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

THE POWDER ROLLING OF MOLYBDENUM AND TUNGSTEN

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THE POWDER ROLLING OF MOLYBDENUM AND TUNGSTEN

by

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ABSTRACT

The powder-rolling process was extensively investigated for Mo and W, including mixtures of $W-UO_2$. The powder-rolled Mo strip could be hot-rolled to sheet sufficiently ductile at 0.020 in. thickness to permit cold stamping of flat shapes; foil of 0.003 in. thickness was also produced. Additional processing of W or $W-UO_2$ strip, other than sintering, was not attempted.

TABLE OF CONTENTS

TABLE

FIGURES

INTRODUCTION

Several years ago it became apparent that powder-rolling techniques might be useful in various programs of the Los Alamos Scientific Laboratory. Early experiments confirmed the feasibility of powder-rolling several of the refractory metals as well as more common metals. Furthermore, it was determined that some of the refractory metals could be processed beyond the powder-rolling stage by heat treatment and re-rolling. This report presents parts of a subsequent investigation involving the powder rolling, hot rolling, and testing of Mo and Mo-UO_2 mixtures, as well as the more recent work on W powder rolling.

Literature references (1-8) on powder rolling deal mainly with the more common metals which are more ductile and usually of a coarser particle size than Mo and W. This difference in ductility, hardness, and particle size (approximately an order of magnitude) explains the differences to be expected in comparing the powder rolling of Mo and W with the powder rolling of the more usual metals.

The commercial Mo and W powders can be described in terms of particles which usually range from 20μ downward in diameter, with 5μ being a typical average value. The more common granular metal powders of commerce are customarily specified in terms of mesh sizes greater than

400 mesh (about 40μ particle diameter). Thus it will be appreciated that powder properties such as flow and bulk density may be greatly different with Mo and W, and that procedures described in the literature may not apply to such considerations as powder feed and strip cohesiveness.

It would also be expected that a greatly increased roll separating force would be required to compact the relatively hard Mo and W particles to green densities in the 85-90% of theoretical range. Much higher temperatures would also be expected as mandatory in sintering or hot re-rolling treatments. The usefulness of the powder-rolling literature, therefore, rested in general principles rather than in specific applications to Mo and W.

The early publications on powder rolling, as well as a good review (including patent references) by Worn (5), were available for this project. The more recent contributions on powder rolling were available only in the last stages of this work, again without pertinent reference to the metals of this report. One article which could not later be located (but thought to be British) briefly mentioned the powder rolling of Mo but gave few data.

References 9-14 serve to review and summarize the metallurgy of wrought Mo with special emphasis on the question of ductility and the ductile/brittle transition. The proposed work, however, did not indicate an opportunity to apply the finer points of Mo metallurgy related to improvement of ductility, and it appeared that the project would have to rely on Mo of ordinary commercial purity. A series of interim reports (15) on Mo was issued by Sylvania Electric Products, Chemical and Metallurgical Division, Towanda, Pa. The Sylvania work gave some attention to powder rolling of Mo, but it followed the Mo work of the present paper, and apparently the powder-rolling phase was incomplete when that part of the project was terminated.

CHAPTER 1

PROCESSING OF Mo POWDERS

1.1 Mo Powders

Table 1 lists the various Mo powders which were used, along with their characteristics. Contrary to the experience with W powders, a broad range of average particle sizes was not available from Mo vendors. Upon request, the WH-1 lot was purposely made as coarse as practical, and is not typical of a regular product. In most cases the powders were used in the condition as-received. Some Mo powders were ball milled before powder rolling; this increased the packing density (decreased porosity) on the Fisher test, and also increased the Scott bulk density. The Fisher average particle size also increased slightly, perhaps due to the change in packing density or to particle flattening. Hammer milling gave similar results. Scott bulk density for Mo might rise as high as 50 grams per cubic inch from the normal figure of 25-35.

1.2 Equipment

A Stanat 2-high mill (Fig. 1) with 10 in. dia x 8 in. wide rolls was positioned horizontally for powder rolling and equipped with a

TABLE I

CHARACTERISTICS OF Mo POWDER FROM VARIOUS SOURCES[1]

Powder Lot	Fisher Test: Porosity	Fisher Test: Avg. Part. Size, μ	Bulk Density: g/cu in.	Bulk Density: g/cc
G-1	---	4.25	---	---
G-2	0.680	5.1	34.1	2.08
G-3	0.695	4.2	29.9	1.82
G-4	0.665	4.6	33.8	2.06
S-2	0.695	3.8	30.1	1.84
S-1	0.740	5.1	28.9	1.76
S-3	0.775	2.0	19.7	1.20
F-1	0.740	4.3	24.9	1.52
F-2	0.756	4.3	22.3	1.36
W-1	0.765	4.2	25.8	1.57
WH-1	0.545	6.1	39.6	2.41
R-1	0.572	6.2	50.5	3.08

(1) All powders were made by H_2 reduction, except possibly R-1, where the process was unknown. The R-1 powder had an unusual appearance, was very dense, and did not cohere well when pressed at 50 tsi; no processing trials were made with it.

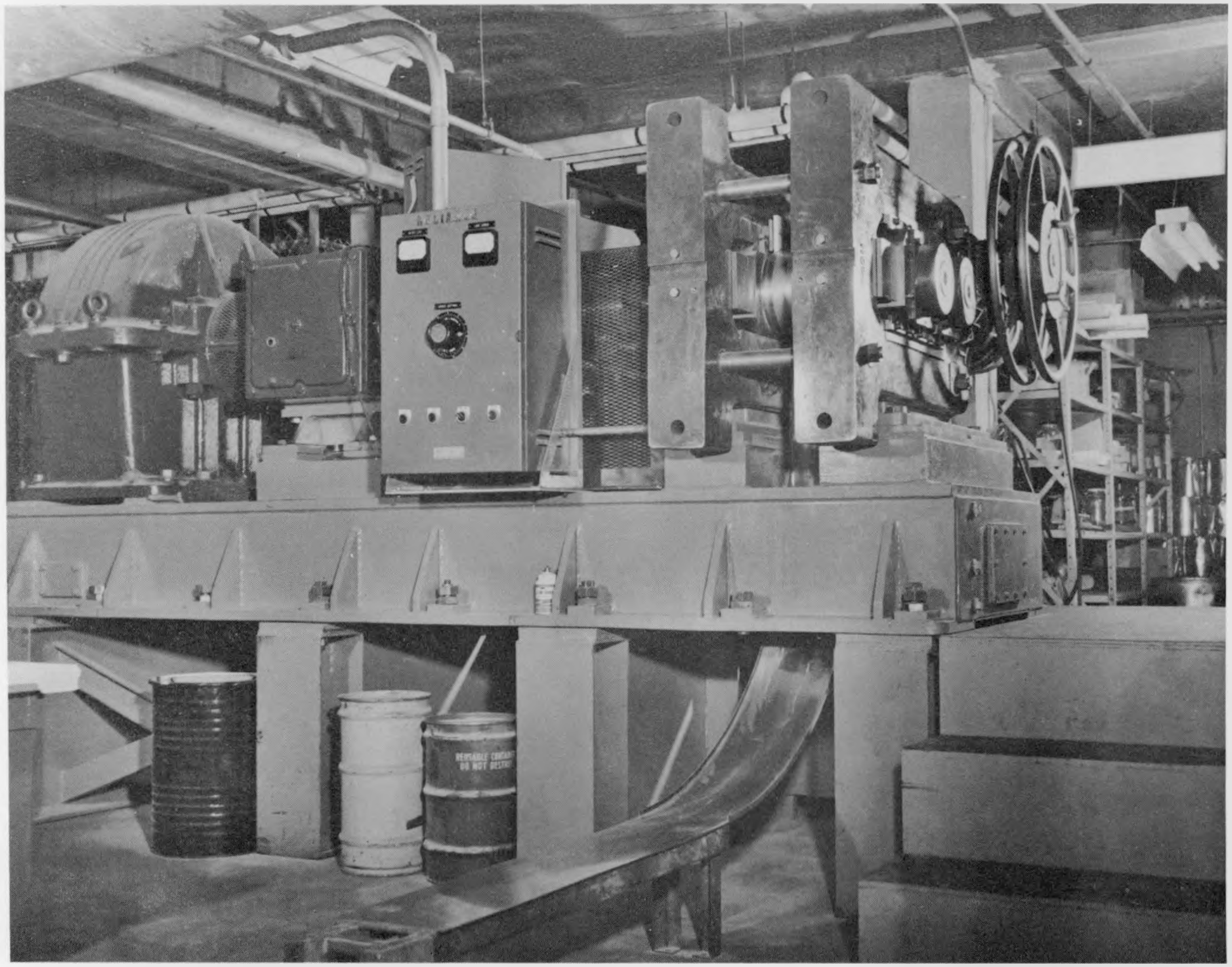


Fig. 1 Stanat horizontal mill for powder rolling.

curved delivery chute for rolled strip. The drive was a 15 H.P. reversible, variable speed (0 to 30 fpm) unit; an auxiliary gear reducer was later inserted between mill and motor to change the speed range to 0 to 6 fpm and obtain better control of the slower speeds.

