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ABSTRACT

Inasmuch as it is extremely difficult to get a uniform distribution of uranium in aluminum-uranium alloys by conventional melting and casting techniques, the "Chip Method" has been devised and applied to the fabrication of homogeneous 85 w/o Al-15 w/o U alloy foils. A description of the experimental techniques and results is given. Alloy ingots were made by conventional vacuum melting and casting techniques. Each ingot was machined completely into chips, and, after being degreased, the chips were thoroughly mixed by tumbling. (If N ingots are needed for the total foils required, the mixed chips from each ingot are divided into N equal subportions, and the subportions cross-blended so that a uniform uranium content is achieved for all chips.) Chips were then cold-compacted, canned, evacuated, hot compacted, and hot extruded into a 1/8" x 1-1/8" flat. The extruded flat was cold rolled to 0.015" ± 0.0003" thick foil and milled to the proper width. Chemical analyses of the extruded flats and rolled foils showed that homogeneous foils were obtained by the Chip Method.

USE OF A CHIP METHOD TO PRODUCE HOMOGENEOUS ALUMINUM-URANIUM ALLOY FOILS

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I. Introduction

The work reported below is the result of a need by Brookhaven National Laboratory for foils containing a homogeneous dispersion of U235 for BNL's Liquid Metal Fuel Exponential Experiment. After the concentration of U235 in terms of grams per square inch of foil surface had been fixed, BNL decided on a foil composition and thickness which were a compromise between the need for a homogeneous concentration of U235 and a minimum amount of impurities and poison. If unalloyed uranium were used, the resulting foil would have been so thin that very small varieties in thickness would have represented large percentage variations in U235 concentration. Accordingly, in November, 1955, BNL requested (Job R-76) bids on foils containing 84.1 w/o Al-15.9 w/o UO_2, these foils to be 0.970" ± 0.002" wide, 0.015" ± 0.0003" thick, and 24.000" ± 0.010" long. The homogeneity specification was that the uranium content of the foil should not vary by more than ± 1/2% of the uranium present, i.e., w/o $UO_2 = 15.9 \pm 0.08$.

To see if the homogeneity specified by BNL were obtainable, a calculation was made at NMI to see what homogeneity could be expected in terms of the raw materials--aluminum powder and UO_2 powder--and size tolerances. These calculations(1) indicated that it is not possible to obtain the required homogeneity by using aluminum and UO_2 powders as starting materials:

(1) Aluminum powder is made from 99.5% pure pig, and the resulting oxygen content is variable.

(2) Enriched UO_2 is variable in uranium content and tends to change composition with time.

(3) At ORNL it was found that UO_2 and aluminum powders are not always stable together.(2)

If an aluminum-uranium alloy is used instead of aluminum-UO_2 powder, high purity aluminum (99.9% or better) and uranium metal of known and unchanging composition can be used. However, aluminum-uranium alloys are very susceptible to segregation during solidification because of the settling of the relatively dense UAL_4 compound particles.. In a 15 w/o uranium alloy, which consists of particles of UAl_4 compound in a matrix of pure aluminum, the density (3) of the aluminum is 2.70, that of the compound 6.06, and of the over-all alloy 3.06 gm/cm^3. To overcome the problem of segregation, it was proposed to BNL that NMI develop a scheme called the "Chip Method", described in detail in the next section, as an alternative to the aluminum - UO_2 mixture. In May, 1956, BNL authorized NMI to proceed with the development of the Chip Method to serve as a basis for the future production of 6400 foils. Because of the limitations of time and funds, an exhaustive investigation of the Chip Method was not possible. This report presents the results obtained, and Appendix A gives the recommended procedure for production of 6400 foils.

II. The Chip Method

As discussed above, it is possible to make alloy castings consisting of pure aluminum and UAl_4 compound by conventional vacuum melting and casting techniques. However, such castings are very difficult to produce without appreciable segregation. If castings could be reduced to powder, homogeneous foils could then be made by conventional powder metallurgy techniques. However, such practice would introduce an appreciable amount of undesired oxygen, and, furthermore, it is difficult to produce powder from such a casting. It was felt that if a casting could be reduced to chips, the chips could be handled by quasi powder metallurgy methods, the resulting foil would be homogeneous, and the oxygen pick-up would be very small. In outline form, the steps in the procedure are as follows:

1. Melt and cast in vacuum.
2. Machine entire casting into chips and degrease.
3. Blend chips.
4. Cold compact.
5. Evacuate and hot compact.
6. Hot extrude into a flat.
7. Cold roll to finish size.

If there are no losses in melting, so that the over-all uranium content of the casting is known from the charge weights, the procedure becomes an absolute method, and the only blending required is for the chips of each casting. However, if there are losses in melting, cross-blending is required, inasmuch as more than one melt is required eventually to furnish stock for 6400 foils. (It should be kept in mind that the homogeneity requirement for the BNL foils is not concerned with the absolute uranium content of the foils, but rather that the uranium content of each square inch of foil shall be the same.) For example, if five melts were needed, the matrix scheme shown below illustrates the method of blending and cross-blending:

A_1	A_2	A_3	A_4	A_5
B_1	B_2	B_3	B_4	B_5
C_1	C_2	C_3	C_4	C_5
D_1	D_2	D_3	D_4	D_5
E_1	E_2	E_3	E_4	E_5

The chips from each melt (A, B, C, D, E) would be blended and each batch of chips divided into five parts (e.g., A_1, A_2, A_3, A_4, A_5). Then A_1, B_1, C_1, D_1, and E_1 would be blended together, and similarly for A_2-E_2, A_3-E_3, A_4-E_4, and A_5-E_5. The limitations of equipment and criticality requirements determine the size of any one batch being blended.

It was realized at the outset that a Pebble Method might eventually be preferable to the Chip Method. In the Pebble Method, shot would be formed directly from the melt, and the molten alloy would be kept hot enough so that each pebble had exactly the same composition as every other pebble from a given melt. The pebbles could then be consolidated and processed into foil. Such a scheme would avoid the steps of making chips and blending. However, the limitations of time and funds did not permit the development of the special equipment that would be needed for the Pebble Method.

III. Application of Chip Method to BNL Requirements

On April 14, 1956, at a meeting held at NMI among H. Kouts and D. Gurinsky of BNL and several representatives of NMI, the following objectives for the application of the chip method to BNL's problem were evolved:

1. Develop a method of producing homogeneous foils of aluminum-uranium alloy, with natural uranium metal and 99.9% pure aluminum as raw materials,

said method to serve as a successful basis for future production of foils containing enriched uranium to meet the following specifications, in terms of enriched uranium:

a. Dimensions:

Thickness: 0.01500 ± 0.00030 inches
Width: 0.970 ± 0.002 inches
Length: 24.000 ± 0.010 inches

b. Composition of foil:

Grams of U235/in^2 of foil = 0.09873 ± 5%
= 0.09873 ± 0.00494 grams

c. Homogeneity: w/o U in any sample of foil having a surface area of one square inch or less shall not vary by more than ± 1-1/2% (relative) of the average w/o U of all foils. Specifically, the composition, in terms of total uranium shall be

14.13 w/o ± 0.21 w/o uranium, the remainder to be aluminum plus whatever impurities are introduced by using the raw materials previously specified.

2. Develop the method as far as possible, within the limits of the money allocated for this work, to serve as a basis for writing a feasibility report for subsequent production of 6400 foils containing enriched uranium.

The total U235 content for the eventual 6400 enriched foils is 14,720 grams. Originally, it was assumed that a 90% yield would be obtained which would require about 16 kg of U235. From criticality considerations, the maximum amount of U235 in any one melt was taken to be 4 kg, which would mean a minimum of 4 melts. It was decided originally that 5 melts, each containing about 3.2 kg of U235 and having a total weight of about 24.3 kg (53.7 lbs) would be suitable for the equipment at hand.

It was not until the work was well along that it was learned that the safety requirements concerned with criticality are much more stringent for chips than for melts of massive pieces. The contained U235 content of any one batch of chips was said to be a maximum of 1000 grams because of the danger of accidental flooding. The maximum batch of chips for the 14.13 w/o uranium would, therefore, be about 7.08 kg, or 15.16 lbs, of aluminum-uranium alloy.*

IV. Experimental Methods and Results

As mentioned above, most of the work was done with large melts as a source of chips.

A. Materials for Melts

Uranium was obtained from a scrap end of extruded tube No. 9599, made from reactor grade uranium. The tube was cut into small pieces which were pickled in 50% nitric acid.

Aluminum was obtained from Alcoa 99.9% pure pig, which was degreased prior to melting.

Graphite for crucible and mold was machined from crucible grade material. No wash was used.

B. Melting and Casting Techniques

Two melts were made, each weighing about 53 pounds. The analysis aim of the first melt was 14.24 w/o uranium, and that of the second melt

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* BNL is presently looking into the possibility of using specially designed poison cans that will safely contain considerably more than 1000 grams of U235. If poison cans do prove feasible, then considerably more than 7 kg of chips can be handled at one time.

deliberately higher, 15.15 w/o uranium. It was felt that having two melts deliberately varying in over-all composition would act as a check on the efficiency of the cross-blending operation.

Uranium and aluminum were added to a graphite crucible, with the uranium placed around the stopper rod at the bottom as shown in Figure 1. After a vacuum was obtained, the charge was melted by induction heating, and superheated to above the melting point of the uranium to insure its complete solution. The melt was poured by lifting the graphite stopper rod. Details of the melting and casting conditions are listed in Table I. The first two cylindrical castings were 8" in diameter and about 10" long, and no attempt was made either to minimize segregation or to get a sound casting. Essentially the entire charge weight was obtained in the finished casting--the losses were 48.2 and 16.0 grams, respectively, out of a total charge weight of nearly 25,000 grams. From these results, it appears that this may be an absolute method; the maximum error, assuming the entire loss to be uranium, was only 0.17 w/o in the first ingot, and 0.05 w/o in the second.

A third melt of a smaller size was made in a similar manner to the first two. Its purpose was to check the losses obtained in a melt of the maximum size allowed by present criticality considerations. The 15.6 lb melt was poured to form a casting 5-1/2" diameter and 6-3/8" tall. The metal loss during this melt was 0.286%, and consisted of metal adhering to the crucible bottom and stopper rod, and also dross remaining in the crucible. In general terms, the relative metal loss increases as the surface/volume ratio of liquid increases. In this case, the maximum error, assuming the entire loss to be uranium, was 0.25 w/o. This indicates the desirability of cross-blending the chips produced from this size melts.

The analysis of extrusions made from cross-blended chips from the first two melts averaged close to 14.70%, which is the average of the over-all composition calculated from the charges, as discussed in detail in a later section.

C. Production of Chips

A piece of scrap 15% uranium-aluminum alloy was used for determining the proper lathe conditions for obtaining chips, such as feed, speed, depth of cut, and tool design. After a suitable set of conditions was determined, the two castings were reduced entirely into chips. The conditions for making chips were as follows:

Tool: Stellite with gentle chip breaker (it is important that the shoulder on the chip breaker be curved rather than sharp)

Feed: 0.010 - 0.020"/revolution

Speed: About 600 surface ft. per minute

Depth of cut: 0.010" on radius

Cutting fluid: None

Although no cutting fluid was used, a slight amount of lubricant from the lathe carriage was picked up by the chips.

The resulting chips were about 1/4" long, and about 0.1" square. Based on a sample of 100 chips, the average weight per chip was 0.15 gram, which would mean an average calculated volume of about 0.05 cm^3.

The time required to reduce each 54 lb casting to chips was about 8 hours.

In each case, a number of small pieces of stock remained which could not be further turned in the lathe. These small pieces were reduced to chips by using a milling machine. When these pieces became so small that they could no longer be held in the vise for the milling cutter, they were flattened with a hammer and sectioned with snips into pieces approximately the same size as the lathe chips.

Because of the lack of time and funds, no elaborate attempts were made to contain all the chips produced by the lathe. Consequently, with a relatively crude guard around the chuck and a partition across the back of the lathe, a very small amount of chips was thrown onto the floor and was lost. In working with enriched chips, it would be necessary to install a more elaborate chip collection method.

D. Cleaning of Chips

Initially, some of the as-machined chips were cold compacted, and it was observed that a noticeable amount of oil exuded from the compact. It was felt desirable, for this reason, to degrease the chips before compacting.

The chips were degreased by placing about two pounds at a time in a wire mesh basket, and dipping into a sequence of three containers, the first two containing trichlorethylene, and the third acetone. The chips were then spread out to dry on a large piece of paper. During the dipping operation, some fines went through the screen into the containers of liquid, the amount being very small compared with the total weight of chips per batch.

E. Blending of Chips

A tumbling barrel was designed so that the entire amount of chips from one of the 53.6 lb castings would occupy slightly less than one-half its volume. The loose volume of this amount of chips was 12-13 gallons. The barrel, formed from 1/8" 2S aluminum sheet, was 20" long and 20" in diameter, as shown in Figure 2. Four longitudinal dividers, 3" tall, were placed at 90° intervals perpendicular to the circumference within the barrel; their purpose was to effect better mixing by raising the chips to a higher position during tumbling. The barrel cover, also of sheet aluminum, was made with a small window of plexiglass for viewing the mixing action. Since the barrel was fabricated by cold forming and riveting, rather than by welding, it was not entirely tight, and very fine particles of dust could work their way slowly through the joints during the tumbling operation.

The tumbling barrel was rotated by a pair of 5" diameter hard rubber rolls, 12" center to center, which were in turn motor-driven. Rotation of the tumbling barrel was at approximately 30 rpm. In order to effect mixing of the chips in a longitudinal direction in the barrel, a wooden wheel was secured to the outside of the rolls, and the barrel rotated while in a slightly tilted position during part of the procedure.

During the tumbling procedure, some fines were produced from the abrasion of chips on one another. These fines formed a coating around each chip particle which could be rubbed off with the fingers. After mixing, the chips were dark in color, and had a worked appearance. The abrasive action also reduced the volume of the loose chips to about 80% of that occupied before mixing.

