material with as much as 45% cold work after the last full annealing treatment. However, tantalum with this much cold work also tends to fold and show seams in subsequent redraws.

Tantalum sheet in the "as-rolled" condition can be given a stress relieving treatment, 200 to 250°C below the recrystallization temperature, and this sheet can be drawn equally as well as the fully annealed material. Tantalum closed end tubes have been fabricated with only stress relieving heat treatments which do not promote any recrystallization, giving a tube with a final heavy cold worked structure.

2.1.4 Microstructure

Figure 6 shows the microstructure of a sixth stage tube being made by deep drawing. The figure shows the structure at various locations on the tube in the "as-drawn" condition. In the fabrication sequence, see Figure 1, the tube is given either a stress relief at this stage of fabrication or an intermediate anneal. The stress relief was usually for 30 minutes at 900 to 950°C in a vacuum. The structure is not changed appreciably at this temperature. The full intermediate anneal was usually for 30 minutes at 1150 to 1200°C and this temperature caused complete recrystallization of all areas of the tube.

Figures 7 and 8 show the microstructure of a sixth stage tube annealed or stress relieved for 30 minutes at temperatures from 850

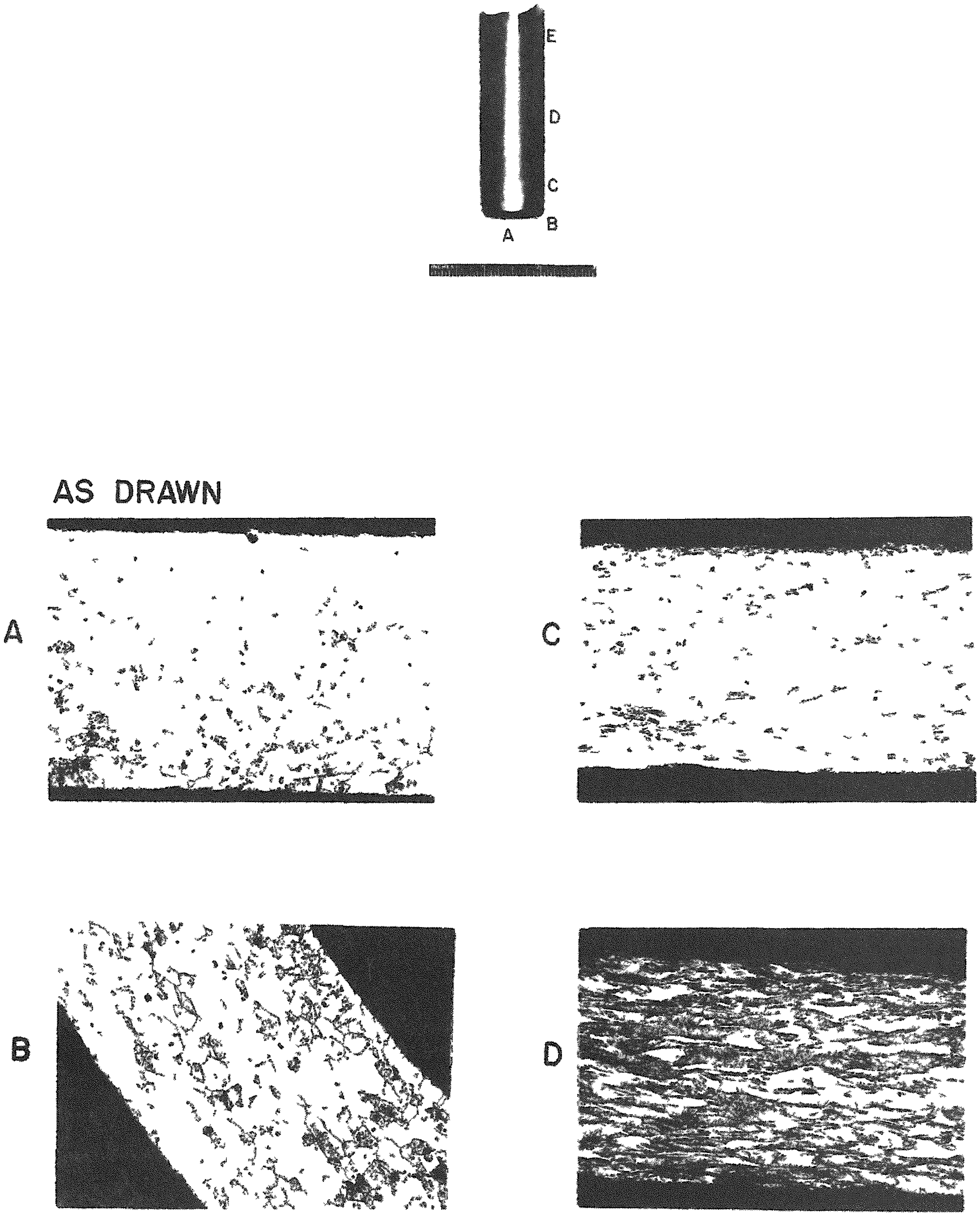


Fig. 6. Structure of a 6th stage tube in the "as-drawn" condition.

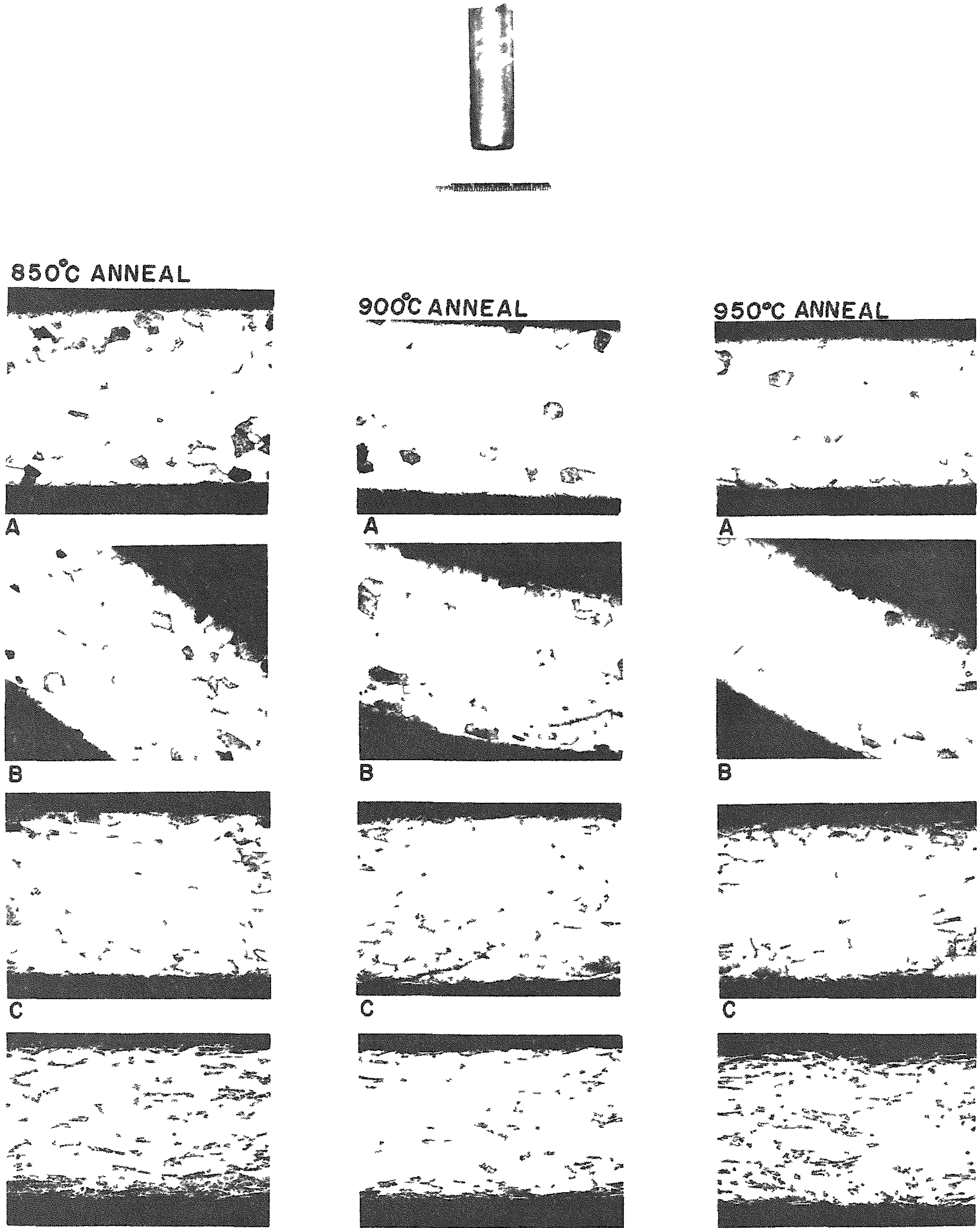


Fig. 7. Microstructure of 6th stage Ta tube as influenced by annealing temperature.