1.3 Regulating Powder Feed

A 5 in. wide Y-type hopper with vibrator (Fig. 2) was used for a great many experiments (usually with 3/8-5/8 in. throat opening) in an effort to obtain uniform powder feed. Originally the steel side plates permitted considerable powder leakage. They were then lined with close-fitting teflon plates which extended well into the roll-vee. Strip uniformity was difficult to achieve, and many different baffle positions (see Fig. 3) were tried. Combination (c) made some improvement. By adjusting powder density and baffle positions in (d), some very uniform strip was obtained. A 5 kg batch would produce a strip about 10 ft long x 4 1/2 in. wide (after trimming soft edges) x 0.050 in. thick. These runs contained 6 ft lengths which varied only 2 or 3 mils in thickness.

On the whole, however, this particular Y-hopper was unsatisfactory. When various parts of the hopper were probed with a vibration pickup, the nonuniform distribution of vibration became evident. A precision Y-hopper was built but was delivered too late to fit into this program.

For short runs involving 1 or 2 kg of powder, it was found expedient to mount the teflon side plates without any hopper, feeding the

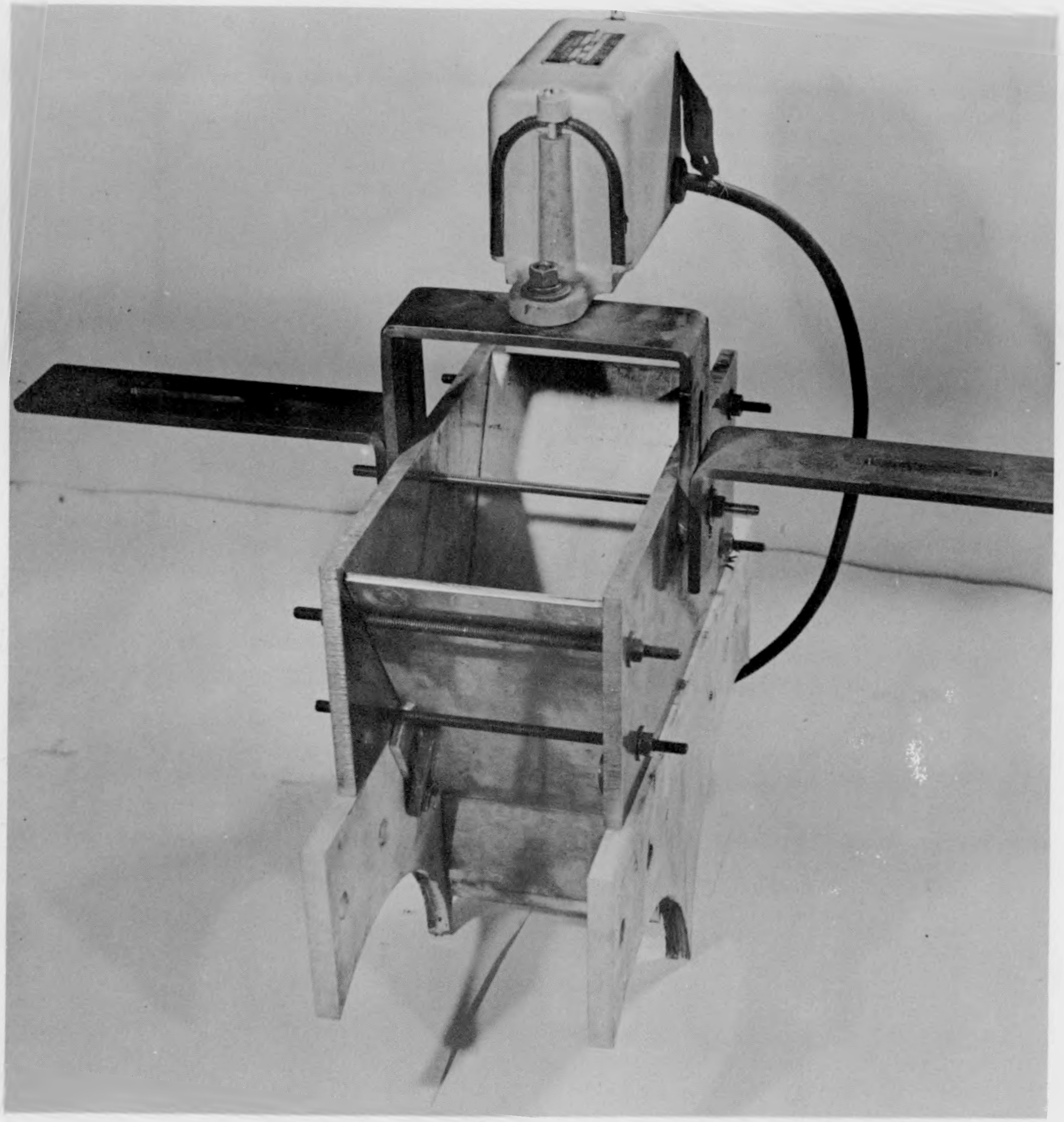


Fig. 2 Y-type hopper with vibrator and teflon side plates.

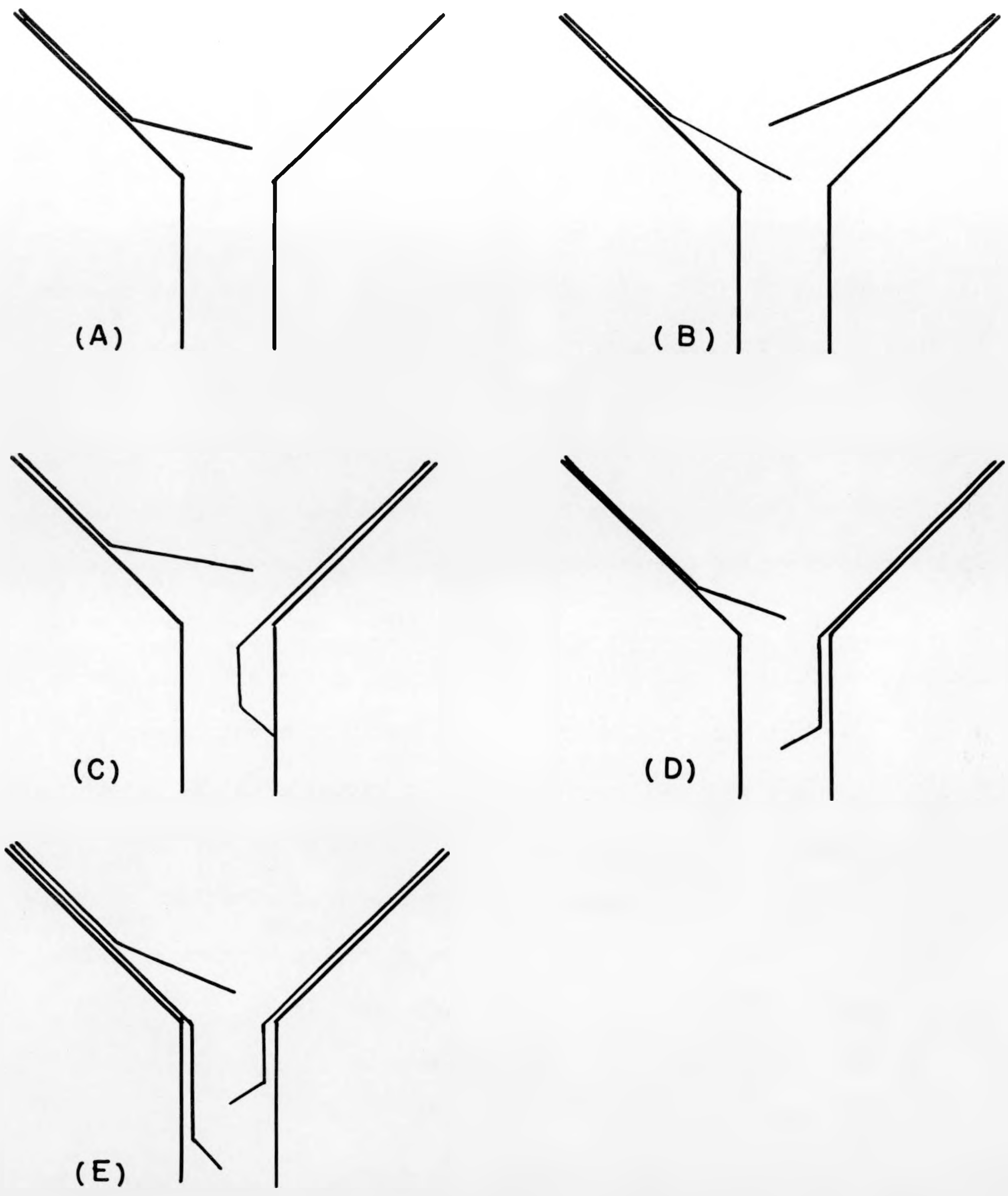


Fig. 3 Various baffle combinations tried with Y-type hopper.

powder manually directly onto the rolls. This worked well with powder of any density, and slight changes in powder level on the rolls did not greatly affect strip thickness. The unrestricted bite, however, tended to give maximum strip thickness for a given powder density, and (as explained later) usually resulted in uniform but laminated strip.