For the initial procedure of blending the entire volume of chips produced from each 53 lb casting, a total mixing time of about 9 hours was used. During 1-2 total hours of this time, the barrel was rotated in the tilting position, with top or bottom end upward alternately for 15 or 30 minute cycles. Approximately one ounce of dust was lost from the barrel during this blending operation because of its loose construction. For the blending of enriched chips, it would be mandatory to fabricate a tight container, preferably of welded construction, having a leak-proof cover. Because of the limitation on the weight of enriched chips that could be blended at one time, a container much smaller than that described above would be used.

For the cross blending operation, 20% by weight of the chips from each of the castings AX57 and AX58 were used, (this is the procedure followed in the five melt scheme) or a total of about 21.5 lbs. 4872 grams each of chips from AX57 and AX58 were carefully weighed, and blended for 2.5 hours in the tumbling barrel. During the procedure, the barrel was occasionally turned end for end to secure additional mixing longitudinally. Two separate cross-blending operations were performed in the same manner. These two groups of blended chips are hereinafter referred to as Group 1 and Group 2 mixed chips. These chips should have a nominal composition half way between that of AX57 and AX58, or about 14.69%.

Due to the criticality requirements as previously mentioned, instead of the cross-blending of one-fifth of the chips from each of five melts a total of about 20 batches of chips (from 20 melts) would be required for cross-blending for the eventual production of 6400 foils.* Initially, each of the 20 batches of chips would be mixed individually. Then, each batch would be subdivided into 20 portions from each of the other batches. This procedure would require a total of 40 blending operations. The total

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*However, see footnote, p. 9

number of melts required would depend upon the non-usable losses of alloy from the various operations.

In the event that the losses in melting could be kept so low that the anaylsis of each melt would be known absolutely, all being exactly the same, then no cross-blending of chips would be necessary. From the data of Section B, however, it appears that the melting loss is of such a magnitude that each melt could not be assumed to be of exactly the same composition. Thus, cross-blending of the batches of chips is recommended as part of the procedure.

F. Cold Compacting

In the steps of the process subsequent to the blending of chips, experimental work was initially performed on small-scale equipment before proceeding to the full-scale work. This was done in order to take advantage of some tooling already available, during the period in which the full-scale equipment was being secured. It was hoped that results of the small-scale work could be applied more or less directly to the full-scale procedure.

The small-scale work was performed with the chips machined from a scrap piece of uranium-aluminum alloy, of 15% nominal composition. These chips had not been subjected to any blending operation, since there was no particular need for achieving complete homogeneity in them. Thus, they differed from the chips from AX57 and AX58 in that they had not been subjected to the abrasive action from the blending procedure, and that they did not have the fine dust coating formed during that operation. Initially, also, they were not degreased.

The equipment used in the small-scale cold compacting work is shown in Figure 3. It consisted of a hardened steel packing die of ID = 2.000", a soft steel sleeve of OD = 2.000" and ID = 1.927", and hardened pistons of diameters 1.966" and 1.919". The larger packing piston was used with the packing die alone; the smaller piston with the sleeve inserted in the packing die. Compacting was accomplished by the use of a vertical press with a movable bottom platform, of maximum capacity about 275 tons.

A summary of the results of the cold compacting work is listed in Table II. It was felt that more dense compacts would result if part of the chips were added at a time, with pressure being exerted after each addition. In Compact No. 1, using the sleeve, nine separate chip additions were made, with 50 tons pressure applied after each addition. The resulting compact was rather loosely packed, and the nine individual "pancakes" came apart very easily, the pressure not having been enough to consolidate them. The approximate density, based on weight and measurement of cross-sectional area and height, was about 90% of theoretical (for a 15% alloy). Also, since these chips had not been degreased, oil exuded from the compact. This oil assisted in the removal of the compact from the sleeve by acting as a lubricant on the wall. In Compact No. 2, the compacting pressure was approximately tripled, with the result that the piston could not be removed upward due to metal which had become lodged between the piston and the sleeve wall. It was necessary to cut the soft steel sleeve in order to remove the compact. The compact, at this pressure, appeared very dense, being approximately 99% of theoretical. Compact No. 3 was made in a similar manner, except that a considerably larger portion of chips was used. This compact, which was about 2" tall, was somewhat more dense at the top (piston end) than at the bottom, due to absorption of some of the compressive force by friction along the walls. Near the bottom, also, the inner sleeve surface showed indentations from the pressure of the chips' forcing the soft steel against the packing die inner wall. This compact was not quite as dense as the previous one. In Compact No. 4, the sleeve was omitted, with the piston and the packing die only being used. A dense compact was obtained, with smooth walls, except for a few scoring marks formed during removal from the packing die. Since the packing die had been hardened, the packing pressure did not cause chip indentations in this case such as with the soft steel sleeve. Again oil was forced from the compact, which assisted considerably in allowing easy removal from the packing die. It was felt, however, that oil contamination of the compact interior was objectionable, and would lead to difficulty in subsequent steps. For this reason, these preliminary chips were subjected to a degreasing treatment using trichlorethylene, and all further compacts were made from degreased chips. Compact No. 5 was made primarily to check the efficiency of the degreasing operation. There was no sign of oil on the resulting compact. More severe scoring of the compact walls was noticeable, however, due to the lack of lubrication during removal from the packing die. In Compact No. 6, four separate chip additions were made, with about 10 tons pressure being applied after each addition; then, after being fully loaded, the pressure was gradually increased to 150 tons. In order to remove this compact from the packing die, which meant exceeding the frictional force between the compact and packing die, more than 50 tons force was required. The increased wall friction due to the lack of lubrication was evident, with some scoring of the compact surface. This also resulted in a considerable apparent density difference between top and bottom of the compact. Compact No. 6 was considerably less dense (95% of theoretical) than Compact No. 4, with the lack of oil lubrication being the only difference. The possibility of the application of a lubricant such as MoS_2 to the walls of the packing die was considered. It was felt, however, that contamination of the chips might result, and that the disadvantages outweighed the advantages to be gained.

The remainder of the small scale cold compacts (Compact Nos. 7 - 16), were made in essentially the same manner as Compact No. 6, with about 100,000 psi pressure being exerted. The densities obtained were approximately the same as that of Compact No. 6, from a visual estimate only. In cases where, after removing the compact, much flash remained on the walls of the packing die, a hot NaOH solution was used to dissolve the U-Al alloy before making another compact. In Compacts No. 9 and 10, a steel insert was placed at the bottom of the packing die, so that the compact had a 45° taper at its bottom. This nose portion was quite loosely packed, due to absorption of force by the tapered steel insert. In general, the compacts No. 7 - 14 had poor surface condition. This was not felt, however, to be of any particular disadvantage for the subsequent steps.

The equipment used for the full scale cold compacting work is shown in Figure 4. It consisted of a hardened steel packing die of ID = 3.515", a steel sleeve of OD = approx. 3.514" and ID = 3.370", and hardened pistons of diameters 3.514" and 3.362". The larger piston was used with the packing die alone, and the smaller piston with the sleeve inserted in the packing die.

The same vertical press, of capacity 275 tons, was used for compacting. Only Compacts No. 15 and 17 were made from the preliminary chips. The remainder were made from blended, degreased chips from Groups 1 or 2, or from AX57 or AX58 chips.

In Compact No. 15, the packing die was set up for cold compacting without the sleeve. After only a 30-ton load had been applied, however, the piston could not be removed upward from the die. After it was pushed out by force of the press, severe scoring of the packing die was noted. This was due to insufficient clearance (0.001") between the piston and packing die, and the fact that a hard material, probably small pieces from the bottom of the piston, had become lodged there. It was necessary to hone the packing die to its original diameter. Most of the further compacts were made with the 16-gage steel sleeve inside the packing die. A total of about three pounds of chips was used for each compact, added usually in 8 portions of about six ounces each. After the addition of each portion, a pressure of about 10 tons was applied; then, after complete loading, the pressure was gradually increased to the maximum capacity of the press, 260-290 tons, or about 60,000 psi. Because of the larger cross-sectional area, the maximum pressure used was less than that used in the small compacts. The density of the compact was correspondingly less, as measured for Compacts No. 17 and 18. The top of the compact was always more dense than the bottom because of the friction at the compact-sleeve surface. Because the upper and lower platforms of the press were not exactly parallel it was necessary to use extreme care in guiding the piston into the sleeve. If the piston became slightly cocked, it tended to compress one side of the soft steel sleeve as well as the chips. Shims were used on top of the piston to obtain the proper alignment. It was always necessary, in order to remove the compact from the sleeve, to cut the sleeve. Indentation marks from the chips were always present on the bottom part of the sleeve, and the compact had a correspondingly rough surface. This was not objectionable, however, since the outer diameter was to be later machined. The other alternative was to compact in the packing die without use of the steel sleeve. For Compact No. 23, made in this manner, the piston was ground to a diameter of 3.506", allowing 0.010" clearance between piston and packing die. Upon removal of the compact from the packing die, a flash of aluminum alloy was left behind, which required removal by dissolving with NaOH. It was felt that compacting within a sleeve, and later removing the sleeve, was preferable to the caustic treatment of the packing die after each compact.

G. Hot Compacting

Although some of the cold compacts were extruded without an intermediate hot compacting step (see next section), hot compacting was found desirable: 1) it provided a very dense billet for the bare extrusion; 2) it reduced oxidation of the chips, when done in an evacuated can.

Initially, several 2" billets of cold compacted chips were hot compacted directly in the extrusion press without canning. (See Table III, Compacts 9, 11, and 13.) In order to aid in preventing the graphite lubricant from penetrating into the pores of the compacts, aluminum foil was wrapped around them; they had previously been machined to a diameter of 1.975". Compact Nos. 11 and 13 were cylindrical, while Compact No. 9 had a 45° cone-shaped nose. A 2.0" diameter ram exerted pressure on the compact, upsetting it so that its diameter corresponded to that of the press liner, slightly above 2". The hot compacting operation, carried out at 900 F or 1100 F, reduced the billet length while increasing its diameter, increasing the density to nearer theoretical. After peeling off the aluminum foil, it was noticed that porosity was still evident in the hot compacted billet, due to the fact that gas entrapped between the chips was not allowed to entirely escape. Also, it appeared that some of the graphite lubricant had penetrated beneath the foil covering, with the possibility of contamination. In addition to these reasons, another factor necessitated the canning procedure, namely the oxidation of cold compacts when heated in an air atmosphere.

In order to check the oxidation tendencies during preheating at 900 F and 1100 F, samples of loose chips and compacts (sample weight = 90 - 100 grams) were weighed before and after 1-1/2 hour exposure in a furnace at these temperatures.

Sample	Original Weight, gm	Exposure Time, hr	% Weight Gain	
			900 F	1100 F
Compact of Chips	90.08	1.5	0.0008	0.096
Loose Chips	96.01	1.5	0.0189	0.106

The appreciable weight increase, especially at the 1100 F temperature, indicated possible difficulty from excessive oxide contamination when heating in air. Accordingly, further hot compacting was performed in an evacuated can.

The arrangement of the equipment used for the small scale hot compacting is shown in Figure 5. The cold compact was machined to fit within a can of ID = 1.875" having a 16-gage wall. The ends were welded onto the can, together with a 1/4" evacuation tube at one end. The can was evacuated overnight to 0.02 microns at 400 F before sealing off the tube. Because of the considerable surface area of the cold compact, a large amount of gas came off during the early stages of the evacuation procedure. The hot compacting was accomplished by the use of a dummy block in front of the ram and a penetrator over which the can slipped during compacting. The diameter of the penetrator was slightly greater than the ID of the can. During the application of the 225 ton load, the end of the can was sheared, slipping over the penetrator for about 3/8". The can was easily removed from the penetrator with a hammer, after which it was machined away from the billet. The surface of the compacted billet was very smooth and dense with no evidence of any porous areas. The removal of the air between the chips had allowed them to be well-bonded under pressure. Only the impression of the chips on the can surface could be seen on the outside of the compact. This procedure produced a hot compact far superior to that compressed in air.

The arrangement of the equipment used for the large scale hot compacting is shown in Figure 6, and a summary of the results is given in Table III. With the 3-5/8" liner in the 1000-ton extrusion press, the layout of dummy block, can, and penetrator were essentially the same as before. Initially, the wall thickness of the can used was 0.067" (16 gage) and the penetrator rings had an outer diameter of about 3.5". In Compact No. 18, during application of the load, part of the wall of the can near the penetrator fractured, allowing some of the U-Al alloy to be forced out over the penetrator. The can was removed from the billet by sawing and machining.

The billet itself was very dense, but because of loss of metal forced over the penetrator, weighed only 3/4 that of the original cold compact. In an effort to avoid the can fracture, the can wall thickness was increased to 0.104", and the outer diameters of both can and penetrator rings were increased to 3.590". This allowed a clearance of 0.035" (on a diameter) between the can and the liner, whereas it had previously been 0.125". It was thought that perhaps the excessive clearance between can and liner had played a part in the rupture of the can. In Compact No. 19 during the application of the load part of the can again fractured allowing the U-Al alloy to be forced over the penetrator. The compacted billet was again very dense. In Compact No. 20, the temperature of the can was lowered to 900 F, in hopes that the additional strength of the steel would prevent breakage. Again, however, much of the circumference of the can near the penetrator fractured. In Compact No. 21, the force was lowered to 600 tons and the can temperature to 800 F. In this case, the can did not fracture, the can end being sheared off, and the can itself being pushed slightly over the penetrator. After removal of the can from the compact, the compact was found to be quite dense; there was, however, a small area of the surface on which slight porosity was evident. In Compact No. 22, the force was increased to 800 tons, at 800 F temperature, with the result that the can again fractured. It was then decided to again increase the wall thickness of the can to 0.170", as shown in Figure 6. This, of course, reduced the diameter of the cold compact to be inserted into the can, requiring that more stock be machined from it, with the attendant loss of metal. With a can temperature of 850 F a force of 600 tons was sufficient to shear the can end and compact the chips, yet without causing rupture of the can (Compacts 23, 25, 26). A force of 700 tons was excessive, and the can again ruptured (Compact 24). At this magnitude of force, the U-Al alloy inside the can, acting as a liquid under pressure, exerts a terrific force on the inner walls of the can, causing the rupture. The hot compacts made at the 600-ton pressure level were essentially dense, but each had a very small area of very slight porosity. Since the adoption of a still heavier can would further reduce the diameter of the billet, it was felt that the use of the 0.170" wall can, with a 600-ton compacting force, was the best procedure for the hot compacting step.