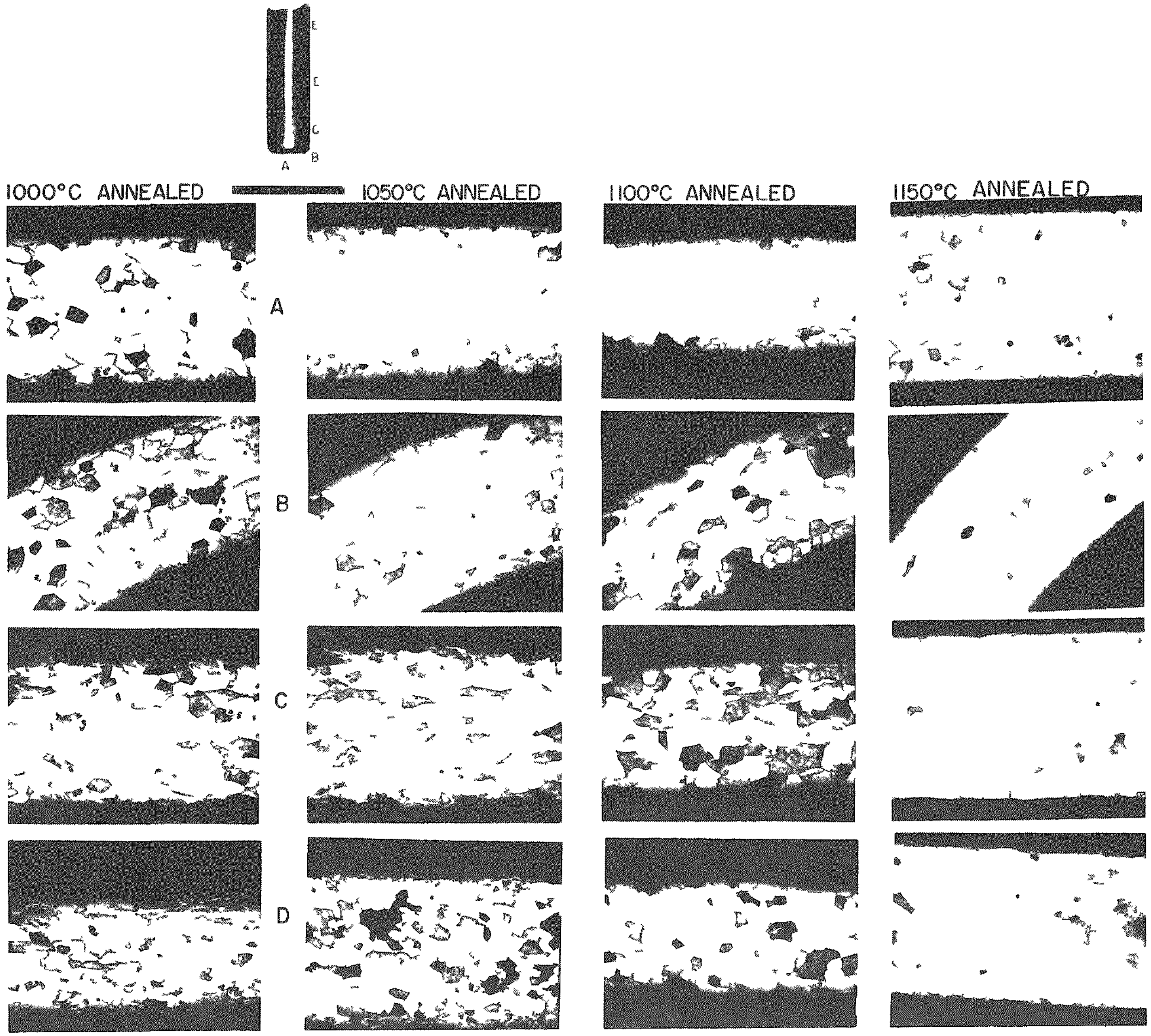


Fig. 8. Continuation of Figure 7 data.

to 1150°C. Complete recrystallization of the tube in all areas starts to occur at the 1050°C temperature.

Figure 9 shows the microstructure at various locations on a finished tube made by deep drawing. The structure of a final tube where the intermediate anneal at the sixth stage was a stress relief at 900°C is shown on the left, and the structure of a final tube where the sixth stage intermediate anneal was a full anneal giving complete recrystallization at 1150°C is shown on the right.

It can be seen that the tube with the intermediate stress relief shows a fairly highly cold worked structure in all areas except the extreme tip. The one with the intermediate full anneal exhibits a partially worked structure throughout.

2.1.5 Cup Test Results

Figure 10 shows graphically cup test results of tantalum compared to three common metals: copper, aluminum, and mild steel. In this type of test, a 1/2 inch diameter steel ball is forced against a firmly held sheet of the material to be tested. The pressure at which the sheet ruptures is recorded as is the cup height formed by pushing the ball against the sheet. The sheet is usually tested at room temperature and several other temperatures to find the point where the maximum cup height and maximum bursting pressure occurs.

An examination of the curves shown in figure 10 indicates that the best deep drawing properties of tantalum occur at room temperature.

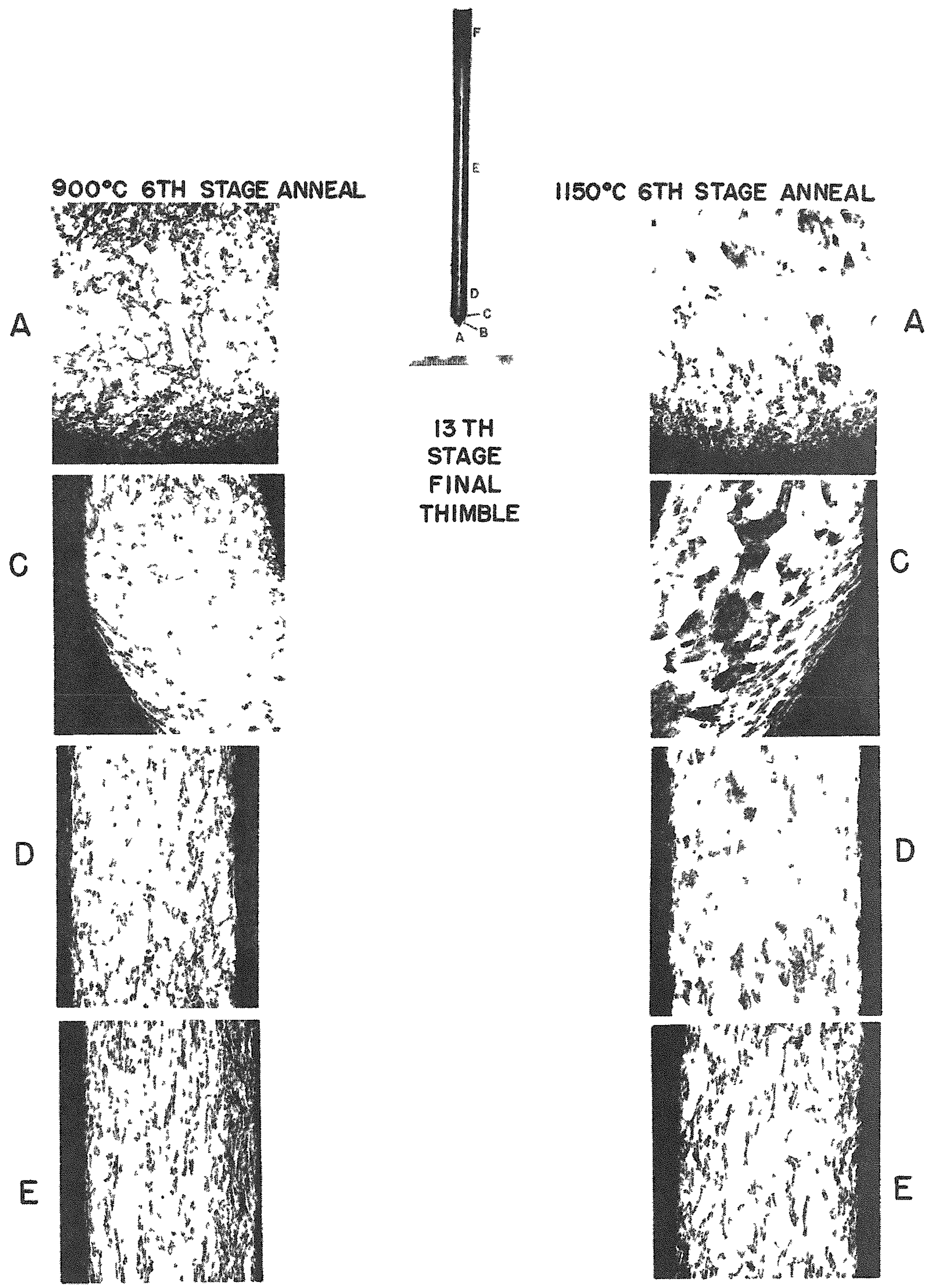
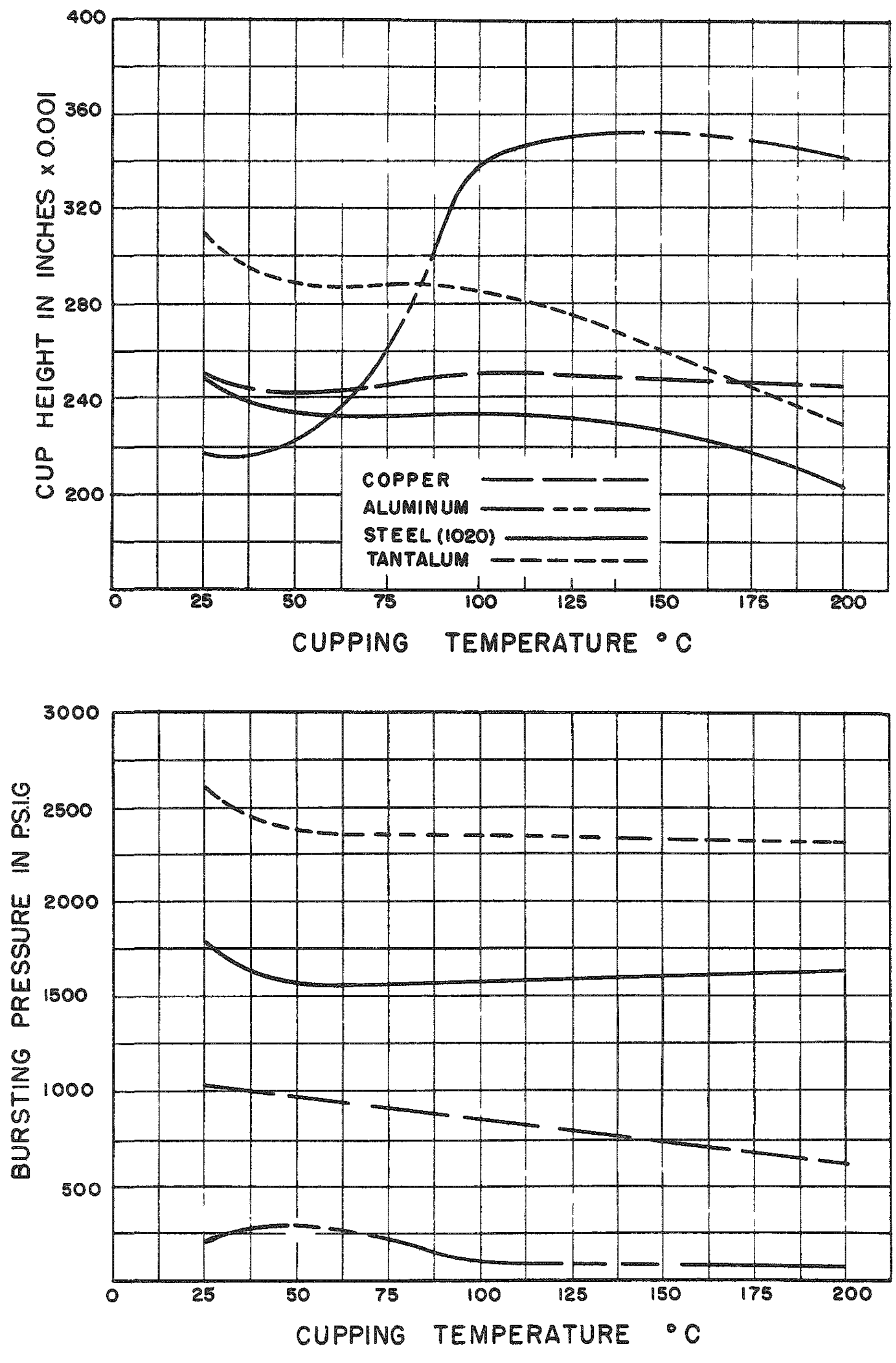


Fig. 9. Microstructure of finished drawn Ta tube (13th stage) as influenced by 900°C and 1150°C heat treatments.