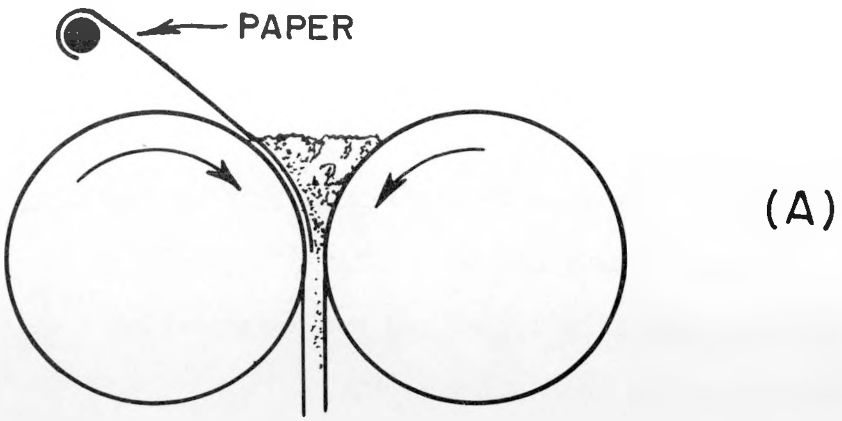
The roll bite (and strip thickness) could be reduced by covering one roll with a strip of paper as shown in Fig. 4, but localized non-uniformity or strip mottling tended to result, even if the powder was freshly screened. This demonstrated that unrestricted slipping of the rolls past a bed of powder is beneficial in removing local nonuniformities in the powder (especially "sticky" powder).

A so-called UU-hopper was formed by inserting metal vanes into slots in the teflon side plates (Fig. 4). In principle this system is much like the one just described, but has a reservoir above each roll to feed powder into the roll-vee at a rather constant height. With the lower density or stickier powders, the gates had to be opened 1/4 in. or more, but with freer flowing powders the openings could be as small as 3/16 in. Some good strips were rolled with this system, which appears to have the following characteristics:

1) There is no excess head of powder, with its attendant control problems, above the roll-vee.

2) Vibration does not appear to be necessary.

3) The system is self-regulatory to some extent because the rolls



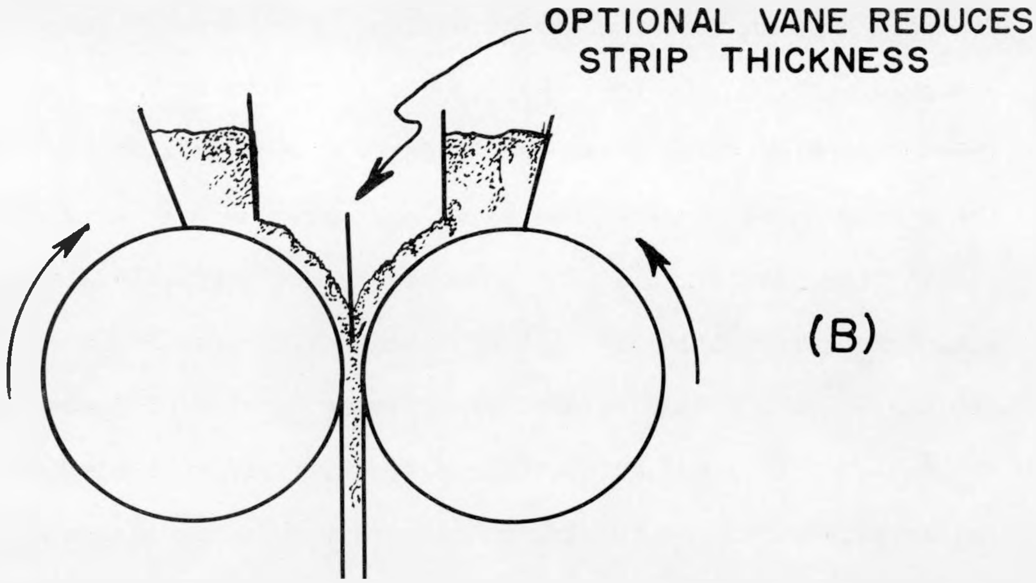


Fig. 4 Powder feeding experiments:

(A) Paper (or foil) strip with teflon side plates,

(B) UU-hopper with teflon side plates.

tend to feed more or less powder as they run faster or slower; also there tends to be greater or less slippage between the rolls and powder as the powder level in the roll-vee rises or falls.

4) Strip thickness can be regulated somewhat by the amount of gate opening. Thickness can be reduced by inserting a vertical vane into the roll-vee as shown in Fig. 4.

1.4 Conditioning of Mill Rolls

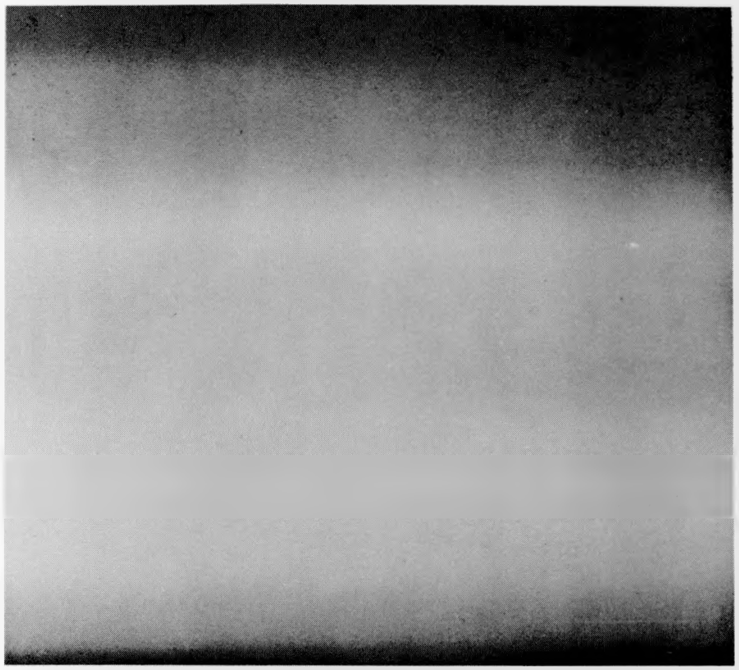
A phenomenon which was not understood very well in the early stages of the work considerably complicated the evaluation of various factors in powder rolling. This phenomenon was the instability of the friction between the steel rolls and the Mo powder. It was known or observed early in the work that the rolls had to be kept clean and free of foreign films (fingerprints or minor smudges on the rolls would show up as density changes in the strip as well as visually). But it took considerable time to observe that perfectly clean rolls would change frictionally just by standing idle overnight or for a few days. This became evident when larger batches of powder were run without first "breaking in" the rolls; the strip thickness would increase pronouncedly with each roll revolution for a length of 10 ft or more.

It thus became necessary to break in the rolls with scrap powder before rolling an experimental lot if observations were to be on a "normal" or dependable basis. The increase in strip thickness "before" and "after" might be as great as 100% in some cases. It was also observed that if Mo powder contacted the stationary rolls for any substantial length of time, a "spot" on the strip might result. Furthermore, Mo powder lots from different vendors might respond differently to hopper and roll variables. Figure 5 shows radiographs of two strips rolled consecutively from very similar powders with the same hopper and mill settings. Most of the streaks in the nonuniform radiograph were not visible on the rolls.