After the hot compacting operation, the steel can must be removed from the billet. The normal procedure for this step has been to machine the steel sleeve from the billet, then pry or machine off the ends. The rather close bond formed between the steel and the billet often resulted in a small amount of the billet stock being machined off in order to completely remove the can. The U-Al turnings from this operation, being mixed with steel turnings, would be of no value for recycling purposes.

In summary of the cold and hot compacting steps of the procedure:

1) Cold compacting was most satisfactorily performed by compressing the chips within a steel sleeve held in the packing die. After compacting with the maximum pressure of the press, the sleeve plus compact were pressed from the packing die, and the sleeve removed by cutting a slit in it and prying it loose. The resulting compact was sufficiently dense to be machined to the diameter required for insertion into the can for hot compacting.

2) The machined compact was press fitted into a steel can, and the cover containing an evacuation tube was welded to the can. After evacuation overnight at 400 F to a small fraction of a micron pressure, the evacuation tube was sealed. Hot compacting was accomplished by forcing the can over a penetrator whose diameter corresponded with that of the ID of the can. In order to efficiently compact the chips, it was necessary to exert a force which would completely shear the can end so as to enable compaction and yet not exert so great a force as to fracture the can. The use of a greater can wall thickness assisted in the latter consideration. Nevertheless, the U-Al alloy, at a temperature of about 850 F acting as a liquid under pressure, exerted a great pressure against the walls of the can, and if the force applied was excessive, the can fractured. This was prevented only by limiting the force applied to less than that required to fracture the can.

3) Hot compacting in air did not yield a dense compact, since the gas trapped between the chips had no means of escaping. The use of an evacuated can for hot compacting the chips yielded a very dense billet. The latter procedure was also deemed necessary to minimize oxidation of the chips during heating at a high temperature.

H. Extrusion Experiments

A number of extrusions were made with 2" diameter billets in the 300-ton extrusion press; the liner diameter was 2.040". It was hoped that the results from the small-scale extrusions would be directly applicable to the large ones. The original plan was to use shear die extrusion through a flat die without the use of a lubricant. In commercial aluminum alloy extrusion practice, the ram of the extrusion press is fitted with a dummy block somewhat smaller than the cylinder (liner) diameter. As the ingot is extruded, this clearance between the dummy block and the cylinder leaves in the cylinder some of the original ingot surface. Furthermore, a small length of the billet is left unextruded (the butt), which is removed before charging another ingot. In the extrusion of the commercial alloys, no lubricant is used, except in cases where mandrels are used.

The arrangement of the tooling used for the small-scale extrusions is shown in Figure 7. In all cases, the extrusion die fit inside the liner, rather than in front of it. This necessitated the use of a backing plate immediately in front of the liner, against which the die was forced during the extrusion. The backing plate, of course, contained a hole in its center of sufficient size to allow the extrusion to pass with ample clearance. In the case of the shear die extrusions, the billet was placed directly back of the die. In streamlined flow extrusions, a steel cone was placed between the die and billet, to cause gradual transition of the plastic mass of metal from the liner size to the die size. In most cases, a scraper, of thickness 1/16" and diameter the same as the liner, was used between the billet and the ram. The scraper served the purpose of cleaning the liner, and also of preventing metal penetration between the ram and the liner. In some cases, a dummy block was used between the ram and the billet, rather than the scraper. This was simply a solid steel cylinder which fit within the liner.

Some of the earlier extrusions were made from billets of cold compacted chips, which were of course less dense than the hot compacts (see Table IV). Billets No. 6 and 8 were 2" diameter cold compacts of about 95% theoretical density, extruded through a shear die into 0.350" round at 900 F. The lubricant used on the die and liner was finely divided lead dispersed in oil. During the first extrusion, there occurred some build-up of metal on the surface of the die, which in turn caused scoring and roughness on the extrusion. This defect began near the nose of the extrusion, and became progressively worse toward its end. The second extrusion was much more satisfactory, with some roughness near the nose only, which did not progress any further. Billet No. 10 was a similar cold compact, extruded through a 0.250" round die under conditions of streamlined flow. Aluminum foil was wrapped around this billet previous to extrusion, to prevent penetration of the lead or oil into the porous mass. Slight defect from die build-up was evident on the first 1-1/2' of the extrusion; the remainder, about 9' long, had a good surface. Billet No. 9, hot compacted in an air medium, was extruded in a similar manner, with good surface condition being obtained. Billet No. 12 was another cold compact, this time extruded through a 0.250" shear die without a lubricant. A dummy block was used between the ram and the billet, with about 0.020" clearance on the diameter. This extrusion required nearly three times the ram pressure as the previous one, due partly to the shear die type of extrusion and partly to the lack of lubricant. Almost complete build-up of metal occurred on the die very early in the extrusion cycle, with the result that the extrusion was very rough along its entire length. During the cycle, some of the billet metal was back-extruded into the clearance space between dummy block and liner. A force of 215 tons was required to remove the dummy block and butt end from the liner, due to the fact that no lubricant was used. This procedure did not appear to be very promising. Billet No. 11, hot compacted in an air medium, was extruded into a 3/32" x 3/8" flat by means of a shear die, using lead in oil lubricant. Again the surface of the extrusion was quite good, with little roughness. This procedure was repeated in billet No. 16, except that no lubricant was used. As before, complete build-up of metal on the die surface occurred, and the surface of the extrusion was very rough. In order to compare the extrusion of this 15% U-Al alloy with 2S aluminum, a billet of the latter was extruded under exactly the same conditions, with no lubricant. In this case, the extrusion was very smooth, superior to any from the 15% alloy, and there was no metal build-up whatsoever on the die. This seemed to indicate that the presence of the 15% uranium in the billets caused the difficulty in extrusion.

With the exception of Billet No. 16, none of the 2" diameter billets had been hot compacted in an evacuated can. This procedure, as mentioned before, was found necessary to avoid oxidation of the chips, and to yield a dense compact for extrusion. It was noted that, upon fracturing a piece of Ext. No. 10321 (which had been hot compacted in air) longitudinally, the internal fiber structure was rather poorly bonded together, even though the outer surface of the extrusion appeared good. It was evident that hot compacting in air had not enabled a metallurgical bond to be formed between adjacent chips.

Because of the poor results obtained on the small billets from shear die extrusion without lubrication, it was decided to concentrate in the large-scale work on streamlined flow extrusion. It was felt that if the shear die technique with lubricant were used, there existed a possibility of contamination of the extrusion due to turbulent flow conditions in the vicinity of the die inlet.

The arrangement of the tooling used for the large-scale extrusions is shown in Figure 8; the diameter of the extrusion liner was 3.625" and the capacity of the press 1000 tons. In all cases, the extrusion die fit inside the liner, and a backing plate was used. A dummy block, of diameter slightly less than the liner, was used between the ram and the billet. All of the billets had been hot compacted in evacuated cans, so that the chips had been bonded together to form a dense structure. Billet No. 21 was extruded through a shear die into 1/8" x 1-1/16" flat, using no lubricant. As was true on the small size extrusions, complete build-up of metal on the die took place, and the extrusion had a very rough surface. This method was not attempted further.

All of the other extrusions made in the 3-5/8" liner were of the streamlined-flow type. Because of the difficulty in changing the shape configuration from a round to a flat by means of a steel cone, most of the tapered section was built into the die, as shown in Figure 9. Only the small part of the tapered section adjacent to the liner wall was formed in the steel cone. The first flat die had land dimensions 1" x 1/8", with the ends rounded. A later die was 1-1/8" x 1/8", the extra width needed for the subsequent operations. The conditions which produced the best quality extrusions were as follows:

1) Billet temperature - 1050 F. At this temperature, the billet plastically deformed with ease.

2) Die temperature - 600-700 F. Better lubrication from the lead was achieved with a heated die.

3) Liner Temperature - 700 F. This was sufficient to liquify the lead in the lubricant, enabling it to lubricate more efficiently.

4) Lubricant - Lead in oil, generously swabbed over the die, cone, and liner.

5) Cut-off - Graphite or magnesium alloy AZ31. Either was easily separated from the extrusion proper.

In general, the lower the billet preheat temperature, the more force was required for the extrusion. During the extrusion of Billets No. 23 and 24, the dies broke with cracks originating at the ends of the lands and proceeding outward.* In these cases, the dies were at room temperature when placed in the liner so there was more than the usual clearance between the dies and the liner. These dies were weakened by the incorporation of much of the tapered cone in the dies themselves, as shown in Figure 9. In the replacement die, the tapered cone within the die was designed to be much more shallow, providing extra strength. The die should be of such dimensions that when ready for extrusion it fits snugly within the liner. This allows the liner to absorb much of the force tending to force the die apart.

Because of limitations of time and funds, no attempts were made to account for all of the aluminum-uranium alloy during the extrusion operations until the last extrusion (ext. No. 12069, billet No. 25). Unfortunately,

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* The die breakage caused metal to build up on the rough fractured surfaces, and the extrusion surfaces were correspondingly poor.

in that extrusion a relay failure in the extrusion press by-pass system allowed the press to overshoot during the extrusion so that the die, cone, dummy block, ram, and part of the cut-off were hopelessly jammed in the liner. In extrusion No. 12069 the billet weighed 1172 grams, the extruded flat weighed 1153 grams, and it was possible to scrape 3 grams out of the die. The remaining 16 grams had to be stuck on the tools or in the liner, but the ease with which this 16 grams could be recovered was not determinable because of the accident described. In any future bare extrusions of aluminum-uranium alloy containing enriched uranium it will probably be necessary to set aside a liner and tools exclusively for the enriched work until the job is completed. That portion of the aluminum-uranium alloy that does not become part of the extruded flat would necessarily have to be accounted for in one or more of the following possibilities:

1) Stuck to the die and/or cone--most of this material would be recovered by mechanical prying and scraping, the remainder by dissolving in NaOH solution.
2) Stuck to dummy block--recover same as (1).
3) Stuck to liner--push scraper through after each extrusion to recover most of material. Small amount not recovered by scraping would be recovered upon completion of all extrusions by dissolving in NaOH solution. It would not be practical to clean the liner with NaOH solution after each extrusion, hence the need for setting a liner aside until all extrusions have been completed.

I. Rolling Procedure

For demonstrating the feasibility of the rolling operation, a small laboratory-type rolling mill was used. Power was supplied by a 2 hp, 1750 rpm motor, and transferred by means of a V-belt pulley to a gear reductor. The pulley arrangement between motor shaft and gear reductor was rigged so that the shaft from the reductor, which was directly connected to the rolls, turned at a speed of 28 rpm. The design of the rolls used is shown in Figure 10. A 2" wide groove was machined in the center of the upper roll shaft, of depth 0.014" initially, so that, when the two rolls were tight against one another the thickness of the sheet being rolled would approximate the required 0.015". It was found from rolling short pieces that the sheet thickness obtained was 0.0002" greater than the depth of the groove. Accordingly, the depth of the groove was increased to 0.0145", to obtain a desired sheet thickness within the 0.0147-0.0153" tolerance.

The lower roll shaft, directly driven by the gear reductor shaft, was also connected to the upper roll shaft by means of gears, so that both rotated at the same speed. In order to be able to coil the sheet being rolled, a take-up reel was designed and built, as shown schematically in Figure 11. Onto the bottom roll shaft a pulley was installed, which transmitted power by means of a flat belt drive to another pulley located at one end of the take-up reel shaft. The diameter of the take-up reel was such that, during the operation of the rolls, the linear speed of the take-up reel was greater than that of the rolls. Because of this, either the foils would be pulled apart, or the flat belt would slip to equalize the two speeds. The design was such that the latter occurred. Thus, the tension on the foil was equal to the force at which the flat belt slipped over the pulley. In practice, this force was sufficient to cause considerable tension in the foil, and to wind it tightly around the reel. The front of the piece to be wound around the reel was attached to a slot in the reel. In most cases, it was also necessary to insert shims in the slot to securely fasten the front of the foil.

Some preliminary rolling of short pieces of extruded 15% U-Al alloy had indicated that the edge cracking was more severe with greater reduction per pass. The adjustment of the spacing between the rolls was by means of a wheel which, through a gear arrangement, controlled the vertical movement of the upper roll shaft. One turn of this wheel was equal to approximately 0.016" reduction. Since it was desired to eliminate edge cracking as much as possible, the usual reduction per pass used was one-quarter turn of this wheel, or approximately 0.004". In the case of the 0.125" flat extrusion, about 28 passes were used to reduce it to 0.015" thickness. Using pieces of 1" x 1/8" flat from Extrusion 10494, the edge cracking measured about 3/64" on a side; thus, from a 1" width extrusion, 0.91" width of crack-free stock could be obtained in the foil. Using the same starting material, after rolling from 0.125" to 0.085" thickness, the piece was annealed for two hours at 900 F, then rolled to 0.015" thickness. The intermediate anneal considerably reduced the edge cracking, to about half the previous amount.

In order to be able to utilize as much of the U-Al foil as possible, a leader of 2S aluminum strip was usually welded on the front of the extrusion to be rolled. Since it was necessary to stop the mill in order to attach the nose into the reel slot, it was preferred that any defects caused by this operation occur in the leader rather than in the U-Al foil. It was found necessary to anneal the area of the weld to prevent rupture of the sheet at this point after only a few passes.