OLSEN CUP TESTS

Fig. 10. Comparison of cup test data for Ta, Cu, Al, and steel.

The curves also indicate that tantalum should deep draw somewhat better than mild steel.

Cupping tests were a very valuable tool in determining if the tantalum sheet was in the proper condition for deep drawing without wasting a large blank on an actual trial run.

2.1.6 Critical Strain

Critical deformation or critical strain in a metal may be defined as a range of permanent deformation which results in a large grain structure when the deformed metal is subsequently annealed. This is a phenomenon which is well known in the nonferrous industry. It is also a mechanism which has frequently been employed to grow large single crystals of a metal.

Tantalum appears to go through a critical strain rate between 11 and 20% reduction. If fully annealed tantalum sheet is given cold reductions of various percentages and then reheated for 30 minutes at 1300 to 1350°C in vacuum, the sheet will exhibit very large grains on the order of 100 microns between the above percentages. As the percentage of reduction increases, the grain size on reheating gradually becomes smaller until we are able to get fine equiaxed grains of approximately 20 microns at approximately 80% reduction.

Figure 11 shows graphically how these maximum grains are related to per cent reduction. As can be seen from the curve the largest grains occur at approximately 14% reduction. However, only about 65% of the

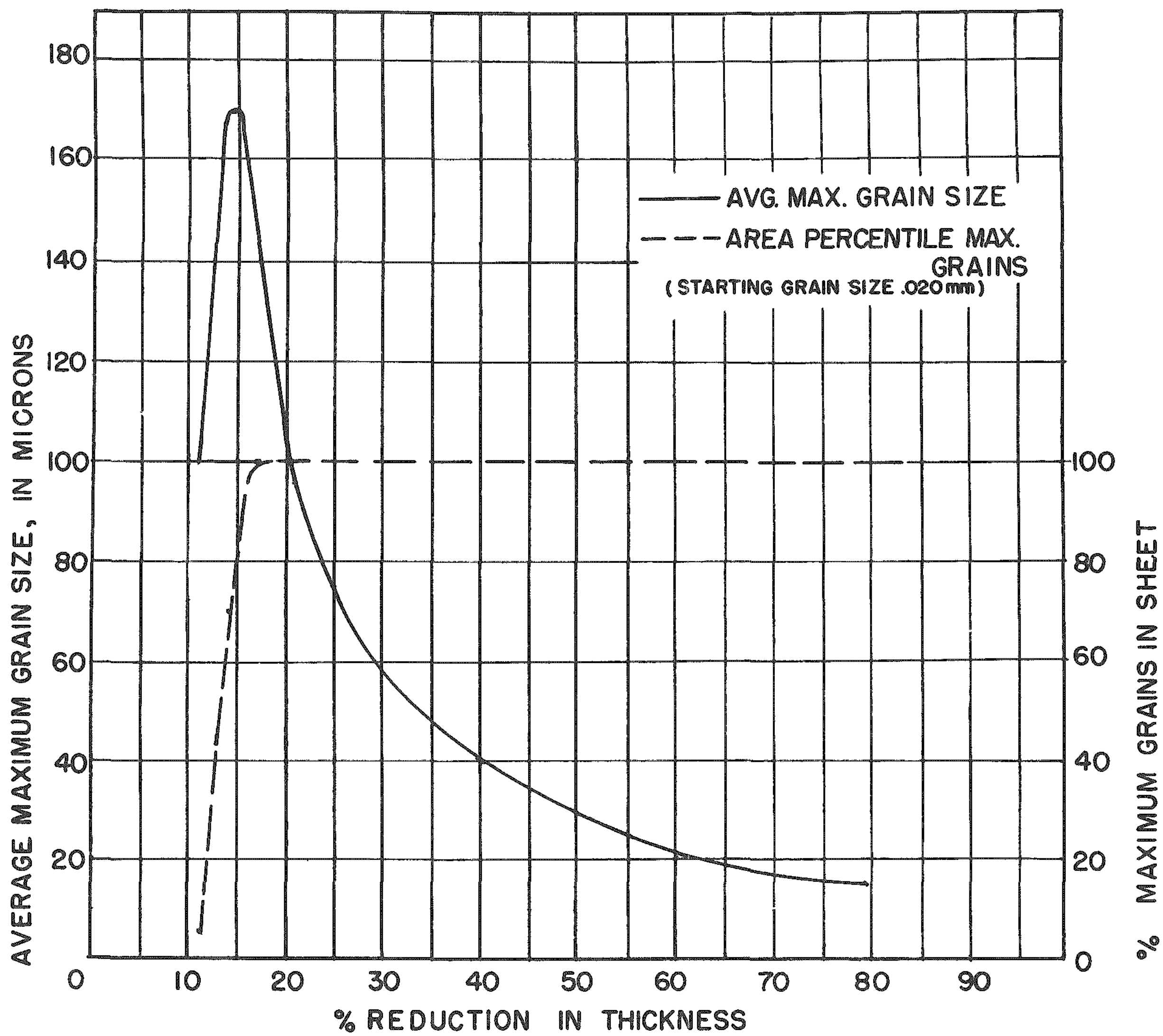


Fig. 11. Grain size vs per cent reduction of annealed tantalum sheet cold rolled the indicated percentages and reheated 30 minutes at 1300°C.

sheet area was composed of these large grains. At approximately 17% reduction we have 100% recrystallization with large grains of approximately 140 microns.

The microstructures of the areas depicted in Figure 11 are indicated in Figure 12.

This phenomenon of critical strain becomes quite important when making containers by deep drawing. It is quite evident that a differential in cold work exists on a deep drawn container. If this tube or container is given an intermediate anneal subsequent to further redraws, then the area having an amount of work equivalent to the critical strain of reduction will exhibit large grains. These large grains (over 100 microns) will cause an "orange peel" effect on the next drawing operation which will make the surface rough.

2.2 Impact Extrusion and Ironing

The fabrication of a container by this method involves the rolling or extrusion of the cast billet into a 1 inch diameter rod. The rod is then swaged to final size where slugs of the correct length are cut. The slugs are then impact extruded into a heavy walled tube having a closed end with a thickness approximately 5 times the tube wall thickness. This impacted tube is then drawn through successive dies which lengthen the tube by ironing the walls and reducing their thickness. The diameter is reduced only slightly with each ironing operation. Figure 13 shows the fabrication sequence for this method of fabrication.

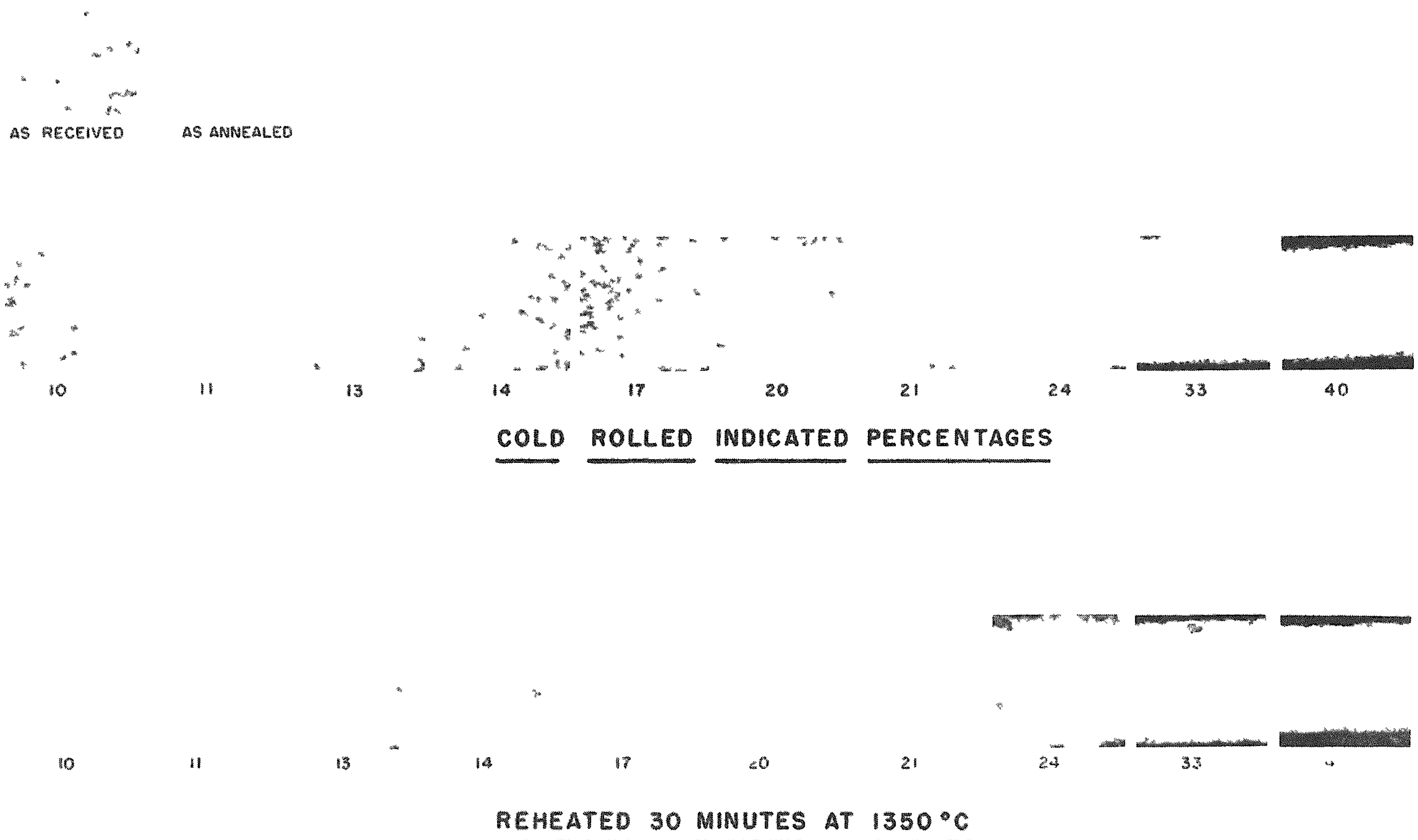


Fig. 12. Microstructure of critical strain area for tantalum.