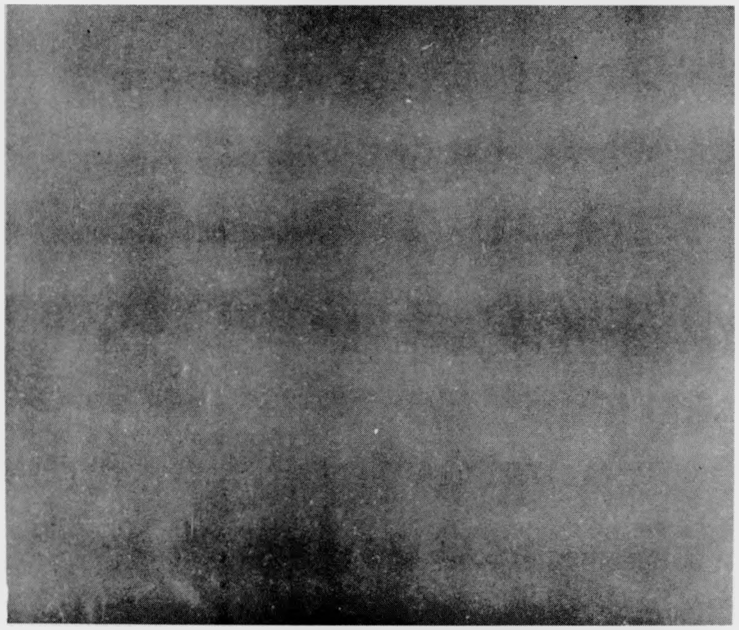
1.5 Mill Variables

During early operations with the Stanat mill a series of experimental runs was made to determine the effect on the strip of such variables as roll speed, roll opening, and hopper opening. Although the data obtained could be plotted as smooth curves, the frictional variable was not under control; and the results are believed unreliable. Some of this information would be merely academic anyhow. It was difficult to obtain quality strip with speeds above 3 fpm; thus the tendency for strip to become thinner and less dense at higher speeds is of little significance. With regard to roll opening, usually set at 0.008-0.012 in., thicker strip obtained with greater openings was achieved at the cost of density and strength (including handling ability).

Rolls with finishes ranging from 10 to 40 microinches were used; the effect of finish other than on appearance was, however, indefinite under the prevailing conditions. Chromium-plated rolls were tried, and



NON-UNIFORM, BANDS RUNNING IN ROLLING DIRECTION



← ROLLING DIRECTION →

UNIFORM, WITH VERY FAINT BANDS IN ROLLING DIRECTION

Fig. 5 Radiographs of two strips rolled consecutively from different but highly similar powders with the same hopper and mill settings.

appeared to reduce the friction between powder and rolls so that only thin weak strip resulted.

1.6 Powder Variables

All the Mo powders listed in Table 1 except R-1 appeared to powder-roll without cracking, regardless of powder bulk density or particle size. The resulting strips varied in thickness, density, and uniformity with different powders; but definite correlations were not made partly because of the frictional variable previously discussed. The breaking-up of powder aggregates by ball milling or hammer milling sometimes yielded denser and thicker strip (due to increased powder bulk density), but otherwise appeared to reduce the natural cohesion of the as-received powder.

The main difficulty common to all the Mo powders was a tendency for the strip to laminate when conditions were suitable to produce a thick, dense strip in the range of 0.050 to 0.068 in., although there were rare occasions when 0.060 in. strip did not laminate. No practical way was found to eliminate this lamination except to control powder feed so that a thinner strip was produced. One "impractical" method, however, was discovered; it was found that mixtures containing considerable UO_2 did not laminate whereas the Mo alone did.

It appears that strip thickness increases with higher bulk density of the Mo powder if other factors remain the same. The control of powder flow and frictional coefficient, however, is difficult; and these factors counteract the effect of bulk density in many instances.

It was hoped that a slight amount of controlled lubrication would perhaps reduce strip thickness to a point where no lamination would occur. Graphite and MoS_2 in the amounts of 0.1% (mixed by ball milling), however, had no effect on the strip thickness, and presumably did not change the friction between rolls and powder. The same amounts of cetyl alcohol and lithium stearate, on the contrary, lubricated the powder (or rolls) so well that only a very thin non-coherent strip resulted.

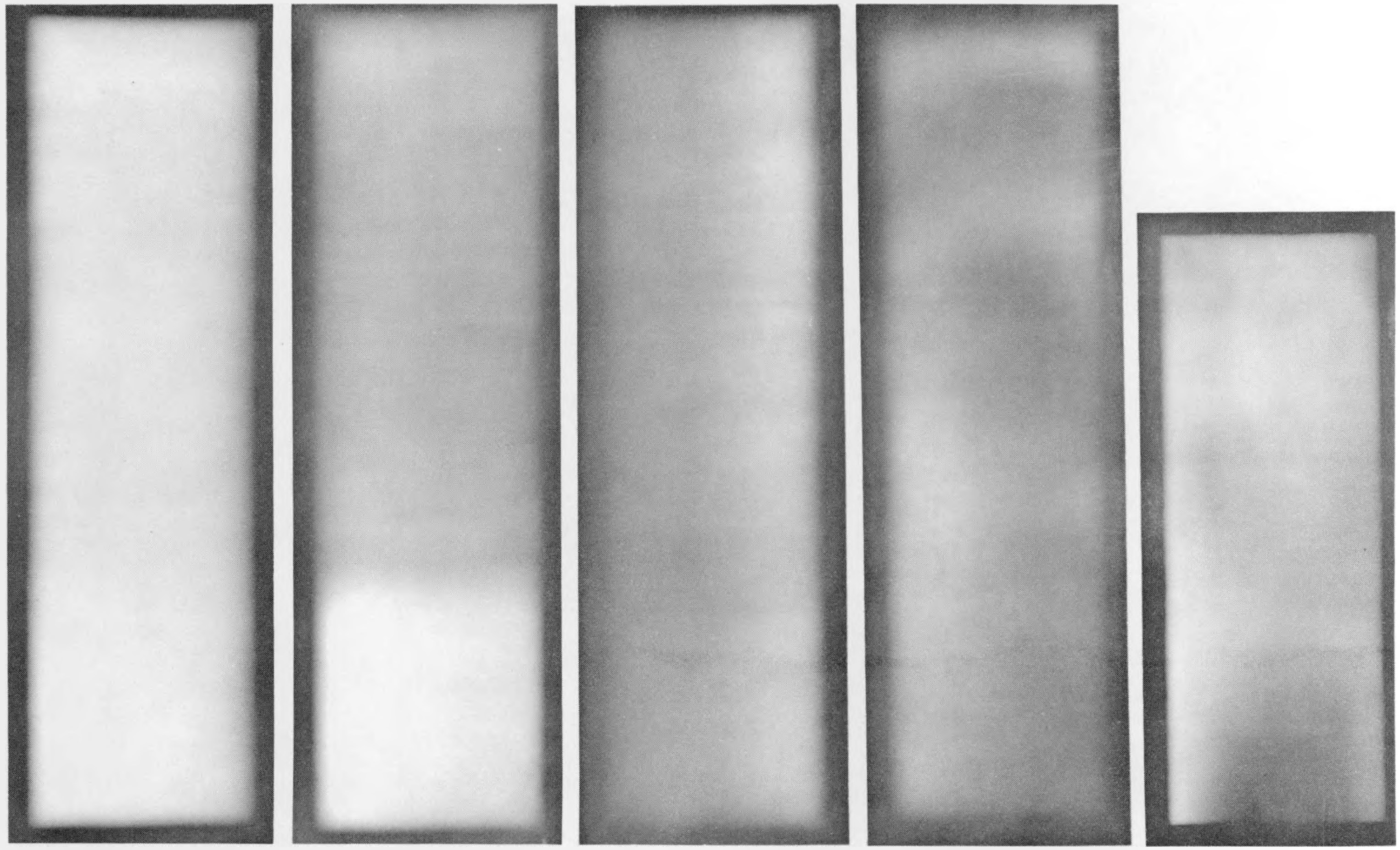
1.7 Determination of Strip Quality

Quality of strip as to uniformity was checked visually for appearance, by micrometers for thickness on a grid pattern, and by radiography for thickness and/or density variation. Exceptional strip uniformity and nonuniformity were both usually apparent to the eye. Density variations were generally registered visually on the surface, so that surface appearance tended to agree with radiographic film. Radiographs representing a uniform strip along with several varieties of nonuniformity are shown in Fig. 6.

1.8 Hot Rolling of Powder-Rolled Mo Strip

1.8.1 Small 4-High Mill

Early hot-rolling runs were made with a small 4-high mill containing 8 x 8 in. backup rolls and 2-1/2 in. dia, high speed steel work rolls. A small Mo-resistor hydrogen-atmosphere furnace was mounted



(A) UNIFORM STRIP

(B) SUDDEN INCREASE IN THICKNESS AT BOTTOM

(C) THICKER OR DENSER ON RIGHT SIDE

(D) GENERALLY NON - UNIFORM

(E) TYPICAL FISH-TAIL NEAR END OF STRIP (BOTTOM)

Fig. 6 Radiographs of powder-rolled strip showing types of nonuniformity. (Reduced in reproduction).

close to the work rolls. Rolling specimens, 3 in. x 1-1/2 in., were taken longitudinally from powder-rolled strip of about 50 mils thickness. After heating in the furnace on a Mo combination pusher-tray, they were shoved through the furnace directly into the rolls with very little loss of temperature.