It was found necessary to install a guide on the entrance side of the rolls to prevent the sheet from wandering out of the groove in the rolls, in which case the sheet was smashed and rendered non-usable. Originally, a wooden guide was used, located several inches from the point of rolls contact. When it was found to be insufficient, a brass guide was constructed immediately in front of the point of entry of the sheet.

A summary of the rolling information for the long strips is given in Table V. Each strip was given 30 rolling passes total; three final passes with the rolls flush were required to reduce the foil to the desired 0.015" thickness. Since a foil width of 0.970" of good stock was desired, it was evident that with the edge cracking the 1" wide extrusion would not be sufficiently wide. Accordingly, extrusions of 1-1/8" width were adopted. During the rolling procedure, one section of Extrusion 12069 was subjected to two intermediate annealing cycles. The amount of edge cracking was reduced to a fraction of that obtained in a similar unannealed piece.

The main problem encountered in the rolling operation was the presence of camber in the rolled foil. The camber was usually on the order of 1/16" per foot length. Every possible adjustment of the equipment was made in an attempt to eliminate the camber, but none was entirely successful. Exchanging the positions of the upper roll bearings merely caused the camber to switch to the opposite direction. There was an indication that the presence of the flat belt pulley on the shaft increased the amount of camber. When the belt was completely removed, however, camber still was retained. The finest adjustment of the gear teeth controlling the vertical movement of the upper roll was made, but no improvement was noted. It was concluded that with the rolling equipment used the camber could not be altogether eliminated. The total camber allowed by the specifications was 0.005" in 24"; this specification could not be met in the rolling operation with the equipment used. However, it should be kept in mind that the rolling mill used in this work was merely a makeshift affair. With the proper type of rolling mill the camber should be much less than that experienced in this investigation.

The only loss of U-Al alloy incurred in the rolling operation was that occasioned by the welding of the 2S aluminum leader onto the extrusion and its subsequent removal after rolling. The loss of alloy due to flaking or peeling was nil.

J. Milling of Foil Edges

One of the rolls of the one inch width sheet was wrapped tightly around a steel core and held on the outside by a tight-fitting clamp. It was milled on the two edges to a width of 0.850", the edge cracking limiting the width obtainable. Upon laying out the sheet after milling, however, it was noted that some camber was retained in the strip; this method had simply removed the edge-cracked stock, but had not succeeded in eliminating the camber.

Eight lengths of sheet, about 24-1/2" long, were cut from the 1-1/8" wide foil which had the least amount of edge cracking (rolled from middle of Ext. 12069). These lengths of sheet were stacked one on top of another, held firmly between two flat steel bars, and milled to a width of 0.965". The desired width of 0.970" could not be obtained without some edge cracking on the piece because of the camber in the strip. The strip obtained from this operation met the straightness specification. The milling procedure caused a very slight burr on the edges of the foil.

K. Shearing the Foil

The 24-1/2" lengths of foils were cut into two foot pieces by shearing. The pieces of foil were laid out exactly perpendicular to the edge of the shear. A steel scale exactly two feet long, was used as a pattern. The eight lengths from Ext. 12069 were sheared to ± 0.010" length tolerance.

V. Results and Discussion

A. Chemical Analyses

In Table VI are listed chemical analyses of a number of the 1" x 1/8" flat extrusions. The samples for analysis were sheared transversely across the extrusions, usually from the front, middle, and rear sections. In two cases, samples for analysis were taken from 0.015" sheet rolled from pieces of the extrusion.

The original melts from which the chips were made, AX57 and AX58, were nominally 14.24 and 15.15 w/o uranium. Analyses of samples from extrusions made from chips from these individual melts (see Table VI) agreed quite closely with these nominal values.

Most of the analyses shown are from extrusions made from compacts of Group 1 or Group 2 chips. Since each of these were blends of equal amounts of AX57 and AX58 chips, it would be expected that their composition would be the average of AX57 and AX58, or 14.70%. In samples from four of the six extrusions, very good agreement was obtained. In Extrusion 10575, two samples taken near the front were 0.29 and 0.47 w/o above nominal, while middle and rear samples were less than 0.10% above it. In Extrusion 11930, the sample analyzed was 0.24% above nominal.

The homogeneity desired in this work in any one sample was 1-1/2% (relative) of the average of the uranium content in all samples: i.e., ± 0.21 w/o uranium. Excepting the front part of Extrusion 10575, all the samples (from Groups 1 and 2) fell within the range of 14.53 - 14.94% or ± 0.20% uranium.

B. Yields and Losses

In thinking in terms of a possible enriched job of this nature, it is important to consider the metal losses in various stages of the operation. This is important especially for the purpose of obtaining the over-all efficiency of recovery of metal. In our case, from such information one can calculate the amount of metal that must be melted to eventually produce a certain number of foils. Metal losses may be subdivided into two parts: 1) Usable; 2) Non-usable. The first refers to metal which, although lost from the main sequence of operations, can be returned at some other point as scrap. The second refers to metal which, because of admixture with other materials, cannot be reprocessed. An example of the first type of loss is that of clean lathe turnings of the U-Al alloy; these can be degreased and returned as chips for reprocessing. An example of the second type of loss is that of lathe turnings obtained from machining the steel can away from the billet. Some admixture of steel turnings with U-Al turnings cannot be avoided. This type of metal, because of contamination, cannot be returned to the operation.

In Table VII are shown examples of typical metal losses in various steps of the sequence of operations. The weights and losses given are for individual steps of the operation, and do not necessarily represent a continuous sequence from one particular starting batch of material. The largest losses of metal resulted from the hot compacting, extrusion cropping, and edge milling operations. In removing the hot compact from the can by machining, the tight bond between can and billet causes loss of some of the billet, in order to completely remove the can. The resulting U-Al chips are mixed with steel chips, and cannot be recycled. The croppings of the extrusion are unavoidable, because of the nose and tail defects. If not contaminated by the cut-off material these croppings can be reprocessed. The losses from milling the edges of the foil are also unavoidable, although they could be reduced if camber and edge cracking were completely eliminated. These millings are too finely divided to be utilized for recycling. Most of the metal losses from the other operations are very small with some of the metal being in such a form that it could be recycled. For instance, the scraps from shearing could be readily compacted after cleaning.

From the standpoint of the subsequent use of enriched uranium in these operations the accountability for metal requires considerable care in some of the steps. Part of this is due to the fact that the U-Al alloy is for the large part processed in the bare condition; i.e., it has no cladding such as is commonly used with enriched alloys. This, of course, means good control and housekeeping from both the accountability and health standpoints. The operations which require especial care from the accountability standpoint are as follows:

1) Machining castings into chips - rather elaborate methods must be

used to recover every chip; probably a completely separate room or area is necessary.

2) Blending - the large number of blending operations, requiring handling of batches of chips several times, mean careful control.
3) Extrusion - in this step, since a bare billet is extruded, U-Al alloy may be retained on die, liner, and dummy. The procedures to be used to recover all uranium have been described in the section on "Extrusion Experiments".

In this work, no attempt was made to obtain complete accurate input and output weights to each operation for accountability purposes. This would have required more elaborate equipment than was used and many more precautions would have been necessary in order to obtain the best results. Inasmuch as the purpose of this work was to develop a method, rather than to arrive at the ultimate refinement of each step, complete information of the type required for a feasibility report has not been obtained.

C. General Discussion of Method and Conclusions.

In addition to the accountability problems discussed above for purposes of work with enriched material, there are also the health and safety factors which must be considered. These considerations would make necessary a number of precautions, which are briefly described for the various steps as follows:

1) Melting - all parts of furnace must be weighed before and after melting.
2) Machining of Chips - need good ventilation of area; must collect fines in flue.

3) Degreasing of Chips - adequate ventilation in room necessary.
4) Blending of Chips - can use only small, tight containers; good ventilation in room required.
5) Storage of Chips - limit of 1000 grams U235 contained in chips in any one container.
6) Cold Compacting - adequate ventilation in press area, since chips have fines incorporated in them. (Take air samples.)
7) Machining Compact - ventilated lathe area.
8) Hot Compacting - must not rupture can.
9) Machining can - ventilated lathe area.
10) Preheating bare enriched billet. Heat in graphite container and cover billet with aluminum foil. (Take air samples.)
11) Extrusion of bare billet - adequate ventilation of area; use 6" diameter pipe to catch extrusion when it emerges from press. (Take air samples.)
12) Rolling of Strip - (Take air samples.)
13) Milling edges of foil - ventilate area of milling machine.

A general summary of the procedure recommended for the production of uranium-aluminum foils by the chip method is presented in Appendix A. The summary refers to the equipment used in general terms only, and does not specify particular sizes of billets, tooling, etc. This summary is not intended to be a final refinement for each of the steps, but it represents the best procedure used in the experimental work.

The strips of aluminum-uranium foil produced in this work, 0.015 x 0.965" averaged 17.3 grams in weight. Assuming their composition to be the same as that of the melt from which they were produced, 15.15% uranium, a simple calculation gives a uranium concentration of 0.1131 grams per in.^{2} of surface. A similar calculation, using the theoretical density instead, gives a uranium concentration of 0.1149 grams per in.^{2} of foil surface. These figures are within 1.5% of each other, which is very satisfactory, considering that the thickness tolerance alone allows a variation of ± 2%. The over-all tolerance, counting both dimensional and composition factors, is ± 5%, based upon the grams of U235/in.^{2} of foil. It is evident that the strips are well within specifications.

The development work described in this report has demonstrated the chip method to be feasible for the 15% uranium-alloy, as an alternative to the regular casting method. Under normal conditions, it is difficult to obtain homogeneous castings in this alloy. The homogeneity obtained by the use of the chip method was ± 0.3% or better in all sampling performed and normally was within ± 0.2%. The steps used in the procedure, briefly, were:

1) Melt and cast in vacuum.
2) Machine casting entirely into chips.
3) Degrease chips.
4) Blend chips.
5) Cold compact.
6) Evacuate and hot compact.
7) Extrude into flat.
8) Cold roll to finish thickness.
9) Mill to desired width.
10) Shear to 2" lengths.

The strips of foil produced by this method conformed to specifications, both for dimensions and composition. The main difficulty encountered was in the rolling procedure, in that camber could not be entirely eliminated in the foil. Accordingly, it was necessary to mill the foil width in such a manner as to remove the effect of camber.

APPENDIX A

*Recommended Procedure for Production of Uranium-Aluminum Foils by Chip Method

1. Melt and cast, in vacuum, melts weighing 15.6 lb (7082 gm), each containing 14.13% total uranium (the contained total uranium content is 1000 grams; the U235 content is slightly less for highly enriched material).
   - a. The melts should be kept liquid long enough to insure complete solubility of the uranium.
   - b. If the castings are the shape of hollow cylinders, they will be more readily machined and also more homogeneous than solid cylinders.
   - c. The total number of melts required depends upon the over-all recovery of metal. An over-all efficiency of 73% requires about 21 melts in order to have sufficient metal to produce 6400 foils.
2. Reduce each of the 21 castings to chips using a chip breaker in the lathe, and making certain that the lathe guarding does not allow the loss of any chips.
   - a. Use no cutting fluid during this operation.
   - b. Machine chips as small as practicable, considering time necessary to do the machining.
   - c. When casting is so small that it cannot be held in the lathe, use a milling machine to reduce into chips.
   - d. Separate chips from each casting and store in separate containers.

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* The actual number of melts and batches of chips may be considerably less than 21 if suitable poison cans are designed by BNL. See footnote p.9.

3. Degrease each batch of chips with trichlorethylene or tetrachlorethylene followed by an acetone dip.
4. Using a completely tight tumbling apparatus blend separately the chips from each melt.
5. Divide the blended chips from each casting into 21 portions. Cross-blend by using one portion from each of the 21 castings and blend in the tumbling apparatus.
   a. A total of 42 blending operations is necessary.
   b. 21 batches of chips each weighing 15.6 lbs, and each of uniform composition are the result of this procedure.
6. Cold compact chips into compacts of size convenient for later hot compacting in press.
   a. A suitable method is to compact chips within a steel sleeve which fits into a packing die. Compact is removed by sawing or machining sleeve.
   b. Use very high pressure in order to obtain a dense compact which can be machined.
7. Machine diameter of cold compact to fit into steel can. Insert into can, after which weld cover and evacuation plug onto top of can. Evacuate can to a low pressure while heating to at least 400 F. Seal off evacuation tube.
8. Preheat canned billet to 850 F. Hot compact the chips by forcing the can over a penetrator, the diameter of which corresponds to the ID of the can.
   a. Conditions for hot compacting are somewhat critical, since force

must shear end of can and effect compacting, yet not break the can.

b. At a compacting temperature of 850 F, a pressure of 7.3 tons/in.^{2} of compact area was satisfactory, the can wall thickness being 0.170".

c. A heavier wall can or a lower compacting temperature than that mentioned above would allow a greater compacting pressure before fracture of the can.

9. Machine the can from the compacted billet, making certain that no pieces of the can remain embedded in the U-Al compact.

10. At a temperature of 1050 F, extrude the hot compact into 1/8" x 1-1/8" flat, using streamlined flow conditions and a lead-in-oil lubricant for liner and die.

a. In order to achieve smooth transition from the round billet to the flat extrusion, the cone may be built into the die, taking care that this does not excessively weaken the die.

b. Graphite is satisfactory for use as a cut-off.

11. The nose and tail of the extrusion are next cropped. The nose defect is very small, but the tail is usually defective for a foot of length.

12. Weld a 2S aluminum leader onto the extrusion to assist in the rolling operation. This weld should be annealed to prevent cracking during the early stages of rolling.

13. Cold roll the extrusion from 0.125" to 0.015" thickness, using a number of passes.

a. In general, the larger number of passes used the less edge cracking will be encountered.

   b. If the entire strip is annealed two or more times during the cold rolling process the edge cracking will be considerably reduced.

14. If there is no camber in the rolled strip, the entire coil can be milled to the required 0.970" width. This would be performed after tightly coiling the strip around a center core.

   a. If there is camber in the as-rolled material, it is necessary to mill the approx. two foot lengths after first clamping between flat steel bars.

   b. With the proper equipment, it might be possible to slit the sheet to the required width during the final rolling operation. This would eliminate the milling procedure.