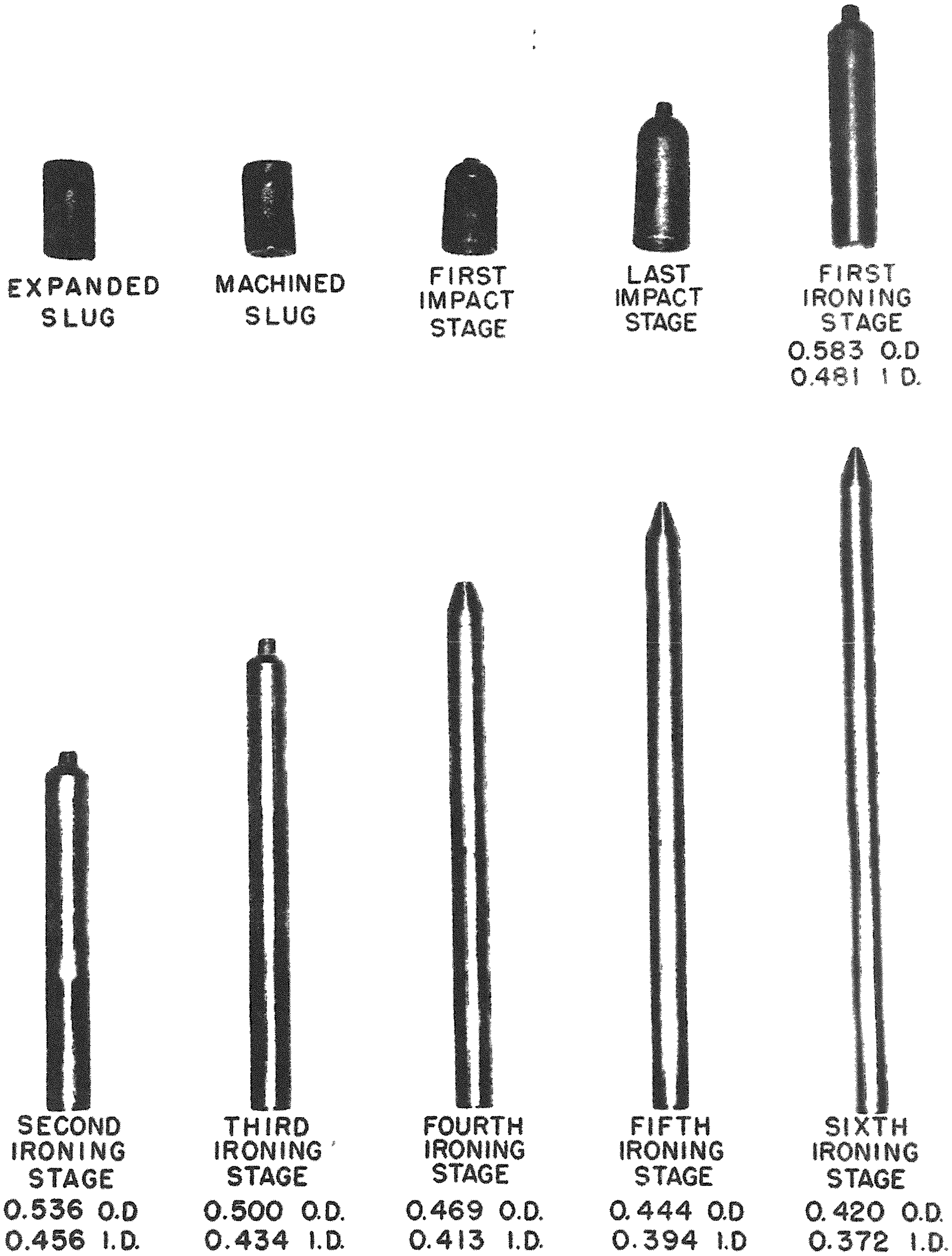


Fig. 13. Tantalum pin-type thimbles -- LAMPRE I.

2.2.1 Extrusion

Dies for cold (room temperature) extrusion of cast tantalum ingots should be shear type dies with a land measuring 1/8 to 3/16 inches. Experimentation has indicated that one satisfactory die material is type 4340 steel hardened to 39 to 41 Rc. Bell mouth dies did not prove feasible due to the large amount of pressure build-up on the exposed surfaces of the die. Excessive corrosion of the die opening was encountered in all cases regardless of the type of steel used. Tungsten carbide inserts were employed successfully in the extrusion die. These inserts required replacement after each extrusion due to fracture of the die face. The surface of the extruded rod was considerably smoother and the diameter more uniform when the carbide inserts were employed. Die costs were also decreased when a replaceable insert was employed. (Aluminum-bronze metal alone was not strong enough.)

The electron beam melted tantalum ingot was machined to a 3 inch diameter and extruded to a 2 inch diameter. This extrusion ratio was limited to 2.5 to 1 due to the size of the press employed -- 500 tons. The extruded material was lightly machined and then annealed at 1200°C in vacuum for one hour and furnace cooled. The annealed tantalum had an average hardness of 70 to 85 DPH. The annealed tantalum was then cold extruded from a 2 inch diameter to a 1 inch diameter. The 1 inch diameter extruded material was treated in the same manner as the 2 inch extrusion prior to swaging to a smaller gage.

Two lubricants were utilized in the extrusion. The billets were precoated with "Bishop's Precoat."* This coating was applied and allowed to dry at least 30 minutes before applying a coating of "Bishop's Jaytolube." This second coat did not dry before the extrusion occurred. The die was similarly lubricated. It was necessary to clean out the billet container after each extrusion to remove the remnants of the lubricant. "Philsolv" was used for this purpose. If the old lubricant was not removed, increased pressures were encountered during the next extrusion.

The extruded tantalum rod reached an average temperature of 275°C as a result of internal friction occurring during extrusion. Some galling and surface pitting was encountered on the extruded rod caused by die break-up during the extrusion operation.

Utilization of a larger capacity press should permit extrusion of a 4 inch diameter rod directly to any size between 1 inch diameter and a 1/2 inch diameter.

Extrusion forces required to produce the 2 inch diameter rod were on the order of 470 tons. The extrusion forces required to produce the 1 inch diameter rod were on the order of 350 tons. The 470 ton force was exerted on a 3 inch diameter rod producing about <u>130,000</u> psi while the 350 ton force was exerted on a 2 inch diameter rod producing about <u>200,000</u> psi.

- -

*Made by Bishop Tube Co., Inc., Malvern, Pa.

The microstructure of the starting billet before and after extrusion is shown in Figure 14.

Figure 15 shows the extrusion press in view A. The starting 3 inch diameter rod is shown in view B and the 2 inch die and extrusion are shown in view D.

The die, 2 inch diameter slug, dummy block, and pusher pin are shown in view C. The final one inch diameter extruded rod is shown in view E.

2.2.2 Rolling Tantalum Rod

As-cast electron beam melted tantalum ingots, 3.75 inch O.D., have been reduced to 1.221 inch O.D. rod by rolling cold. The tantalum rod was rolled dry through cold rolls at room temperature. The as-cast rod was reduced from 3 3/4 inch O.D. to an oval approximately 2 inches by 1 1/2 inch in the blooming mill. Each pass through the bulldog was followed by a pass through a set of edgers. The oval produced by the blooming mill was then run through six stands on the continuous mill. The procedure on the continuous mill was to form an oval in the odd numbered stands followed by a rounding pass in the even numbered stands. The rod temperature as a result of the reduction did not exceed 150°C. The final rod size produced through the sixth stand of the continuous mill was approximately 1 7/32 inch diameter. Further reduction could be made using additional stands.

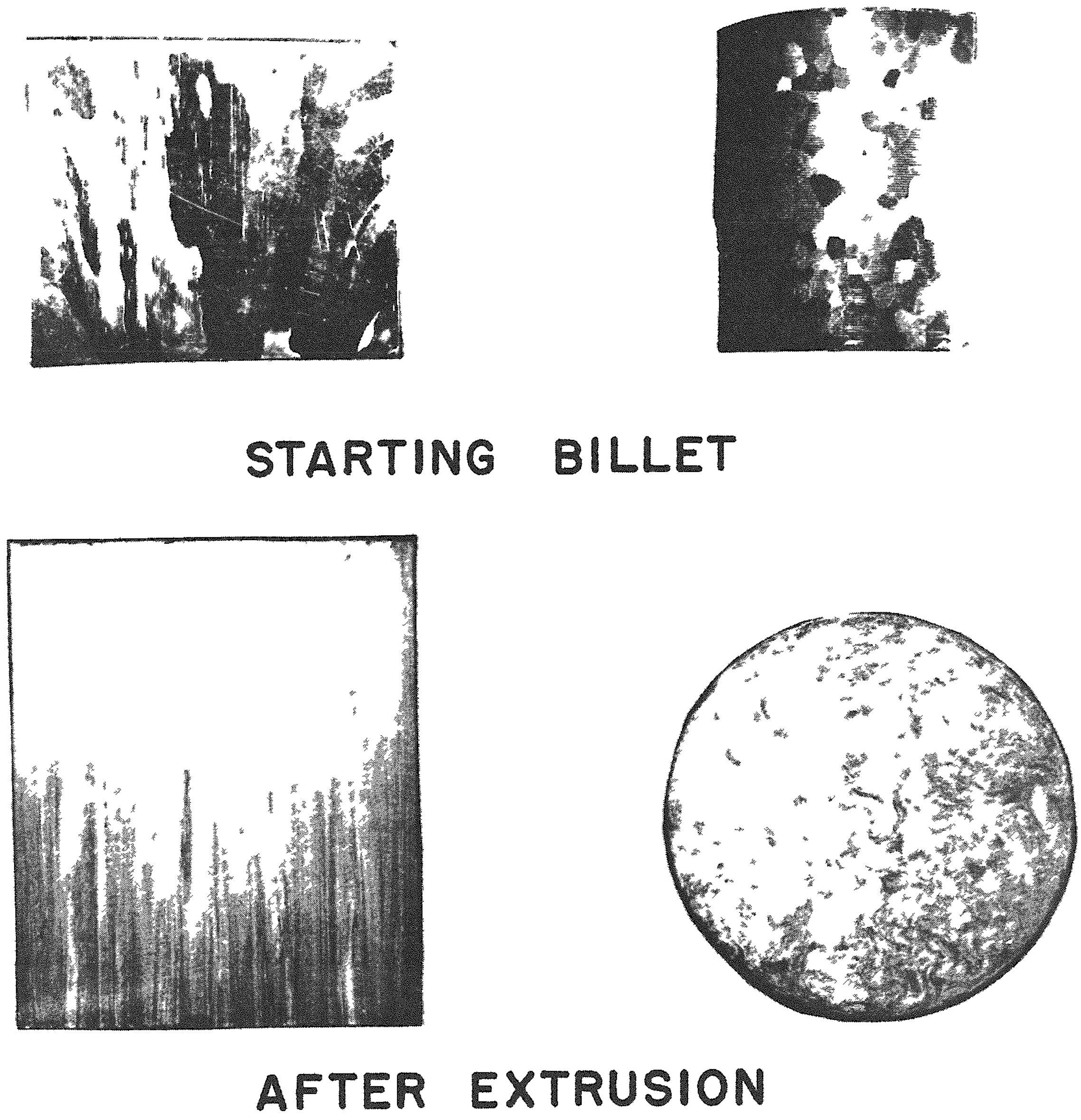


Fig. 14. Tantalum cold extruded on 500 ton press.