For warm rolling an oil pan with electric heaters was mounted immediately in front of the rolls.

Furnace temperatures near 1500°C (2730°F) were suitable for the breakdown passes. Although the furnace flame-heated the rolls to about 200°C (390°F), chilling in the rolls was quite pronounced; only in the breakdown passes did the piece emerge from the rolls with any reddish heat color. Both green and sintered (1700°C-3090°F) pieces responded similarly to the rolling. With green pieces, it was the practice to hold them in the furnace 5 min before the first pass; thus they were partially sintered before entering the rolls. The mill had a maximum roll speed of 40 fpm, but best results were secured at 20-30 fpm; at slower speeds there was more edge cracking and splitting.

Hot rolling on a descending temperature schedule similar to the following was also satisfactory:

Thickness Range	% Reduction	Furnace Temperature
0.047 to 0.034 in.	29	1550°C (2820°F)
0.034 to 0.025 in.	27	1275°C (2325°F)
0.025 to 0.021 in.	16	1225°C (2235°F)
0.021 to 0.015 in.	29	1155°C (2110°F)
0.015 to 0.003 in.	80	Hot oil bath or room temperature

A reduction of 4 to 6 mils on each of the first two passes and 4 mils on each of the next two passes worked out well on powder-rolled strip of 45 mils and thicker. Reductions as high as 8 to 10 mils often resulted in cracking or splitting. After breakdown, the reductions were about 2 mils per pass, and only 1 mil below 0.020 in. Below 0.007 to 0.010 in., the hand screw-down was as tight as it would go, and reductions per pass were very small.

With the heavier strip, some "ductility" was apparent at about 30 mils thickness. Density determinations showed that theoretical or maximum density was attained at about 40-60% reduction from original thickness. Figure 7 shows the powder-rolled specimens and 3 mil foil resulting from hot and cold rolling.

No special efforts were made to obtain a fine grain structure or maximum ductility in the as-rolled or annealed condition. Figure 8 shows as-rolled and high-temperature recrystallized grain structures for Si-doped and undoped Mo. Photograph D reveals unsoundness developed at 2300°C (4170°F) apparently by the vapor pressure of residual Si-dope; prior sintering at 1700°C (3090°F) before hot rolling appears to volatilize most of this "dope," resulting in a typical "non-sag" grain structure (F) when heated at 2300°C (4170°F).

1.8.2 Large Rolling Mill

A new 16 in. Waterbury-Farrel 2-high, 4-high mill with a 75 H.P. drive, rated at 1,000,000 lb separating force, became available somewhat later. Figure 9 shows this mill (as 2-high) with a Mo-wound

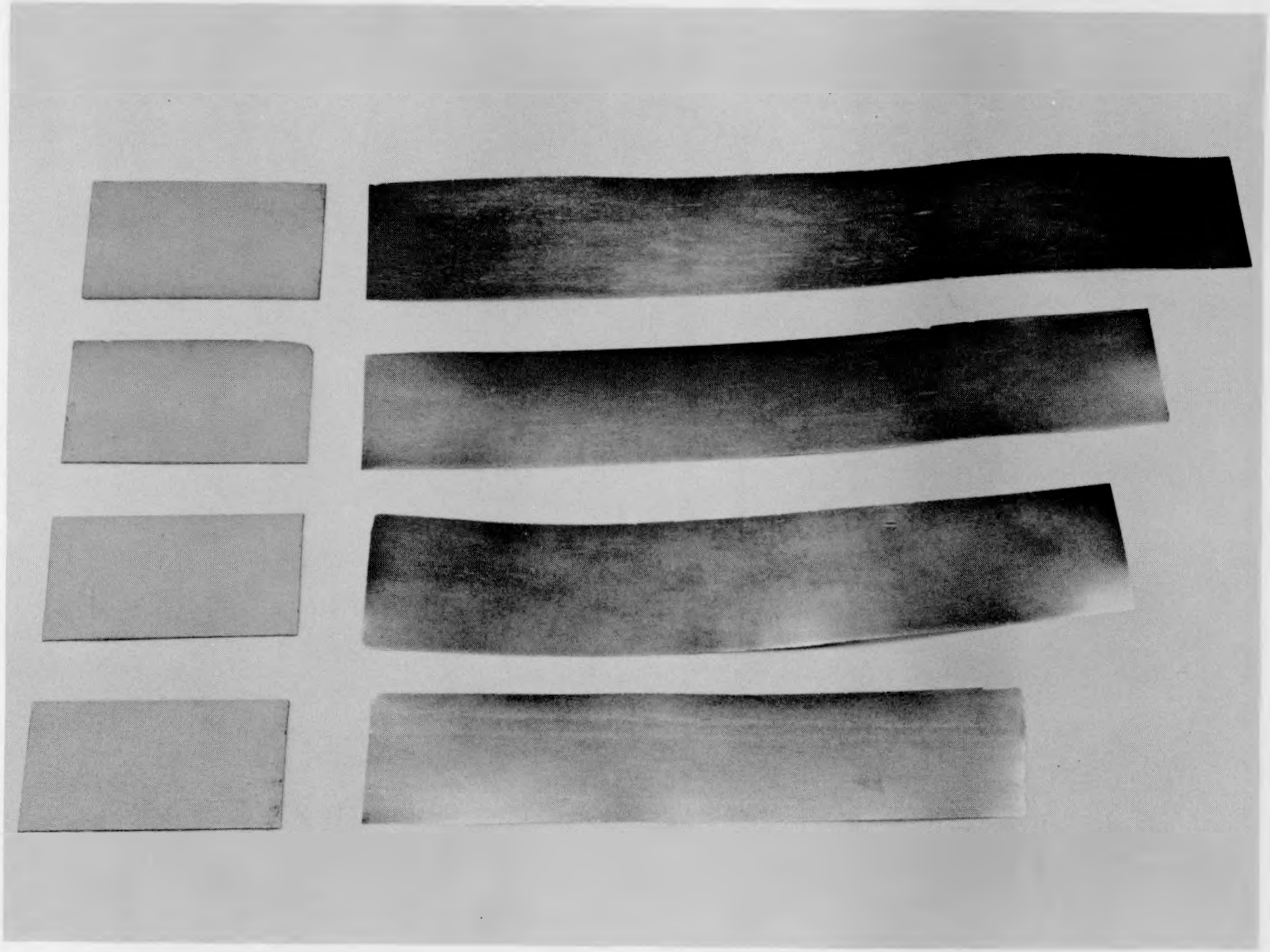


Fig. 7 Powder-rolled specimens (1-1/2 in. x 3 in.) and resulting 3 mil foil.