15. The foils are sheared to the required 24.00" lengths.

REFERENCES

(1) J.L. Klein, NMI, Letter to A. Oltmann, Mech. Eng. Section, BNL, Dec. 9, 1955.

(2) J. Cunningham, ORNL, Personal Communication.

(3) L.R. Aronin and J.L. Klein, "Use of a Density (Specific Volume) Method as a Sensitive Absolute Measure of Composition, and Its Application to the Aluminum-Uranium System," AEC Report, NMI-1118, 1954.

Table I. Melting and casting conditions for chip stock.

Melt No.	AX57	AX58	AX68
Date Melted	5/16/56	5/17/56	11/16/56
Charge			
% Uranium	14.24	15.15	14.13
Wt. U, Kilos	3.481	3.694	1.0021
Wt. Al, Kilos	20.970	20.680	6.0918
Wt. Total, Kilos	24.451	24.374	7.0939
Vacuum, microns	200-400	150-500	15-25
Total Time Power On	66 min.	58 min.	38 min.
Total Time All Molten	43 min.	35 min.	18 min.
Pouring Temp.	1135 C	1220 C	1200 C
ID of Mold	8"	8"	5.5"
Casting Height, avg.	10-5/8"	10-3/8"	6-3/8"
Casting Wt., Kilos	24.403	24.358	7.0736
Loss, Gms.	48.2	16.0	20.3
% Loss	0.19%	0.066%	0.286%
Calculated Over-all Ingot Composition			
(1) If loss assumed all Al	14.26	15.16	14.18
(2) If loss assumed all U	14.07	15.10	13.88

Table II. Results of cold compacting.

No.	Sleeve thickness	Die or sleeve ID	Chips used	Loading method	Wt. of chips, g	Compact dia. in.	Force applied tons	1000 psi	Approx. density g/cm^3	% of theor. density	Surface appearance	Remarks
1	.037"	1.927"	Prelim.	9 increments	435.0	1.94	50 ea. increment	33.8	2.76	90	Loosely packed	9 individual parts; oil exuded.
2	.037	1.927	Prelim.	one portion	71.9	1.943	145	98.0	3.05	99	Dense; walls smooth	Unable to lift piston after 145 ton load applied, due to flash around piston; oil exuded.
3	.037	1.927	Prelim.	one portion	290.7	1.934	150	102.	3.03	99	Indentations from pressing into sleeve; Dense	Oil exuded
4	None	2.000	Prelim.	one portion	387.2	2.004	150	95.5	3.05	99	Smooth but slight scoring	Oil exuded.
5	None	2.000	Degreased Prelim.	one portion	129.1	2.005	150	95.5	3.05	99	Smooth but scored	Vapor degreasing eliminated oil contamination.
6	None	2.000	Degreased Prelim.	4 increments	382.6	2.005	150	95.5	2.93	95.5	Smooth but scored	Much more dense at top than bottom of compact.
7	None	2.000	Degreased Prelim.	one portion	119.0	2.005	70	44.5			Smooth but scored	Unable to lift piston after 70 ton load applied, due to flash.
8	None	2.000	Degreased Prelim.	6 increments	261.0	2.005	175	111.			Smooth but scored	
9	None	2.000	Degreased Prelim.	5 increments	370	2.005	150	95.5			Smooth but scored	Nose cone used, loosely packed in this area.
10	None	2.000	Degreased Prelim.	5 increments	355	2.005	175	111.			Smooth but scored	Nose cone used, loosely packed in this area.
11	None	2.000	Degreased Prelim.	Several increments	317.5	2.005	150	95.5			Scored	
12	None	2.000	Degreased Prelim.	Several increments	311.5	2.005	170	107.			Scored	
13	None	2.000	Degreased Prelim.	Several increments	314.9	2.005	150	95.5			Walls badly scored	
14	.037	1.927	Degreased Prelim.	2 increments	113	1.94	150	101.5			Indentations from pressing into sleeve	
15	None	3.515	Degreased Prelim.	one portion	113	3.515	30	6.00			Very loosely packed	Could not lift piston after 30 ton load applied, the 0.001" clearance between piston and die was insufficient.
16	.037	1.927	Degreased Prelim.	6 increments	341.1	1.925	175	120.			Indentations from pressing into sleeve	
17	.067	3.372	Degreased Prelim.	6 increments	1238	3.370	258	57.8	2.75	90	Indentations from pressing into sleeve	Piston did not enter sleeve exactly true, compressed can on one side.
18	.067	3.372	Group 1	8 increments	1372	3.370	262	58.5	2.85	93	Indentations from pressing into sleeve	
19	.067	3.372	Group 2	8 increments	1343	3.375	266	58.7			Indentations	
20	.067	3.372	Group 1	8 increments	1387.5	3.375	266	58.7			Indentations	
21	.067	3.372	Group 2	8 increments	1377	3.375	266	58.7			Indentations	
22	.067	3.372	Group 1	8 increments	1353	3.375	290	64.9			Indentations	
23	None	3.516	Group 2	8 increments	1330	3.51	270	55.8			Scored wall	
24	.067	3.372	AX57	8 increments	1804	3.375	270	60.4			Indentations	
25	.067	3.372	AX58	8 increments	1376	3.375	270	60.4			Indentations	
26	.067	3.372	Group 1	8 increments	1402	3.375	270	60.4			Indentations	

Table III. Results of hot compacting.

No.	Can wall thickness in.	Can ID in.	Method	Temp.F	Compact dia.in.	wt.g	Force applied tons	1000 psi	Results
9	Al Foil	None	Air	900	2.035	368.3	225	138	Voids not closed
11	Al Foil	None	Air	1100	2.040	320.5	225	138	Voids not closed
13	Al Foil	None	Air	1100	2.024	316.3	225	138	Voids not closed
16	.067	1.866	Evac. can	1050	1.866	325.3	225	165	Dense compact
18	.067	3.370	Evac. can	1050	3.370	1366	1000	224	Can ruptured, Al over mandrel
19	.104	3.375	Evac. can	1050	3.375	1341	700	157	Can ruptured, dense compact
20	.104	3.375	Evac. can	900	3.375	1384	700	157	Can ruptured, dense compact
21	.104	3.375	Evac. can	800	3.375	1372	600	134	Quite dense
22	.104	3.375	Evac. can	800	3.375	1346	800	179	Can ruptured
23	.170	3.250	Evac. can	850	3.25	1104	600	145	Quite dense
24	.170	3.250	Evac. can	850	3.25	1731	700	169	Can ruptured
25	.170	3.250	Evac. can	850	3.25	1257	600	145	Quite dense
26	.170	3.250	Evac. can	850	3.25	1273	600	145	Quite dense

Table IV. Extrusion conditions and results.

Ext. no.	Billet or compact no.	Type compact	Die			Liner		Billet				Lubricant	Ram speed in./min.	Cut-off	Ext. ratio	Max. force tons	K tons/in.^{2}	Die condition	Surface appearance
			Type	Size, in.	Temp. F	Dia. in.	Temp. F	Dia. in.	Wgt. g	Length in.	Temp. F								
10232	6	Cold	Shear	0.350	R.T.	2.040	700	2.005	382.6	2.52	900	Lead	15	None	32.6	100	8.78	Build-up	Rough, from build-up
10233	8	Cold	Shear	0.350	R.T.	2.040	700	2.005	261	1-21/32	900	Lead	15	None	32.6	105	9.22	Slight build-up	Rough near nose
10268	10	Cold	Streamlined	0.250	R.T.	2.040	700	1.975	353.2	2-1/2	900	Lead	15	None	66.6	85	6.19	Good	Good
10289	9	Hot	Streamlined	0.250	R.T.	2.040	700	1.979	350.5	2-1/2	1075	Lead	15	None	66.6	105	7.64	Good	Good
10290	12	Cold	Shear	0.250	R.T.	2.040	700	1.938	311.5	2-1/4	1075	None	15	None	66.6	280	20.4	Complete build-up	Very rough
10321	11	Hot	Shear	3/32 x 3/8	R.T.	2.040	700	2.00	318.1	2	1075	Lead	15	None	93	120	8.1	Good	Good
10448	16	Hot	Shear	3/32 x 3/8	R.T.	2.040	700	1.875	323.5	2-3/8	1050	None	15	None	93	240	16.2	Complete build-up	Very rough
10463	--	2S-Al	Shear	3/32 x 3/8	R.T.	2.040	900	2.00	300	2-1/4	1050	None	15	None	93	115	7.76	Good	Good
10494	18	Hot	Streamlined	1/8 x 1	R.T.	3.625	700	3.37	986	2-1/8	1050	Lead	13	Graphite	86	270	5.83	Slight build-up	Slight roughness
10574	19	Hot	Streamlined	1/8 x 1	600	3.625	760	3.37	1199	2-3/4	1050	Lead	70	Graphite	86	320	6.91	Good	Good
10575	20	Hot	Streamlined	1/8 x 1	400	3.625	780	3.37	1069.5	2-1/2	1050	Lead	13	Graphite	86	340	7.34	Slight build-up	Very slight roughness
10741	21	Hot	Shear	1/8 x 1-1/16	R.T.	3.625	700	3.37	1370	3	1050	None	13	None	77.7	400	8.92	Complete build-up	Very rough
10742	22	Hot	Streamlined	1/8 x 1	600	3.625	700	3.37	1220	2-3/4	900	Lead	13	Graphite	86	390	8.4	Slight build-up	Slight roughness
11930	23	Hot	Streamlined	1/8 x 1-1/8	R.T.	3.625	760	3.25	1034	3	850	Lead	13	AZ31	73.5	410	9.25	Broke, causing build-up	Rough
11931	24	Hot	Streamlined	1/8 x 1	R.T.	3.625	760	3.25	1590	4-1/4	850	Lead	13	AZ31	86	450	9.81	Broke, causing build-up	Rough
12069	25	Hot	Streamlined	1/8 x 1-1/8	700	3.625	700	3.25	1172	3-3/4	1050	Lead	13	Graphite	73.5	230	5.19	Good	Good

Table V. Rolling of long strips

Extrusion No.	10494	10575	10575	12069	12069
Location	Front	Middle	Rear	Front	Middle
Orig. Width, In.	1.0	1.0	1.0	1.125	1.125
Orig. Thickness, In.	.127	.127	.127	.126	.126
Orig. Length, Ft.	4	4	2-1/3	4	4
No. of Passes	30	30	30	30	30
Final Length, Ft.	33	33	19	33	33
Final Thickness, In.	.015	.0155	.015	.015	.015
Edge Cracking/side	.045	.045	.045	.045	.015
Width of Good Stock, In.	0.91	0.91	0.91	1.03	1.11
Camber	Some	Some	Some	Some	Some
Anneals	None	None	None	None	.086" .044"
Remarks		Became caught in guide in last pass.			

Table VI. Chemical analyses

Extrusion number	Chips from	Sample location	Type sample	Iron ppm	% U	Avg. % U for ext.	*Variation from nominal
10494	Group 1	Front	Ext.	63	14.67		- 0.03
		Front	Sheet	73	14.70		0.00
		Front	Sheet	67	14.54		- 0.16
		Middle	Ext.	88	14.53		- 0.17
		Rear	Ext.	122	14.79	14.65	+ 0.09
10575	Group 1	Front	Ext.	125	14.99		+ 0.29
		Front (Dupl.)	Ext.	81	15.17		+ 0.47
		Middle	Ext.	323	14.73		+ 0.03
		Rear	Ext.	1060	14.79	14.92	+ 0.09
10742	Group 1	Middle	Ext.	45	14.66	14.66	- 0.04
10574	Group 2	Front	Ext.		14.72		+ 0.02
		Front (Dupl.)	Ext.		14.64		- 0.06
		Middle	Ext.	111	14.78		+ 0.08
		Rear	Ext.	1228	14.67	14.69	- 0.03
10741	Group 2	Middle	Ext.	43	14.83	14.83	+ 0.13
11930	Group 2	Middle	Ext.	46	14.94	14.94	+ 0.24
11931	AX57	Front	Ext.	53	14.37		+ 0.13
		Middle	Ext.	63	14.32		+ 0.08
		Rear	Ext.	95	14.29	14.33	+ 0.05
12069	AX58	Front	Ext.	159	15.07		-.0.08
		Rear	Ext.	152	15.19	15.13	+ 0.04

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* For Group I and Group II chips, nominal is 14.70 w/o U, the average of the two melts used; for AX57 and AX58, nominal is taken as the composition calculated from the charge weights, 14.24 w/o and 15.15 w/o, respectively.