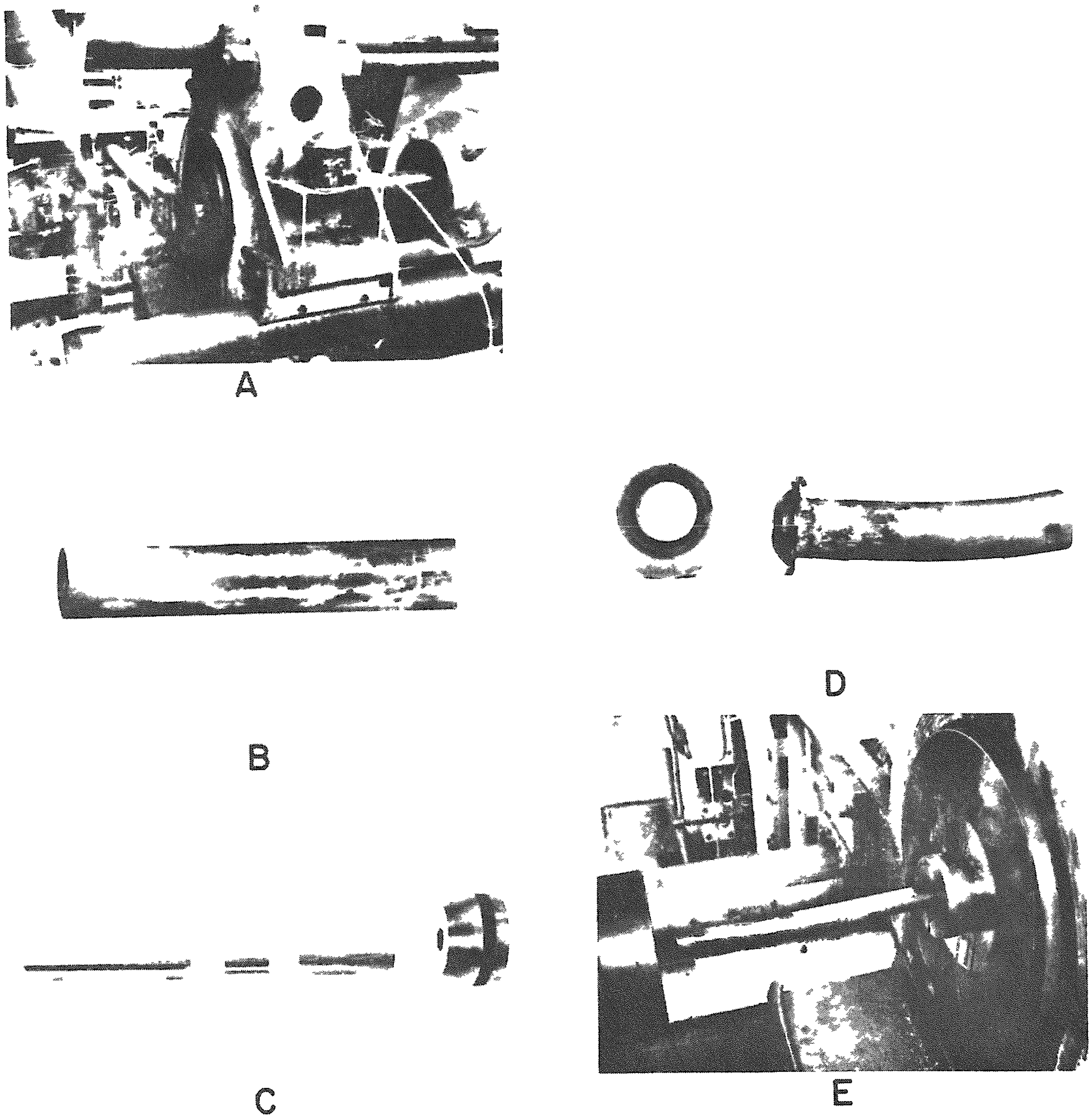


Fig. 15. Cold extrusion of tantalum (3 inch dia. to 1 inch dia.).

The hardness of the "as-rolled" rod varied from 82 to 93 R_B going from the outside to the center of the rod.

Figure 16 shows the starting 3 3/4 inch diameter billet approximately 15 inches in length with the 1.22 inch diameter rod which was produced from it by cold rolling. The finished rod was in one continuous length but was cut into sections for convenience in shipping.

2.2.3 Tooling

All of the impact extrusion work has been done using a 100 ton back geared crank press with a 17 inch stroke and a ram speed of approximately 109 feet per minute. All of the dies and punches used in conjunction with this press were made from AISI L-6 oil hardening tool steel.

All of the ironing procedure was done on a 75 ton hydraulic press which had a ram speed of approximately 0.97 feet per minute. The dies used for this operation were constructed of aluminum bronze encased in a mild steel retaining ring to provide adequate strength. The punches were made of AISI L-6 tool steel which had been polished in an axial direction and plated with approximately 0.00015 inch of chromium; the punches were then repolished in the same direction. This procedure tended to reduce galling and seizing during drawing and stripping.

Figure 17 shows illustrations of the punches and dies which were used throughout the fabrication process.

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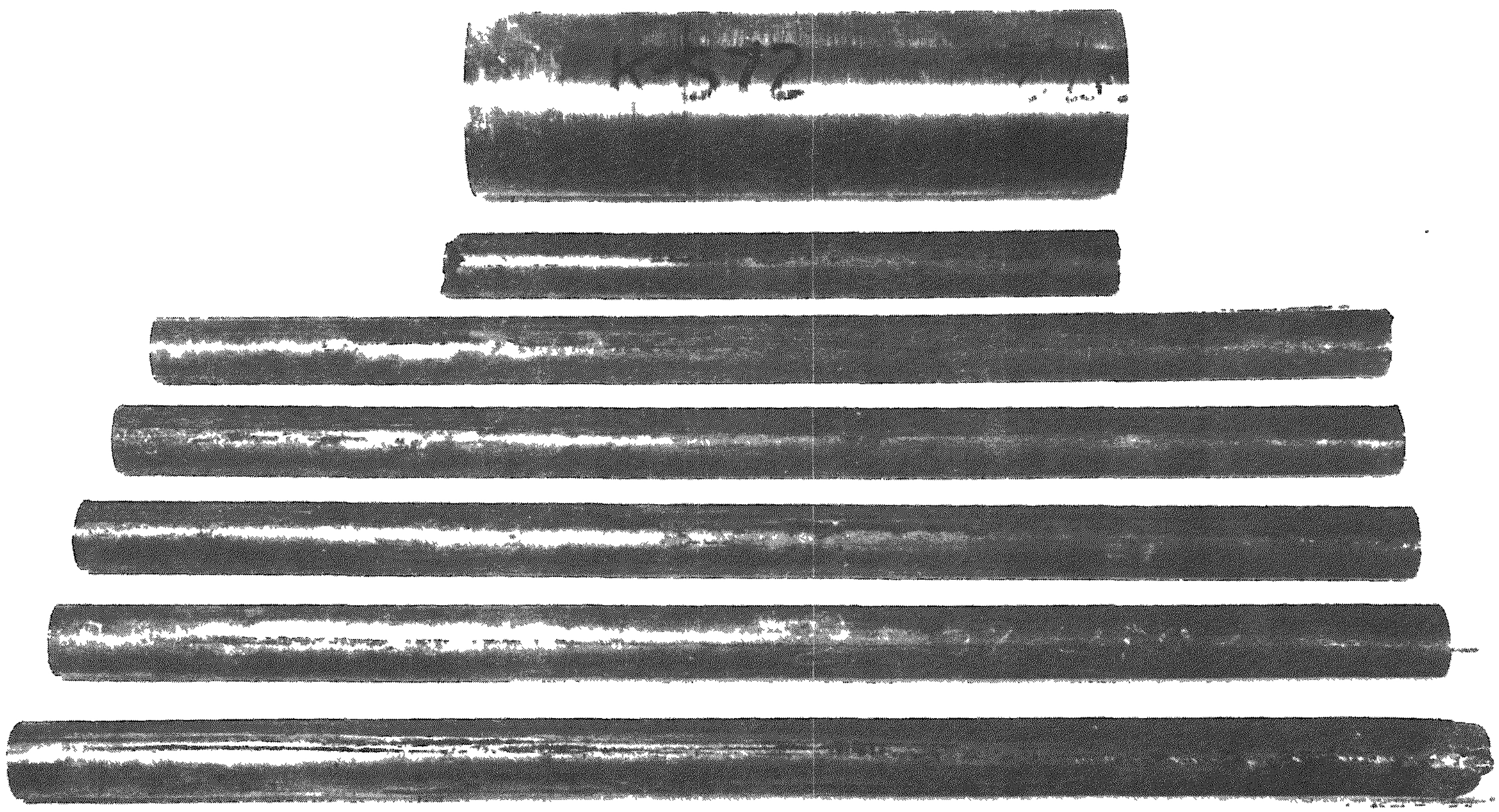
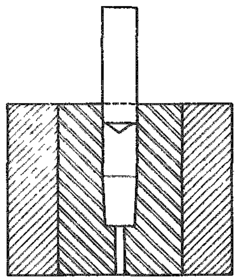
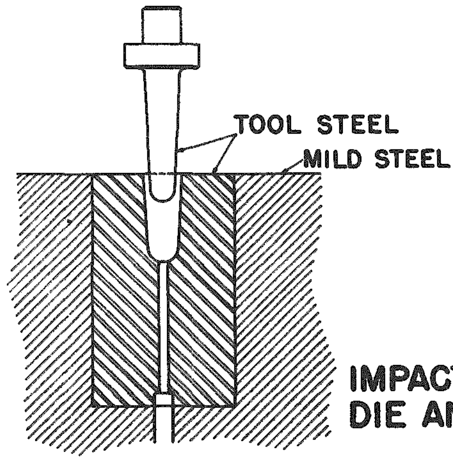


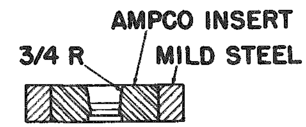
Fig. 16. Tantalum billet and rod rolled from a section of the billet.



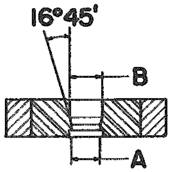
PREFORMING DIE AND PUNCH



IMPACT EXTRUSION DIE AND PUNCH

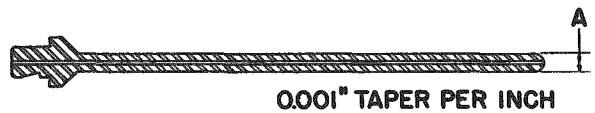


FIRST STAGE IRONING DIE



IRONING DIES

STAGE	A	B
1	.594	——
2	.547	.604
3	.510	.557
4	.479	.520
5	.454	.489
6	.428	.464



IRONING PUNCH

STAGE	A
1	.480
2	.455
3	.433
4	.412
5	.393
6	.371

Fig. 17. LAMPRE I impact and ironing tools.

The first die shown is used for pre-forming the 5/8 inch diameter material into a tapered slug which may be used in the impact extrusion die. The pre-forming operation also forms a conical centering recess in the top of the slug. This pre-forming procedure is used to insure a more nearly uniform wall in the finished product.