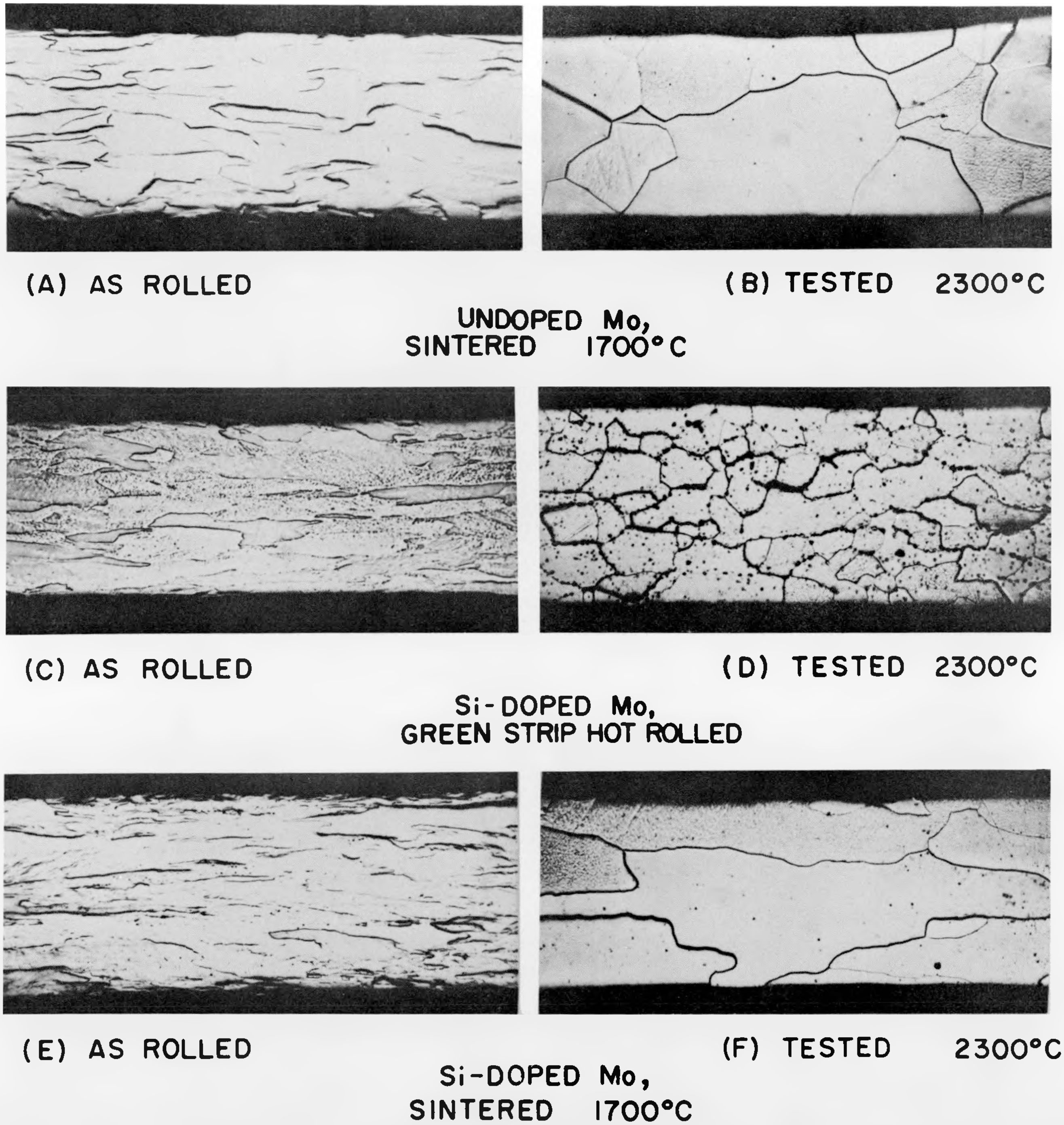


Fig. 8 Effect of Si-dope and processing on microstructure of 7 mil sheet after heating in H_2 to 2300°C (4170°F). 150X

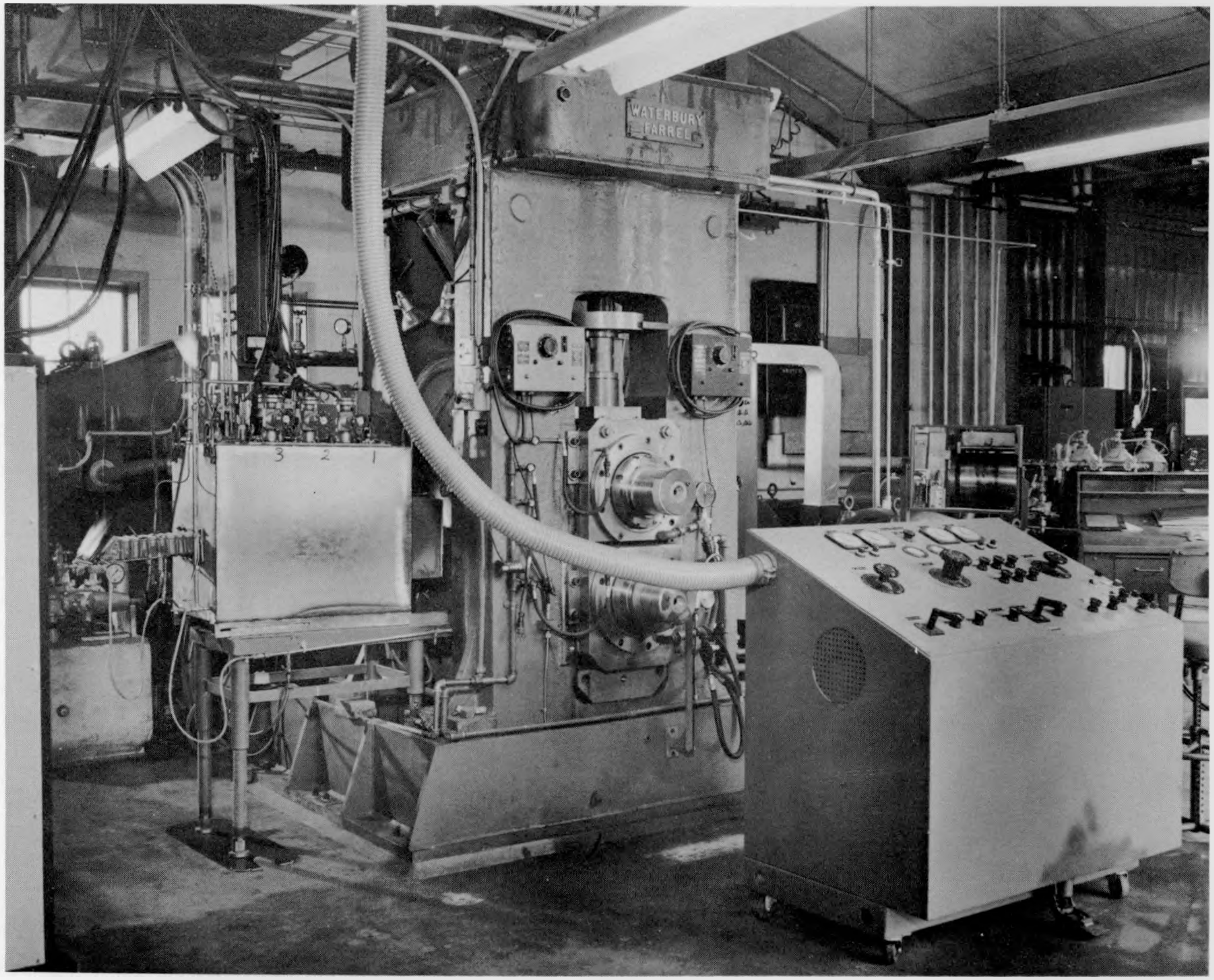


Fig. 9 Large rolling mill with heating furnace and control unit.

furnace built at Los Alamos, arranged so as to slide close to the rolls. With this mill it was possible to hot-roll the powder-rolled strip in full widths of 4-1/2 in. The large diameter rolls were much better than the small mill previously used in regard to edge cracking of the strip as well as hairline surface cracks.

Pieces of powder-rolled strip 12-15 in. long were heated to 1450-1500°C (2610-2700°F) in the furnace and pushed through the furnace directly into the rolls turning at 20-30 fpm. A 15 in. long piece 0.055 in. thick required about 18 passes on the 16 in. rolls (2-high) to emerge (after dividing) as two 17 in. pieces 0.020 in. thick.

Green strip or strip sintered overnight at 1450-1700°C (2610-3090°F) rolled equally well if the starting piece was uniform. Sintered powder-rolled strip is shown in Fig. 10. When rolled to 0.020 in. sheet such strip was capable of being stamped at room temperature into 3 in. dia washers without cracking (Fig. 11).

The 4-high arrangement with 5 in. work rolls was tried briefly, but exhibited much more strip cracking on the breakdown passes. On powder-rolled strip previously hot rolled to 0.035 in., however, the 4-high setup reduced the sheet to 0.020 in. in about one-third as many passes as required with the 2-high 16 in. rolls. The separating force was quite high even with the 5 in. rolls, ranging from about 150 tons for a 4 mil pass on 0.035 in. sheet to about 330 tons for a 2 mil pass on 0.022 in. sheet.