Table VII. Typical metal losses in various steps of operation

Operation	Original Weight, g	Final Weight, g	Loss g	% Loss	% Recovery	Type of Loss
Melting (in vacuum)	24451	24403	48	0.19	99.81	Oxide and Skull
Degreasing & mixing chips	9744	9740	4	0.04	99.96	Fines
Cold compacting	1343	1343	0	0	100	----------
Machining to fit in can	1377	1372	5	0.36	99.64	Lathe turnings
Hot Compacting	1257	1172	85	6.7	93.3	Lathe turnings (mixed with steel)
Extrusion	1172	1156	16	1.36	98.64	Coating in Liner & Die
Cropping Extrusion	1156	1098	58	5.0	95.0	Croppings
Rolling	302	302	0	0	100	
Milling Edges	164	142	22	13.4	86.6	Fine Millings
Shearing	142	139	3	2.1	97.9	Shearing Scraps

Total % recovery = .9981x.9996x1.0x.9964x.933x.9864x.950x1.0x.866x.979 = 73.4

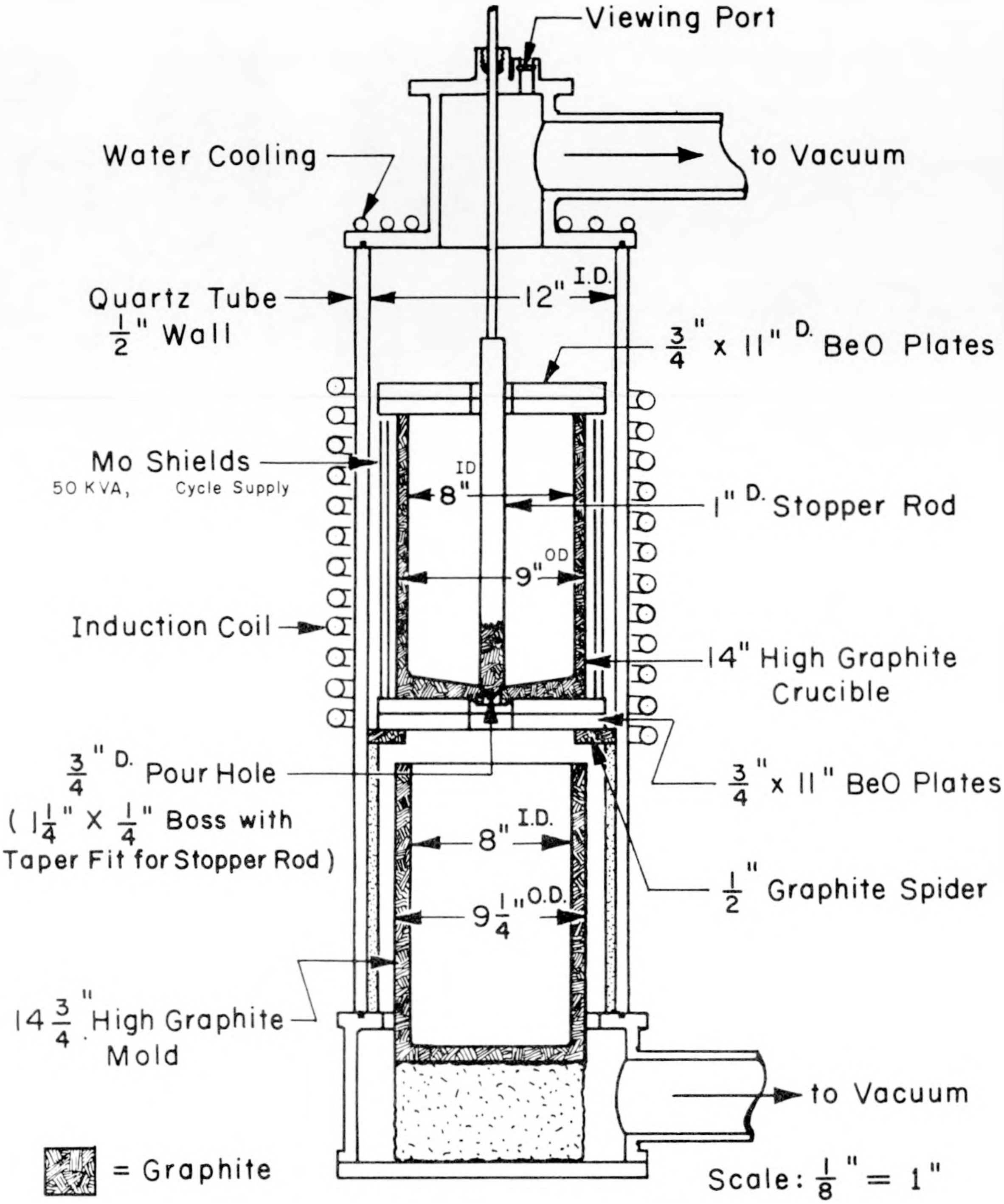


Fig. 1. Equipment Used for Melting U – Al Alloys

15324

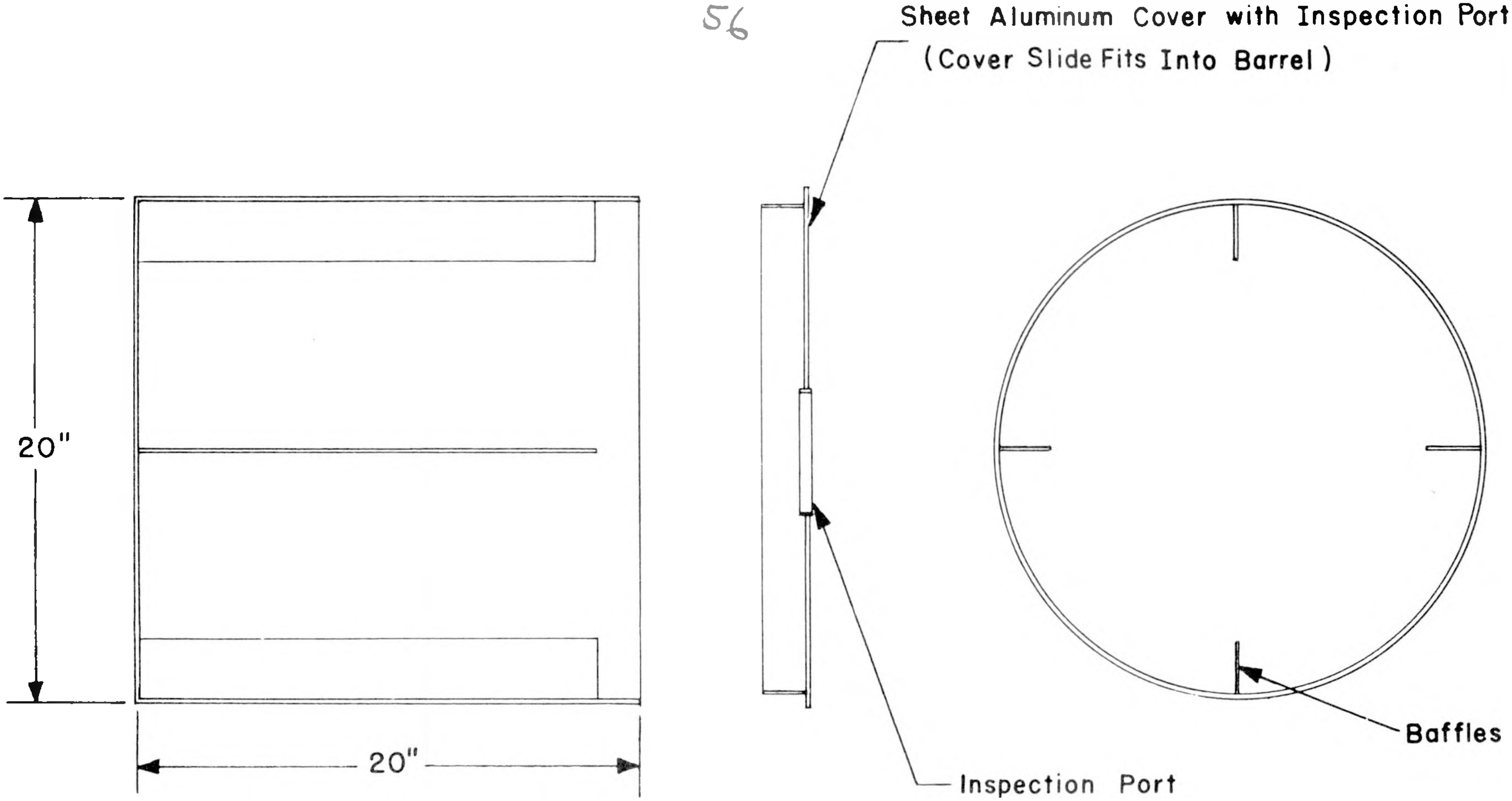
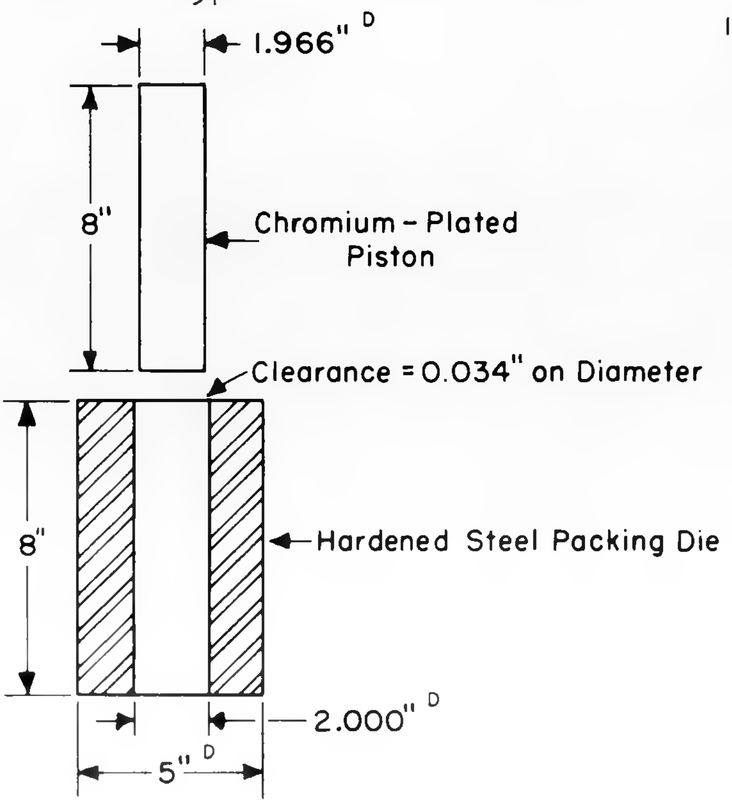
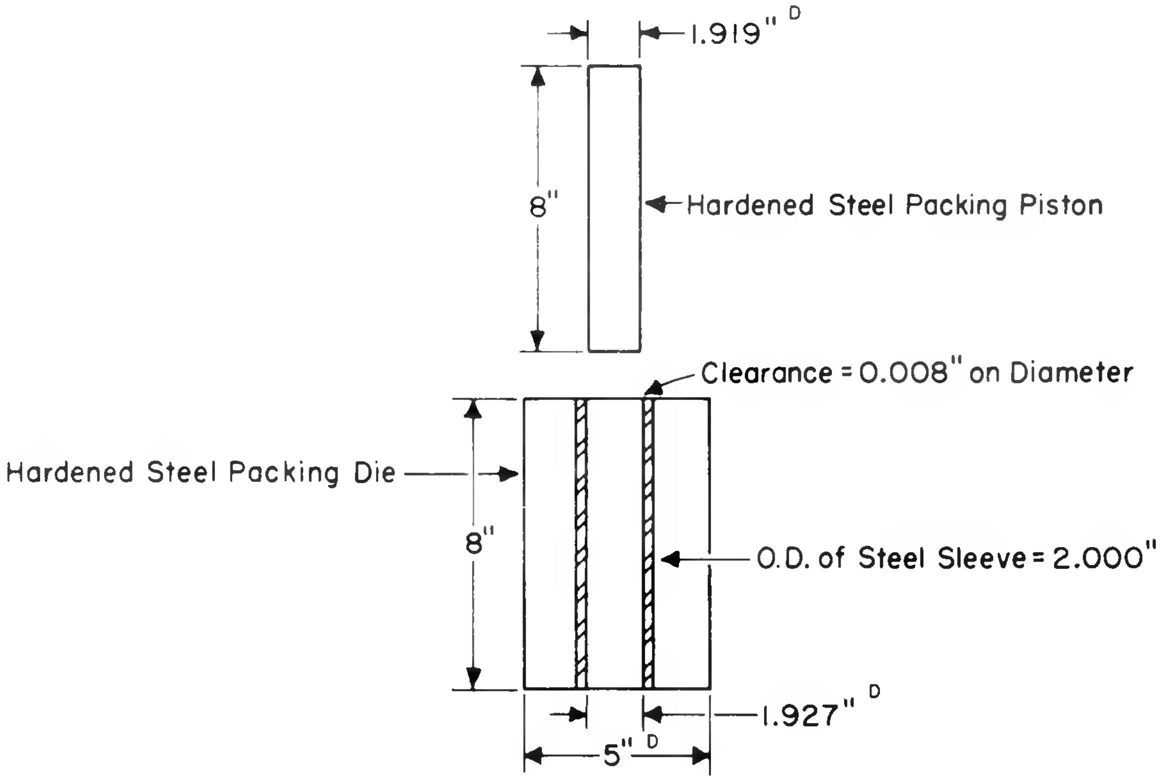