The second illustration represents the impact extrusion tools which have been used. The die and punch are tapered at a 2° angle to facilitate extraction of the formed piece.

The third and fourth pictures show cross sectional views of the ironing dies which are used. Aluminum-bronze is used because it is one of the few metals which does not seize and gall when used as a die material for drawing or ironing tantalum. The mild steel ring is shrunk around the aluminum-bronze insert to offset the tensile stresses set up during the ironing operation.

A cross section of the ironing punches is shown by the last figure. The 1/16 inch diameter hole through the punch is for the purpose of extracting the finished piece from the punch. The punch is also tapered slightly to aid in extraction.

2.2.4 Procedure

The 1 5/16 inch long slugs are first trimmed so that the ends are square. The slugs are then preformed with a 2° taper and a conical centering recess. The preforming operation is carried out in a hydraulic press and requires loads of 16 to 20 tons. If the preforming pressure is not adequate to form the tantalum slug to the configuration of the preform die, the wall thickness of the final thimble will vary considerably.

2.2.5 Impact Extrusion

Before impact extrusion of each lot of slugs, the crank press was carefully aligned so that the punch was within 0.0015 inch T.I.R. of being concentric with the die. The depth of penetration of the punch for the first impact stage was set by using mock-up slugs of lead.

The lubricant used for the impact extrusion was a thin coating of beeswax which was applied by dipping the tantalum slugs in molten beeswax at 150°C. The beeswax coating was then hardened by lowering the temperature of the slugs to 0 to -10°C; this hard beeswax shell was an excellent lubricant and almost completely prevented seizing and galling between the tool steel and tantalum.

Table II shows data which were collected during the impact extrusion of the tantalum slugs. The table shows the approximate reduction of the bottom thickness which was taken for each of the five impact stages. The solid bottom of the original preformed slug was about 1 inch and the solid bottom of the finished impact extruded thimble is about 0.280 inch. Table II also shows the average impact force in tons for each stage and the reduction in wall thickness.

If the thinning of the wall could be prevented, the operation might be done in less stages. Using straight walled punches and the dies instead of tapered tools might do this.

TABLE II

IMPACT EXTRUSION DATA

Stage	Reduction of Bottom, in.	Total Bottom Reduction, in.	Wall Thickness, in.	Wall Reduction, in.	Impact Pressure, tons
1	0.160	0.160	0.080	- - -	35
2	0.140	0.300	0.0775	0.0025	33
3*	0.140	0.440	0.075	0.0025	37
4	0.165	0.605	0.072	0.003	32
5	0.110	0.715	0.0705	0.0015	40

*Stress relieved after 3rd stage (900°C 1/2 hour).

The impact extruded thimbles are stress relieved in a vacuum for 1/2 hour at 900°C. The thimbles are then ironed through six stages with no intermediate heat treatments.

The slugs and thimbles are cleaned before each heat treatment as follows:

Remove beeswax with hot kerosene.

Degrease with acetone.

Bright dip in solution of 90% Con HNO_3-10% (80% HF).

Pickle in aqua Regia for 1/2 hour.

After the final ironing stage the thimbles are degreased and pickled in aqua Regia for 1/2 hour.

Figure 18 shows the steps which are taken in reducing the 5/8 inch diameter slug to the finished Ta thimbles. The heavy solid tip is pointed by machining after the first ironing stage to prevent excessive

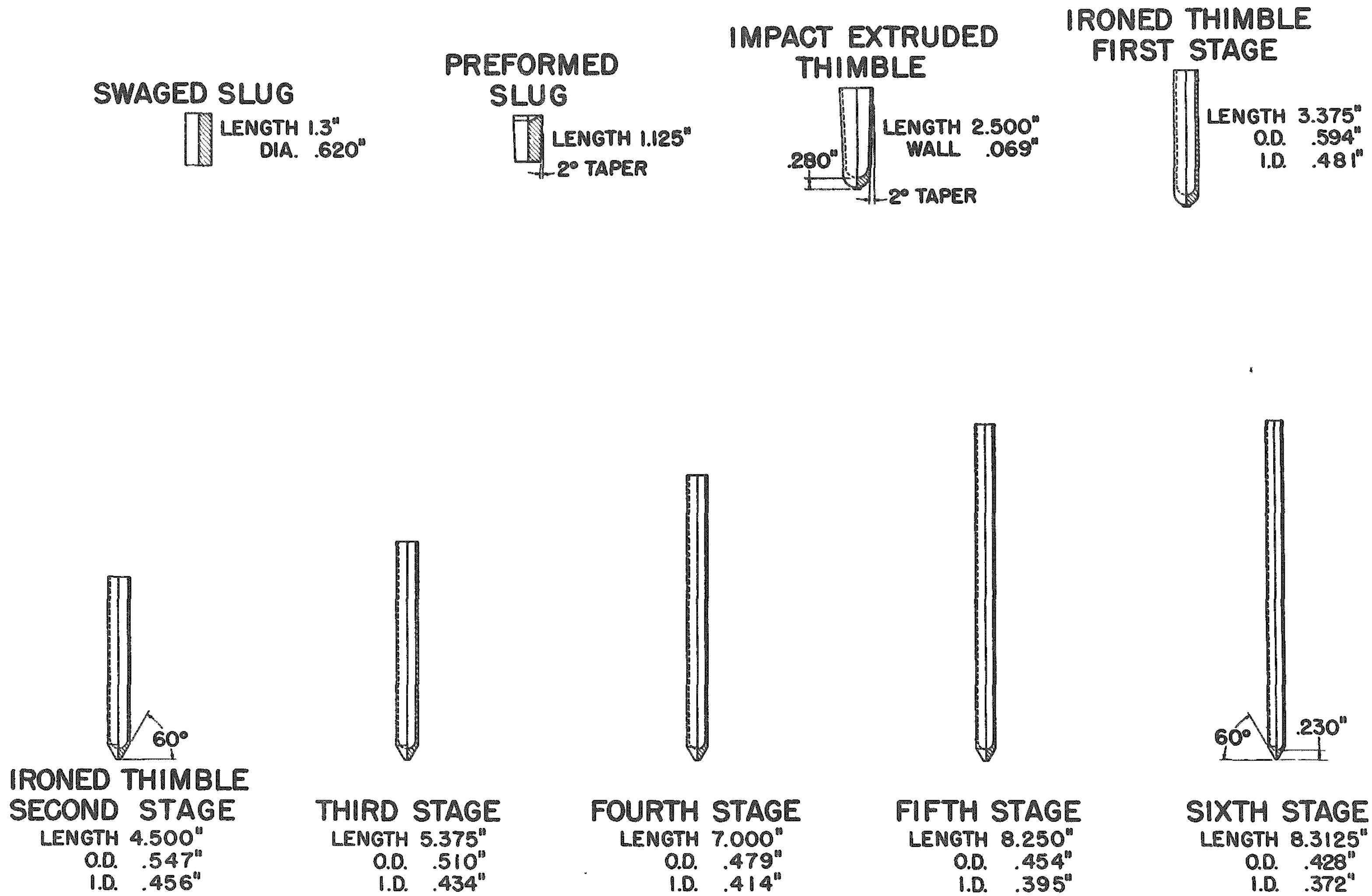


Fig. 18. Impact extruded and ironed LAMPRE I thimbles.

pressure build-up and possible distortion during ironing. The final tip configuration is also machined.

Table III shows the various stages of processing the respective dimensions and percent reduction of area. The pieces were annealed at 1200°C for 1 hour after extrusion to a 1 inch diameter rod. Stress relieving at 900°C was utilized after the final swaging step, after the third impact extrusion stage, and after the final impact extrusion stage. No intermediate heat treatments were used during the ironing procedure. Although the total reduction is 95.8%, this does not indicate the total amount of work in the piece or even that the structure would be the same as that of a piece worked to this degree without intermediate heat treatments.

TABLE III

DATA FOR DEEP-DRAWN LAMPRE TUBES

Stage	O.D., in.	I.D., in.	Reduction of Area, %	Total Reduction of Area, %
Extruded Rod	1.	- - -	- - -	- - -
Swaged Rod	3/4	- - -	43.8	43.8
Swaged Rod	5/8	- - -	31.7	61.6
Impact Extrusion	0.664	0.520	57.3	83.1
Ironing Stages				
1	0.594	0.481	26.3	87.6
2	0.547	0.456	24.8	90.6
3	0.510	0.434	20.9	92.8
4	0.479	0.414	19.4	94.2
5	0.454	0.395	13.8	95.1
6	0.428	0.372	9.9	95.8

Figure 19 shows the nearly linear relationship between the wall reduction and the forming or draw pressures required. Since the total reduction in area was changed so little during the ironing process, no evidence of the effect of this variable was noted. Had the per cent of total reduction of the area during ironing been greater, the effect might have been quite noticeable.

The data for plotting the curve shown in Figure 19 is recorded in Table IV. The lubricant for all of the ironing operations was a thin film of beeswax which had been applied molten by dipping and then chilled to about 0 to -10°C. This lubricant was far more successful for this operation than any other lubricant which had been tried.

It should also be noted that spring back during these operations was negligible.

TABLE IV

DEEP DRAWING DATA THROUGH 6 STAGES, LAMPRE TUBES

Stage No.	Wall Reduction, in.	Reduction of Wall, %	Draw Pressures, lbs.
1	0.015	20.8	2930
2	0.011	18.9	2310
3	0.009	19.2	1850
4	0.005	13.2	1070
5	0.0035	10.6	850
6	0.003	10.2	620

Figure 20 shows the major stages of fabrication of the tantalum thimbles and various grain structures formed by the fabrication procedure.