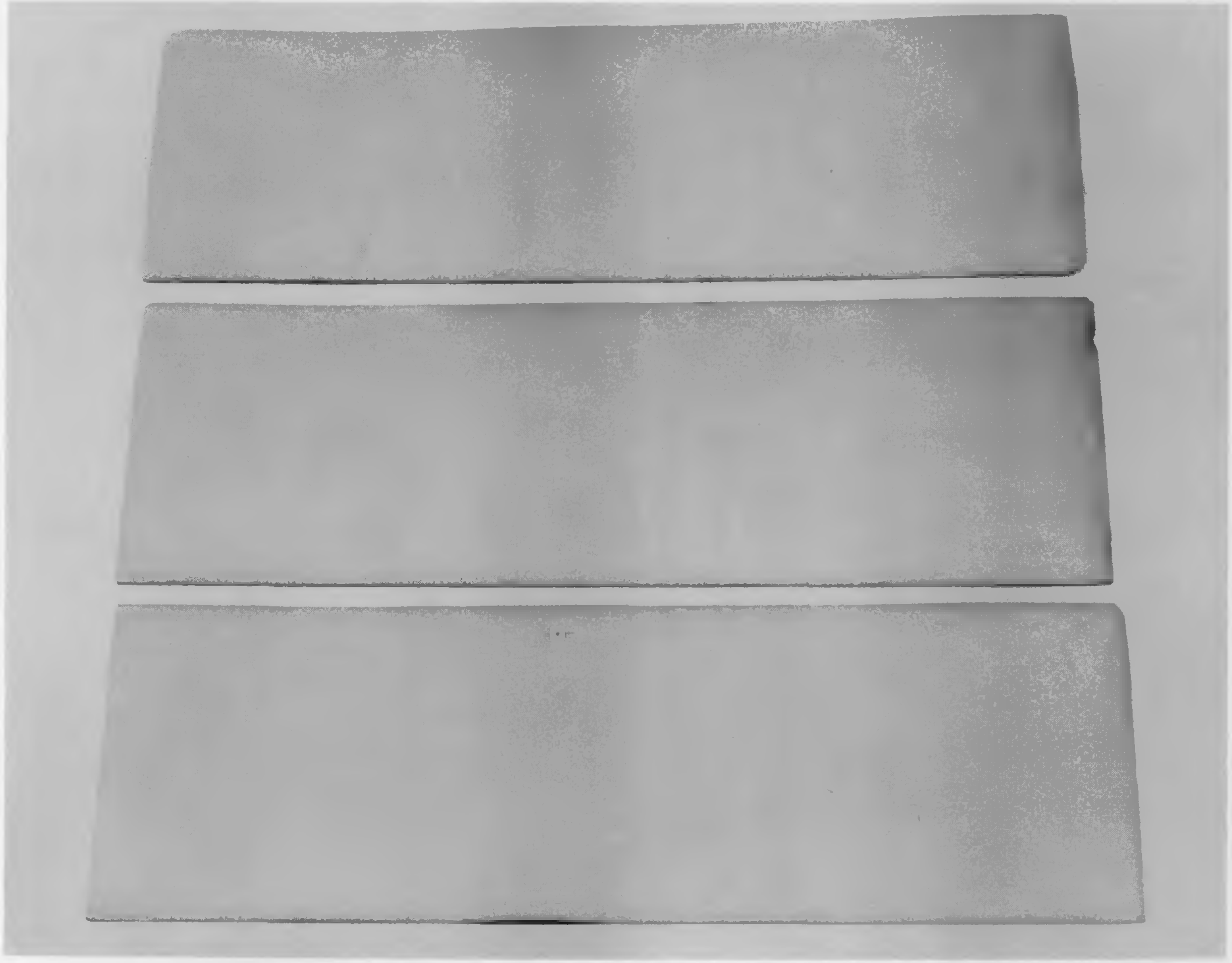


Fig. 10 Sintered pieces (4-1/2 in. x 15 in.) of powder-rolled strip, edges sanded before sintering.

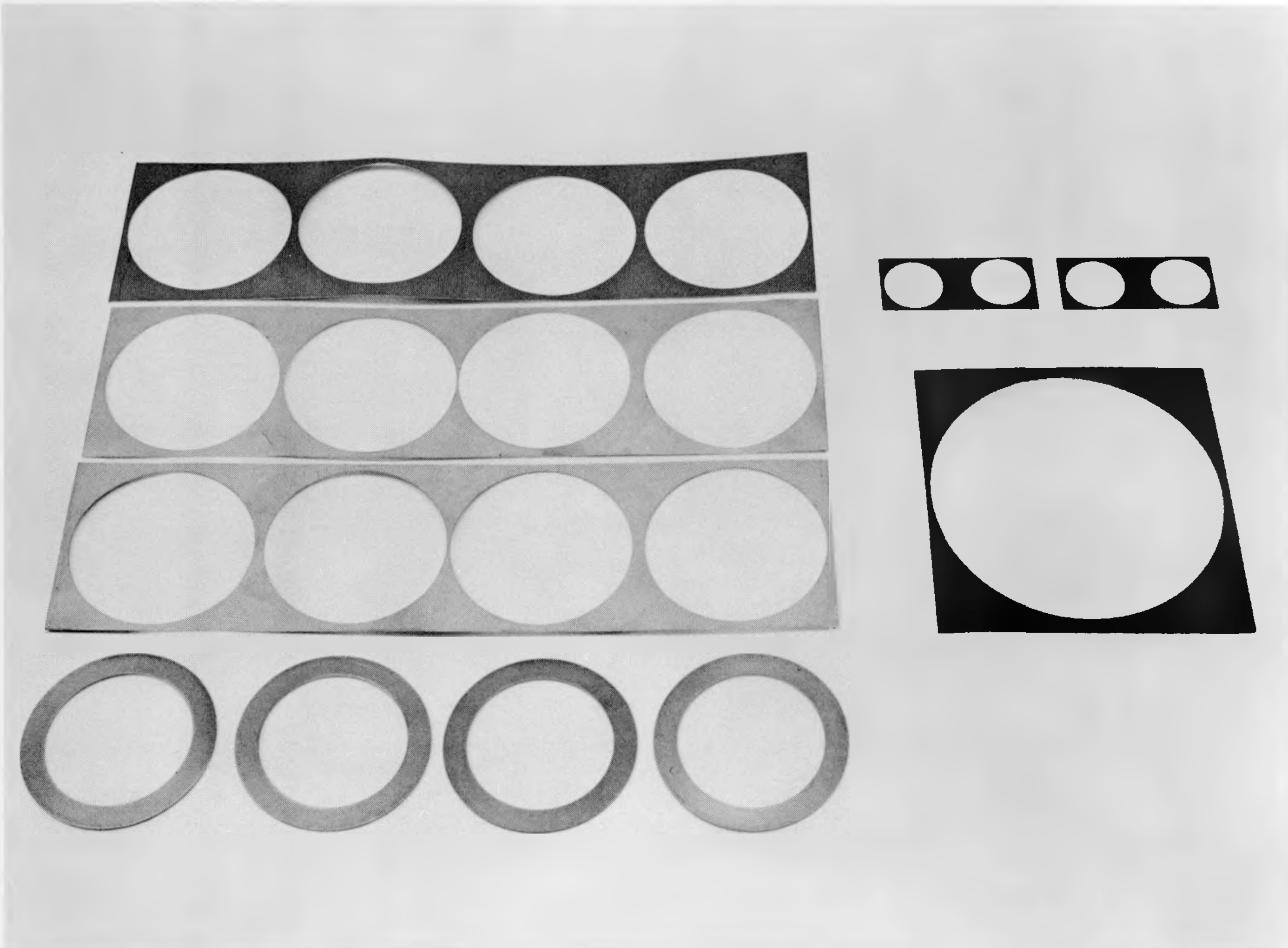


Fig. 11 Washers (3 in. O.D.) stamped cold from 20 mil hot-rolled Mo sheet.

CHAPTER 2

PROCESSING OF W POWDERS

Since Mo and W powders are similar in many ways, and since the same equipment was used, it would be expected that the powder-rolling operations and results might be similar also. This turned out to be true, and it will only be necessary to highlight the differences and to state the results achieved. The main fundamental differences are that W powders can be obtained in a greater range of particle sizes and the particles are harder and less ductile than Mo.

The work with W on the Stanat horizontal mill utilized only the teflon side plates; these were marked so powder could be hand-fed to heights of 1 to 4 in. Tungsten powders varying from 1 to 10 microns average particle size (Fisher) were tried for rollability in the "as-received" condition. The powders with 3 micron and smaller averages did not roll well and cracked very easily. The coarser powders tended to cohere well and yielded higher as-rolled densities. Some of these coarse powders flowed so well, however, that powder leakage was severe. By ball milling these "leaky" powders a short time, the flowability was reduced, bulk density increased, and leakage controlled.

Tungsten powders containing UO_2 were also powder-rolled. The problems of cracking and noncoherence became greater as the amount of UO_2 increased and as the UO_2 particle size became finer. With judicious selection of the UO_2 particle size, however, mixtures containing 50 v/o UO_2 could be powder-rolled satisfactorily. Densities of such powders after rolling and sintering tended to be about 5% lower (based on per cent of theoretical) than straight W.

Figure 12 shows some of the earlier results in the sintered condition as related to powder type. Figure 13 shows a variety of strip rolled with different powders. The upper specimen was rolled with side plates spaced 5 in. (Powder consumption, partly due to the high density of W, was so rapid that the side plates were spaced at 2-1/2 in. for most work).

A frictional instability existed between W powder and the steel rolls similar to that encountered with Mo. A given powder might yield a strip 50 mils thick on the first roll revolution, gradually increasing to 65 mils thick on the fourth revolution. Breaking-in the rolls with suitable scrap powder thus became a standard procedure.

The best combinations of thickness and density appeared to result from powders of high bulk density run at a speed of 1 fpm with roll opening of 10 mils and powder head as high (up to 4 in.) as possible without cracking the strip. Maximum thicknesses attained in 2-1/2 in.