Fig. 2. 2S Aluminum Tumbling Barrel Used For Blending Chips

15326

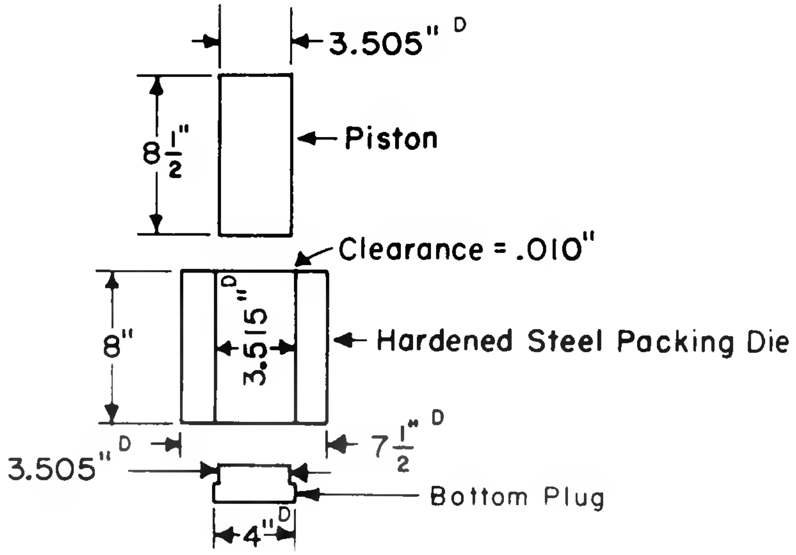


(a) Compacting in Packing Die

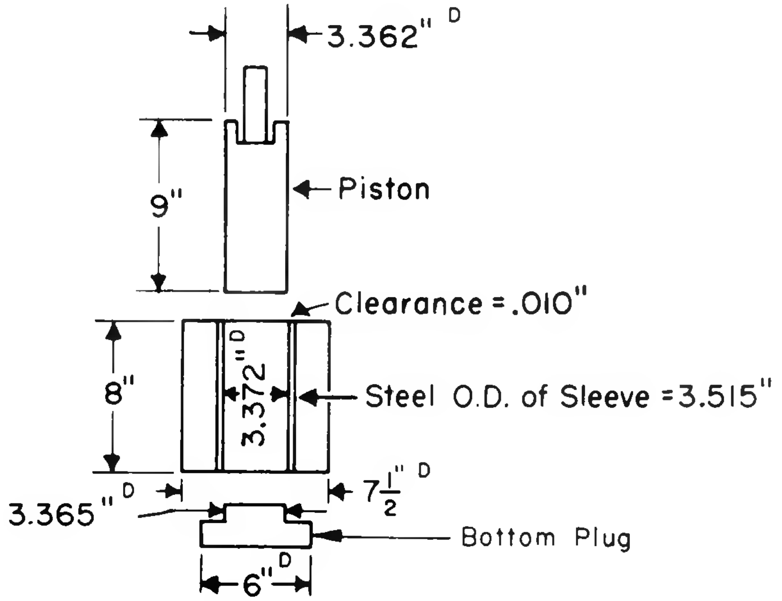


(b) Compacting in Sleeve within Packing Die

Fig. 3 Equipment for Small Scale Cold Compacting



(a) Compacting in Packing Die



(b) Compacting in Sleeve within Packing Die

Fig. 4 Equipment for Large Scale Cold Compacting

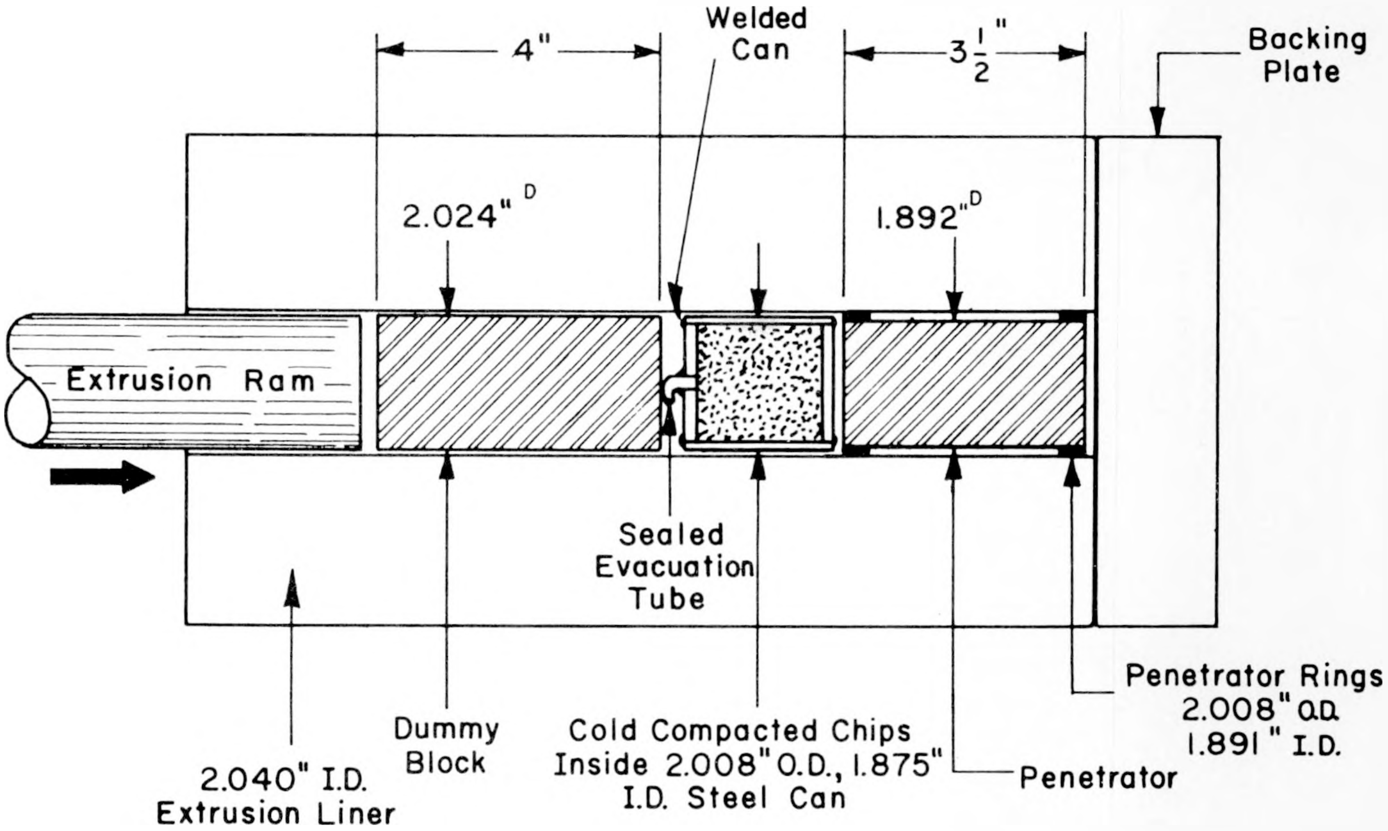
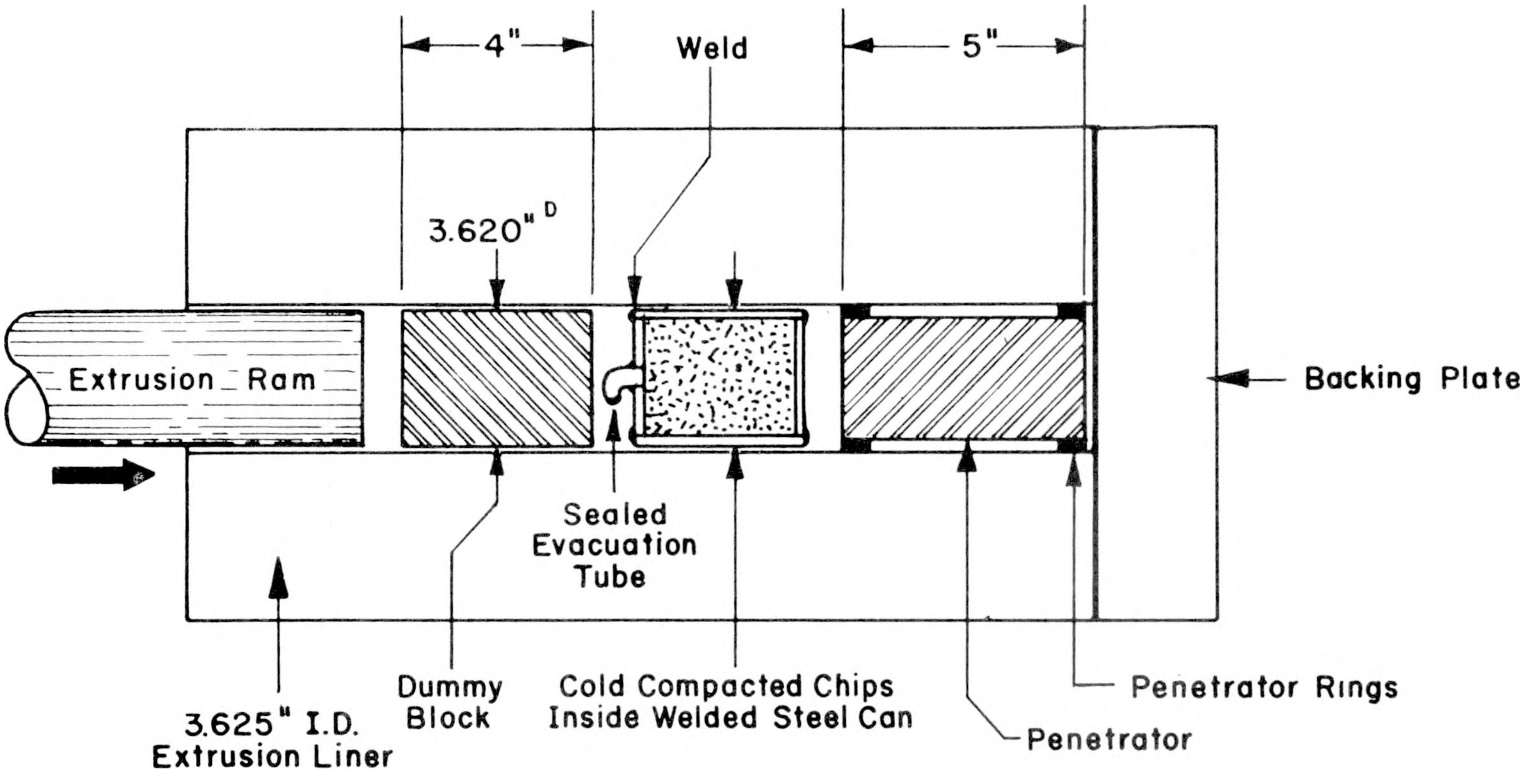


Fig. 5. Equipment for Small Scale Hot Compacting



Dimensions

Can Wall Thickness	Can		Penetrator Diameter	Penetrator Rings	
	O.D.	I.D.		O.D.	I.D.
.068"	3.510	3.375	3.382	3.510	3.381
.107"	3.590	3.375	3.380	3.590	3.379
.170"	3.590	3.250	3.246	3.590	3.247