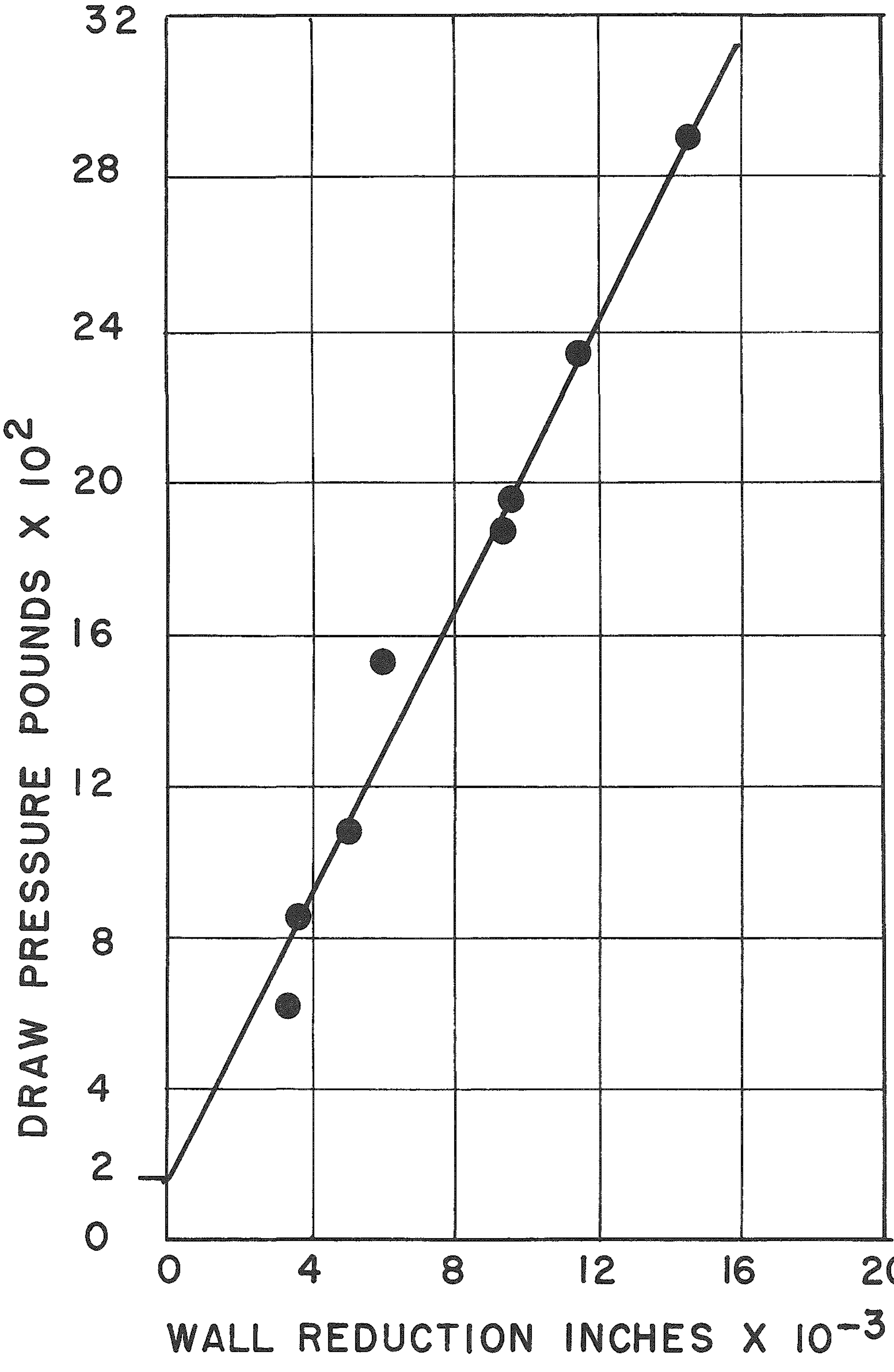


Fig. 19. Relation of draw pressure to wall reduction, deep drawing of LAMPRE tubes.

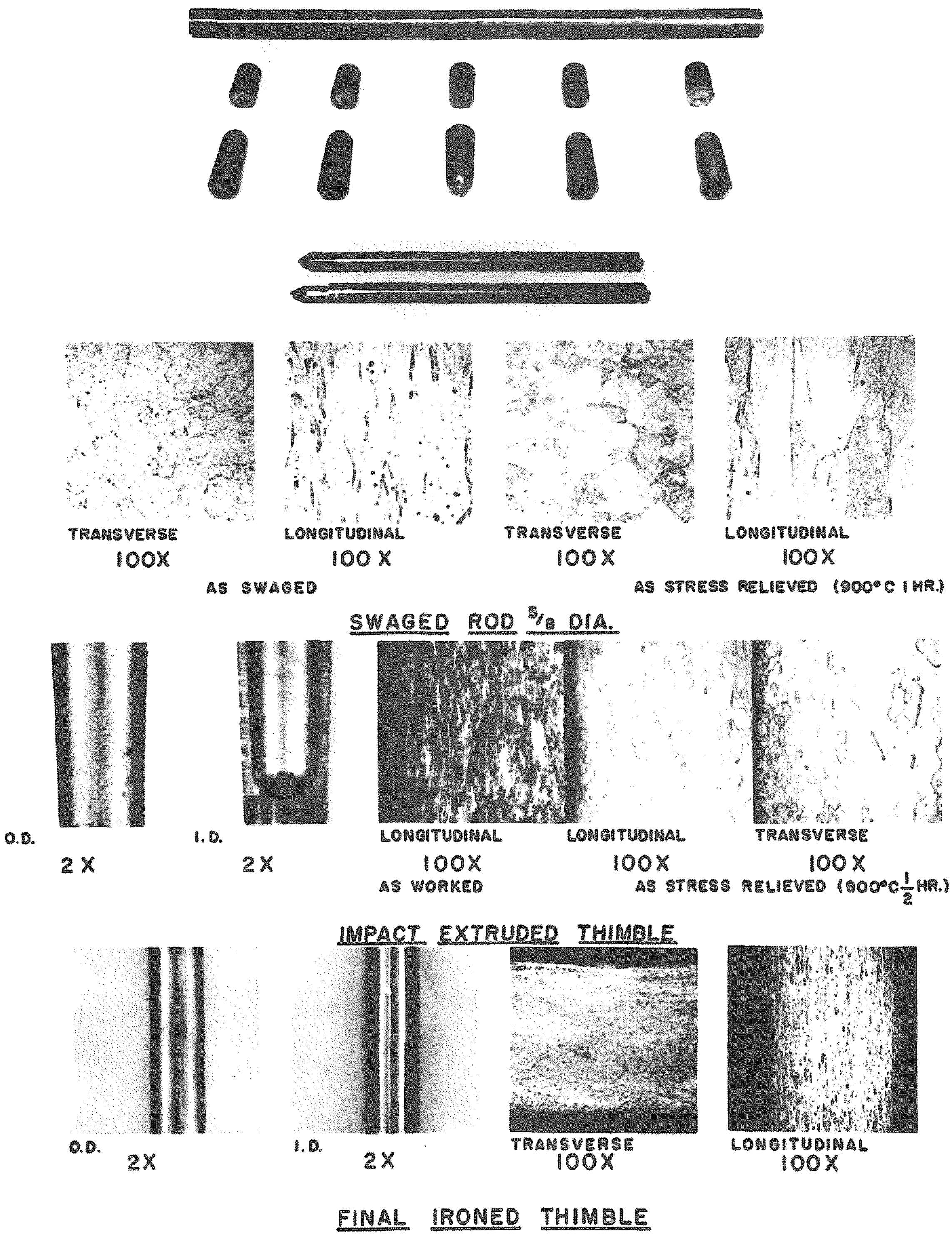


Fig. 20. Stages of fabrication of tantalum thimbles.

The top figures show the swaged 5/8 inch diameter rod, the preformed slugs, the impact extrusion product, and finally the final thimble showing a completed piece and a cut-away view.

The top group of micrographs shows the structure of the swaged rod. The pictures on the left show a transverse and longitudinal view of the as-swaged material. The pictures on the right show the same material after stress relieving at 900°C for 1 hour. These pictures would also be fairly representative of the structure of the preformed slugs.

The next group of pictures represent the surface and structure of the impact extrusion product. The first and second pictures show the outside and inside surfaces of the impact extruded material. The surface appearance at this stage seemed to be quite dependent on the grain size of the starting material, the smaller grain size producing a smoother material.

The third picture shows a micrograph of the as-extruded wall section. The last two pictures show longitudinal and transverse sections of the same material after stress relieving at 900°C for 30 minutes. It is apparent that considerable recrystallization has taken place.

The bottom series of pictures show the outside and inside surface of the final ironed thimble. The last two pictures show the highly worked structure of the final thimble.

CHAPTER 3

LUBRICANT TESTS

3.1 Equipment

The following equipment, illustrated in Figure 21, was used for testing the efficiency of various lubricants on tantalum sheet when pulled through aluminum-bronze metal dies.

1. 25,000 inch-pounds draw bench.
2. 50 ton press.
3. Two transducers, 0 to 3000 psi and 0 to 5000 psi.
4. Moseley Autograf recorder.
5. Heise Gauge, 0 to 5000 psi.

The draw bench was used to pull the tantalum specimens through the aluminum-bronze dies installed on the 50 ton press. The press, powered by a hand pumped hydraulic jack, applied a normal load on the specimen.

A 0 to 3000 psi transducer was installed to measure the draw bench oil pressure. Similarly, a 0 to 5000 psi transducer was placed to

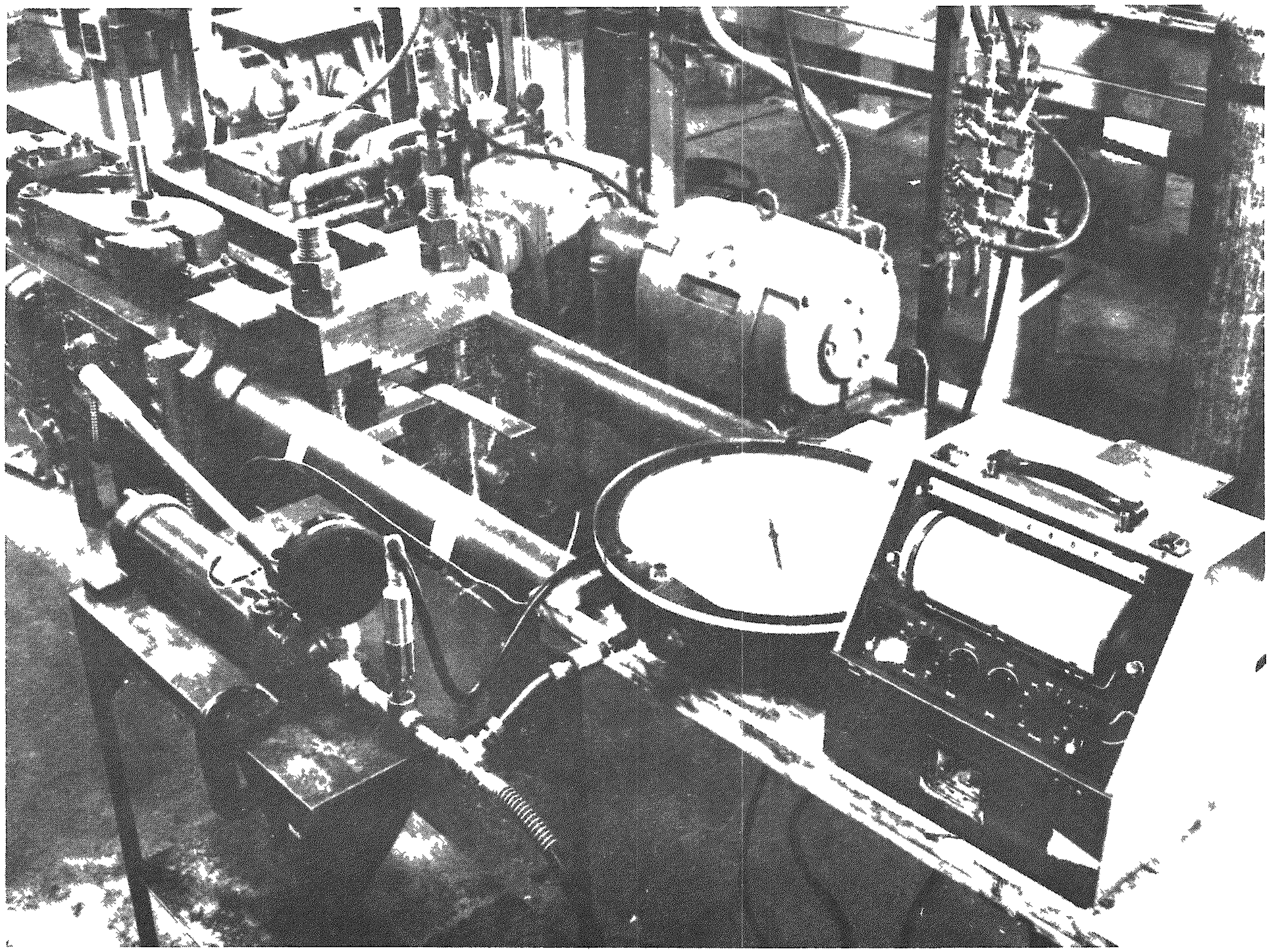


Fig. 21. Physical set-up for lubrication studies on tantalum.

measure the press oil pressure.

The signals from the transducers were supplied to a x-y recorder, the two channels controlling the movement of a drum and a pen. A sheet of 8 1/2 x 11 graph paper was used to record the pen trace. The drum, measuring the press (or normal) pressure, was calibrated with a precision gauge to record 0 to 5000 psi. Likewise, the pen channel, recording the draw pressure, was calibrated to 0 to 2500 psi.

3.2 Testing

The test specimens were cold rolled tantalum strips two inches wide, 0.072 inch thick, three to four feet long. The eight lubricants listed below were tested on both "as-rolled" and anodized surfaces.*

1. Jaytolube on Jaytolube precoat.
2. 50% Jaytolube, 50% beeswax on Jaytolube precoat.
3. Glycerine.
4. 1/3 Beeswax, 1/3 trichloroethylene, 1/3 glycerine.
5. 74.2% Beeswax, 13.3% kerosene, 12.5 glycerine.
6. Beeswax.
7. Texaco drawing compound #2.
8. Dag 41 (colloidal graphite in naptha).

The beeswax and the beeswax-Jaytolube mixture were applied to warm

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*Anodizing treatment: Make chemical clean piece anodic in 1.% H_2SO_4 at 120 v. for 10 minutes.

tantalum followed by a cold water quench. The other lubricants were all applied at room temperature.

The following conditions were common for all tests.

1. Room temperature.
2. 600 psi preload on press dies before draw bench was operated.
3. Draw speed approximately 2 feet per minute.
4. Three runs were made using each lubricant.
5. The normal load was increased in 250 psi increments with a pause at each step to allow the recorder to stabilize.
6. Aluminum-bronze was used for the die material in all tests.

3.3 Results

The three tests for each lubricant were plotted by the recorder on one paper starting from the same origin. An average curve was obtained by drawing a line through the three experimental curves.

Six arbitrary valves, of 600, 1000, 2000, 3000, 4000, and 5000 psi normal pressure, were chosen on each average curve and the corresponding draw pressure was determined. The coordinates were converted to actual stresses on the tantalum strips and Table V was prepared from these data.

TABLE V

NORMAL PRESSURE VS DRAW PRESSURE

COLD ROLLED TANTALUM ON ALUMINUM BRONZE

	DRAW PRESSURE PSI							
	AS ROLLED TANTALUM							
Normal Pressure psi	Texaco No. 2	Dag 41	Glycerine	Jaytolube-Jaytolube Precoat	Beeswax Trichloroethylene Glycerine	Beeswax	Beeswax Kerosene Glycerine	Beeswax Jaytolube Jaytolube Precoat
700	3000	9200	15000	2000	5100	4100	3000	2000
1200	4100	13800	20000	3000	10800	6100	4100	3000
2400	8200	26600	46000	5100	17100	9200	6100	6100
3600	13100	39100	68000	9200	20400	13800	9200	9200
4800	27500	52000	- - -	20400	26300	18400	14500	12200
6000	49300	64400	- - -	35500	32000	22000	20400	15000
	ANODIZED TANTALUM							
700	3000	3000	10200	2000	3600	5100	4100	1500
1200	4500	5100	19400	4100	4500	6100	4500	2000
2400	6100	11800	50600	9900	8200	9900	6100	4600
3600	15000	20400	76900	14500	10200	13100	9200	7200
4800	37500	33500	- - -	22000	14500	16400	11200	9200
6000	81500	46700	- - -	31600	17700	20400	13100	11800

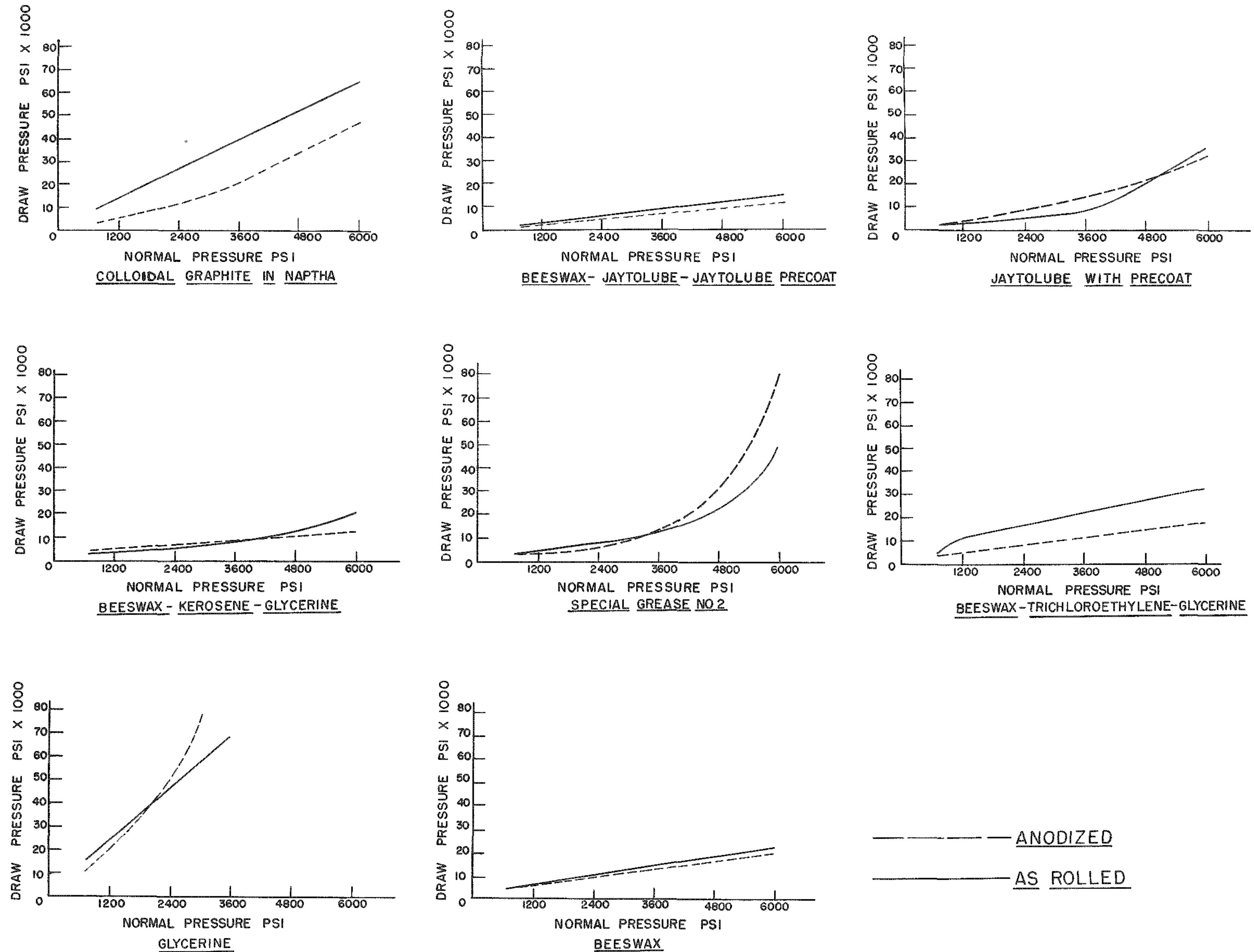

Fig. 22. Effect of various lubricants on Ta drawability using Al bronze dies.

Table VI compares the various lubricants on as-rolled and anodized tantalum. They are listed in order of decreasing effectiveness.

TABLE VI

COMPARISON OF SEVERAL LUBRICANTS IN ORDER OF EFFECTIVENESS IN DRAW TEST

	As Rolled	Anodized
1.	Beeswax, Jaytolube, Jaytolube Precoat	Beeswax, Jaytolube - Jaytolube Precoat
2.	Beeswax - Kerosene - Glycerine	Beeswax-Kerosene-Glycerine
3.	Beeswax	Beeswax-Trichloroethylene-Glycerine
4.	Beeswax - Trichloroethylene-Glycerine	Beeswax
5.	Jaytolube, Jaytolube Precoat	Jaytolube, Jaytolube Precoat
6.	Texaco #2	Dag 41
7.	Dag 41	Texaco #2
8.	Glycerine	Glycerine

Graphs demonstrating the effect of normal pressure on draw pressure were constructed from the recorded data and are presented in Fig. 22.

Reproducibility of data, in general, was as follows:

Good Lubricants: The three experimental curves were within 2 to 6% of each other. Consistent results were obtained on different days.

Poor Lubricants: The results were not consistent. Curves varied 10 to 30%.

Texaco, Dag 41, and glycerine are considered poor lubricants. The best results for each of these lubricants were reported.

In conducting these tests, beeswax was considered a good lubricant and glycerine a poor one. Complete tests were only run on promising lubricants, with a few poor ones shown for comparison.

Other lubricants tested, their values following, in general, between Texaco and glycerine were:

1. Magic Tap
2. Moly Lube
3. Johnson Wax Draw
4. Lubriplate
5. Other beeswax base lubricants with varying amounts of Linseed oil, Texaco No. 2, Kerosene, and Glycerine.
6. Johnson Glo Coat. This was a poor lubricant but shows promise as a precoat.

On the basis of the tests performed, beeswax and beeswax base lubricants are considered the best known for drawing tantalum on aluminum-bronze tools. This conclusion is consistent with known practice.

The lubricant used for deep drawing was a thin coat of beeswax applied by dipping the blank or pieces into molten beeswax at 150°C.

In the ironing operation, beeswax applied as a deep drawing was used but the beeswax coating was then hardened by lowering the temperature of the tantalum pieces to 0 to -10°C. This hard beeswax shell almost completely prevented seizing and galling between the tool steel and the tantalum.