Fig. 6. Equipment for Large Scale Hot Compacting

15320

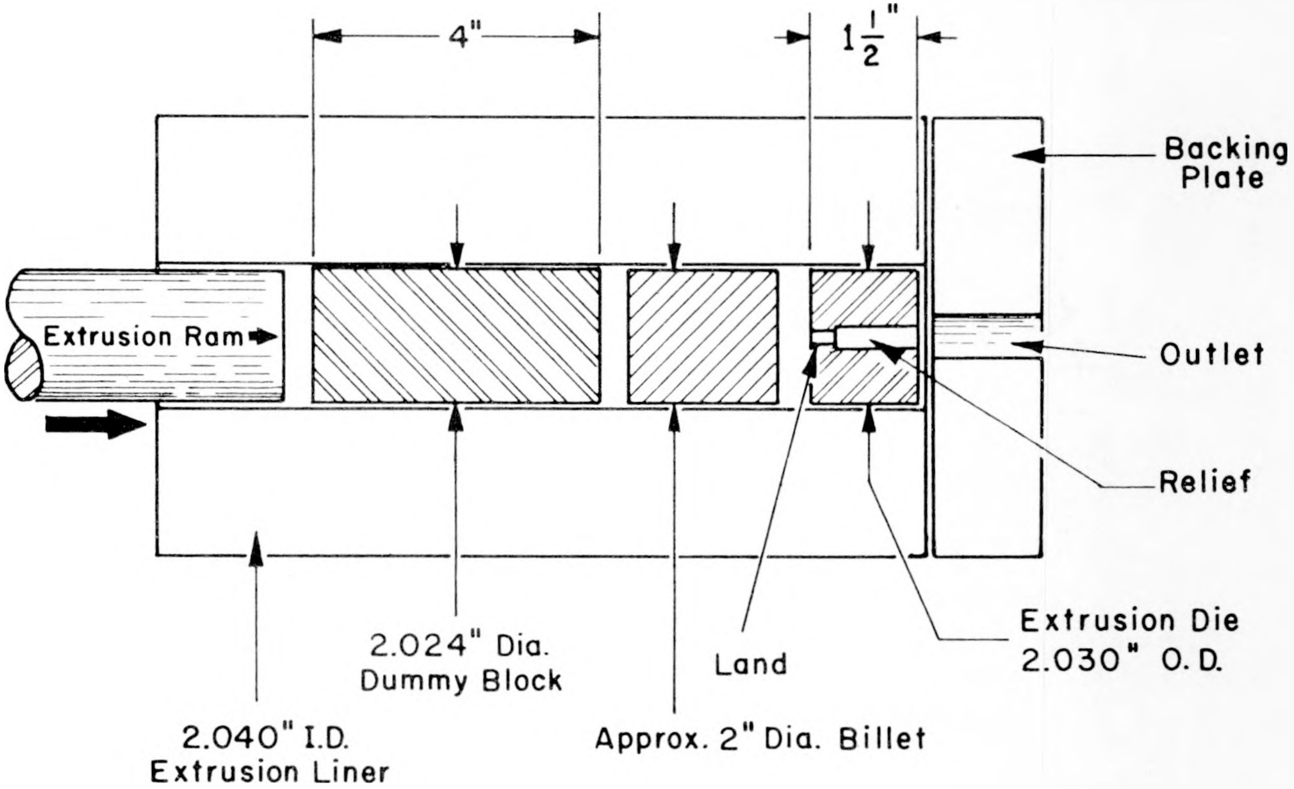


Fig. 7. Equipment for Small Scale Extrusions

15325

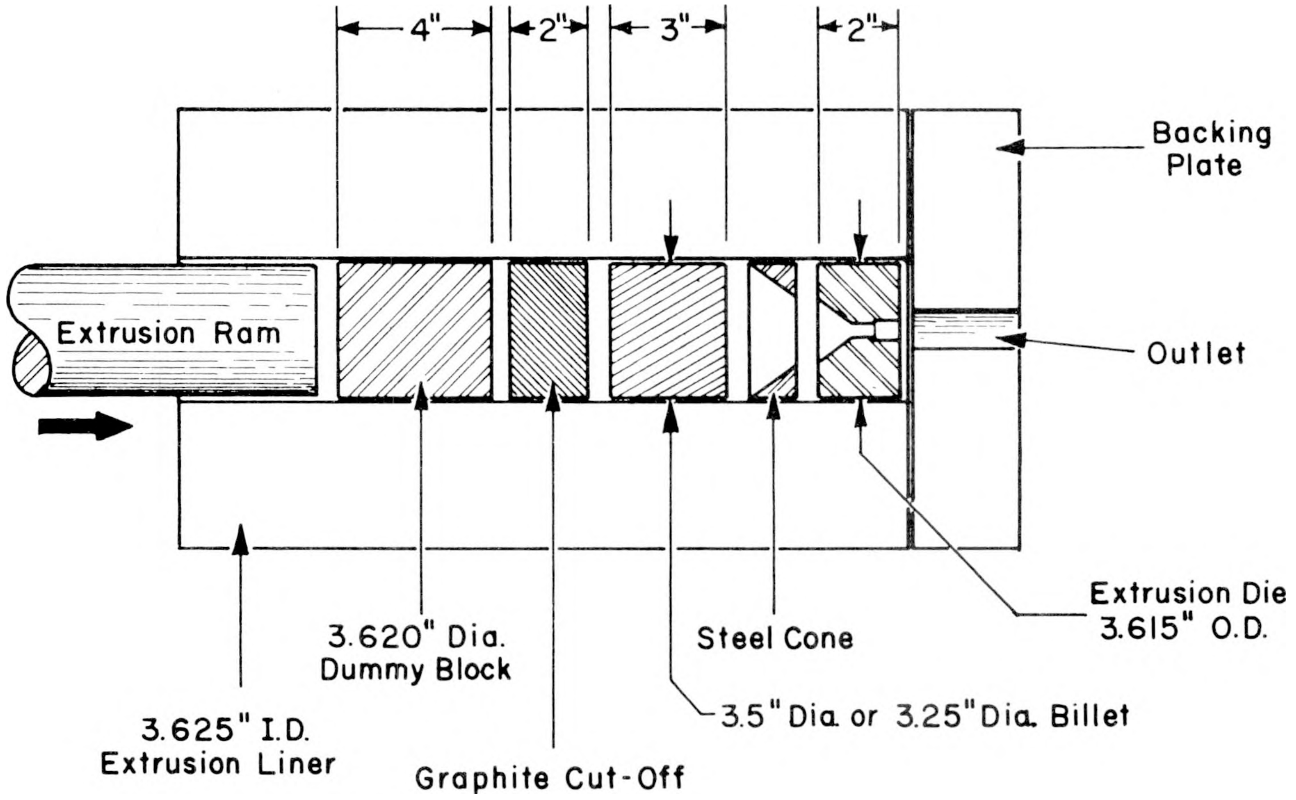
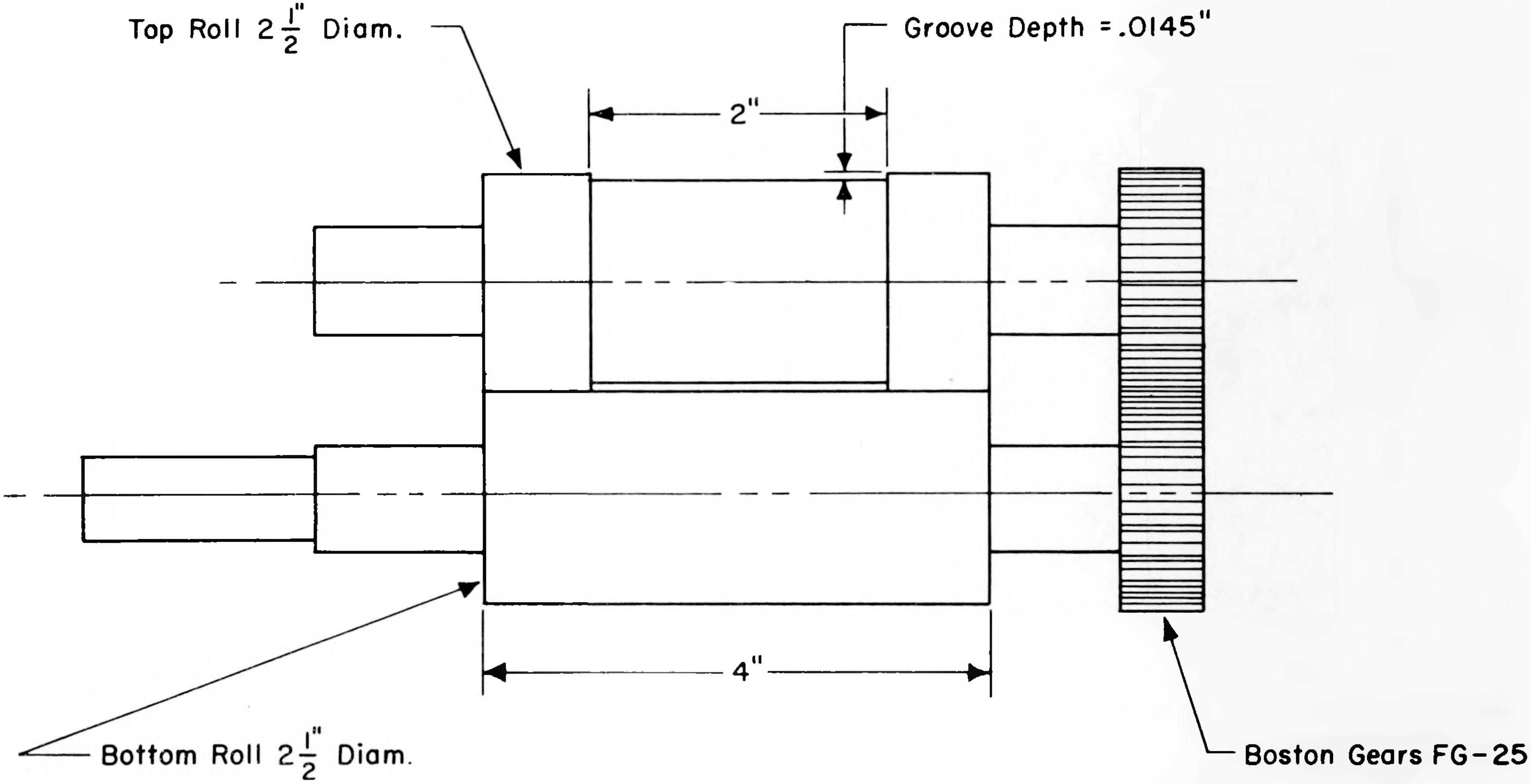


Fig. 8. Equipment for Large Scale Extrusions

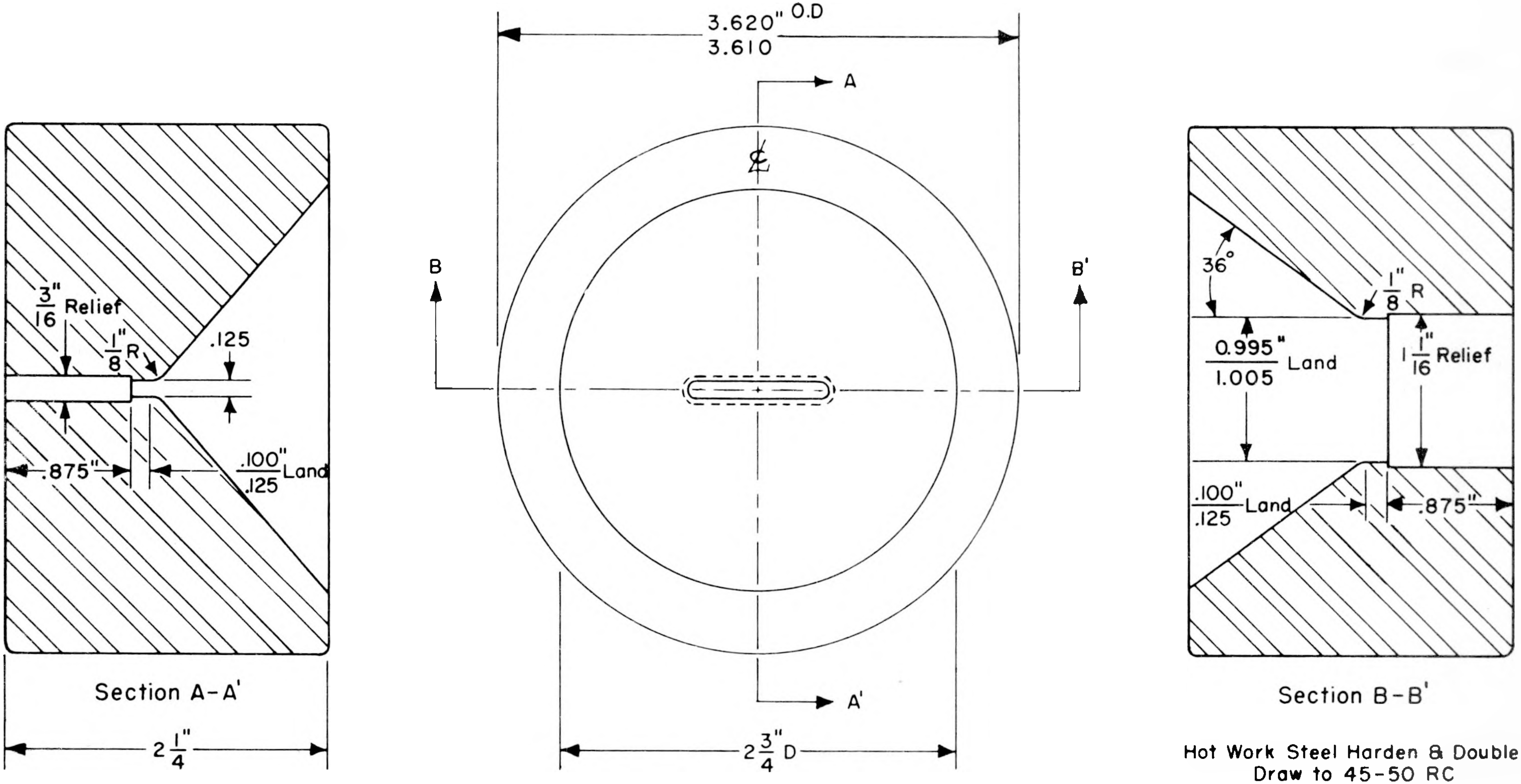
15318



Note: Diameter of Reduced Section of Top Roll to be Same as Diameter of Bottom Roll

Fig. 10 Design of Rolls

15321



Hot Work Steel Harden & Double Draw to 45-50 RC

Fig. 9 Design of Flat Die

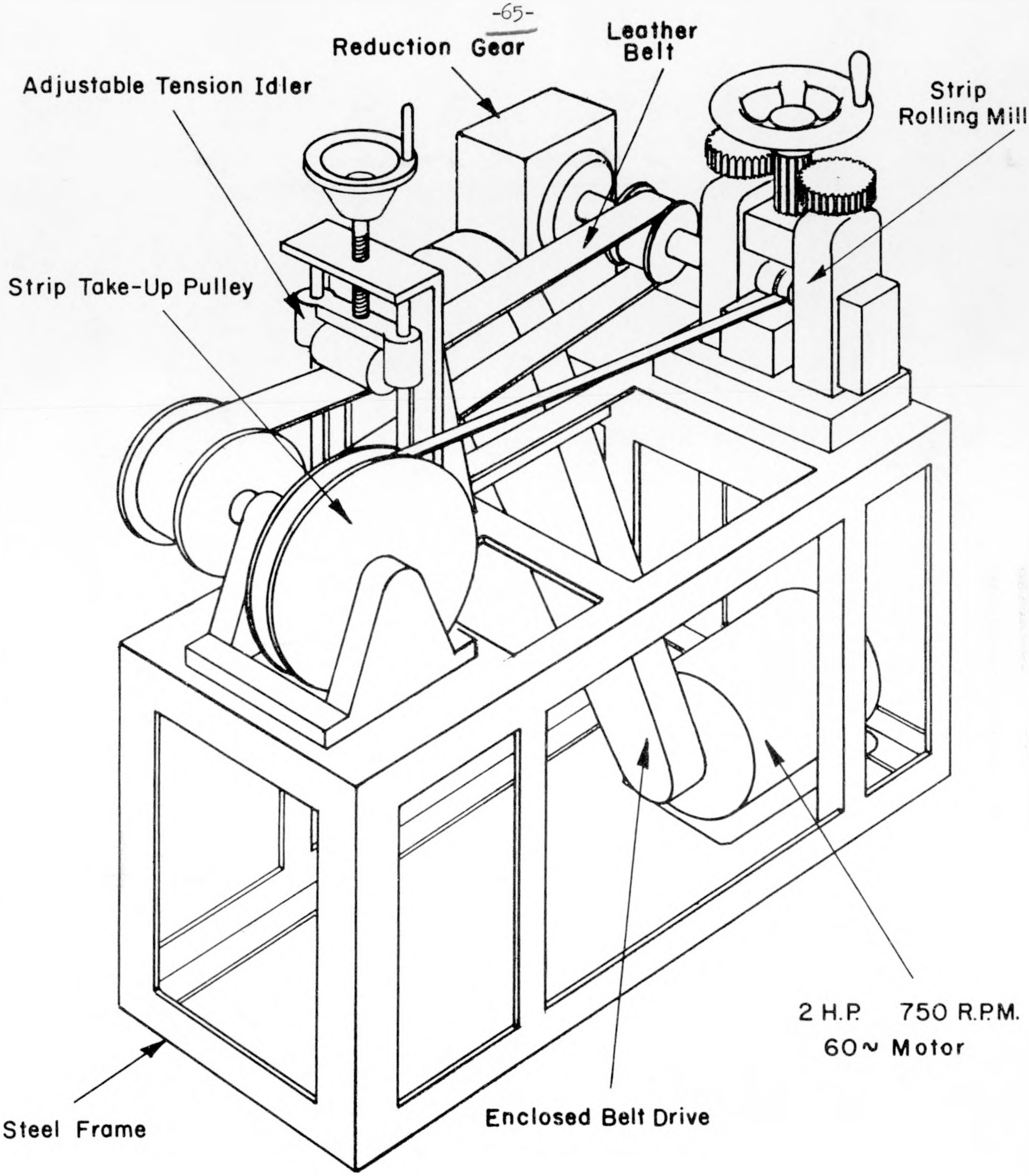


Fig. 11.
Schematic of Rolling Mill