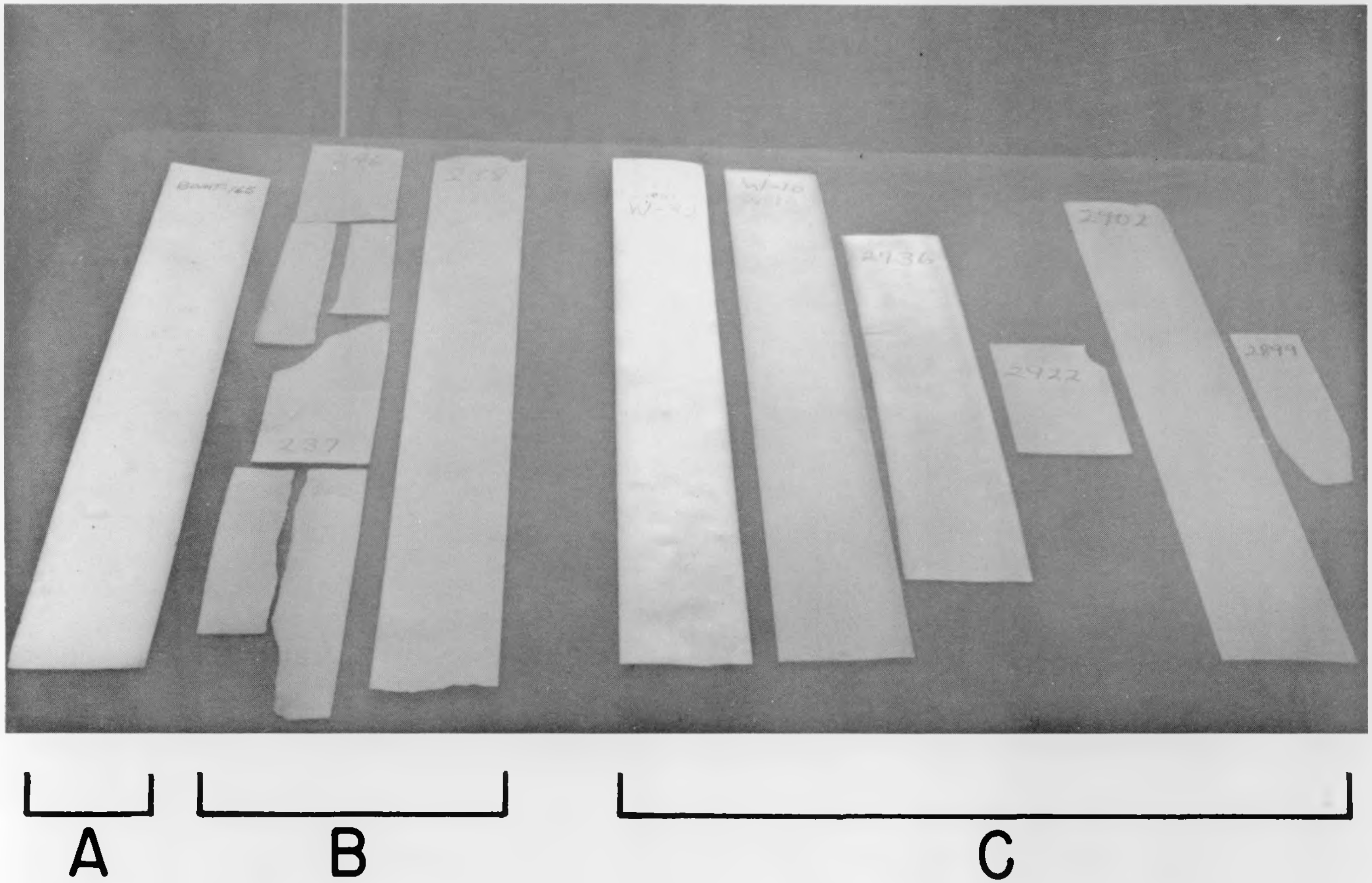


Fig. 12 Sintered (1700°C, 3090°F) pieces of 2 in. wide strip:

(A) W plus 20 v/o UO_2,
(B) W plus 50 v/o UO_2,
(C) W powders ranging from 2-10u average.

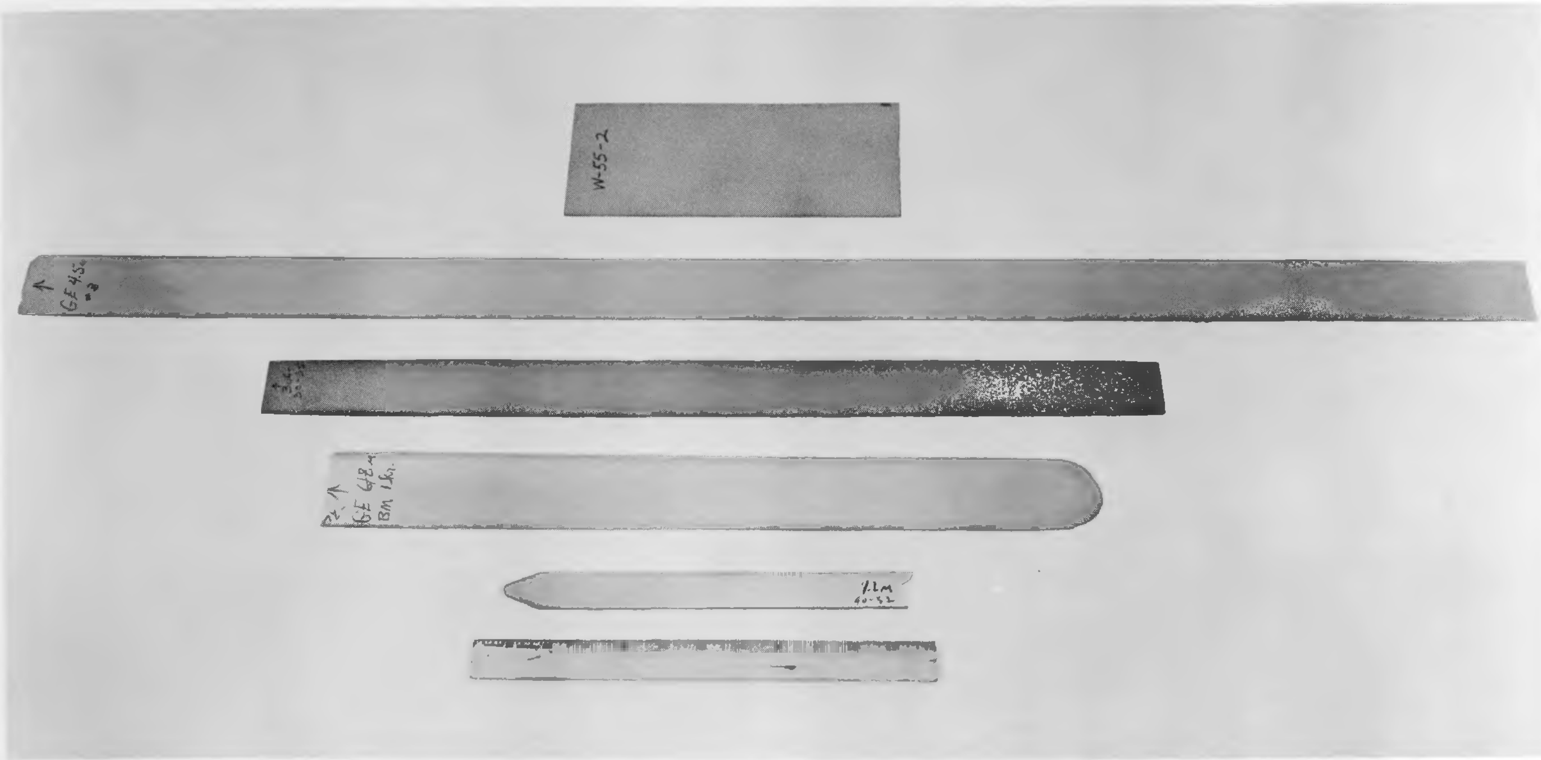


Fig. 13 As-powder-rolled W pieces, top to bottom:

1) 6.8μ W, 4-1/2 in. x 10 in., 5 in. feed width
2) 4.5μ W, 40 in. long, 2-1/2 in. feed width
3) Mixture of W with 50 v/o UO_2, 2-1/2 in. feed width
4) 6.8μ W, ball milled 1 hr, 2-1/2 in. feed width
5) 7.2μ W, as-received powder, 2-1/2 in. feed width

wide strip were in the range of 60-68 mils, with green and sintered (1700°C, 3090°F) densities running about 80-85% and 90-95% respectively. Lamination tendencies were not encountered until thicknesses near 70 mils were reached. This advantage as compared with Mo would appear to be due to the greater roll deflection caused by W in order to attain the same per cent of theoretical density. Figure 14 shows the results obtained in systematic rolling tests with a particular powder.

Slight changes in powder compositions can drastically affect rollability. One powder which rolled well without cracking under a variety of mill settings became very "touchy" and tended to crack when only 1% of sub-micron TiO_2 was blended with it by ball milling.

Roll finishes varying from 10 to 50 microinches were tried, and it was determined that sound strip of about 65 mils thickness could be rolled with each of them under optimum conditions.

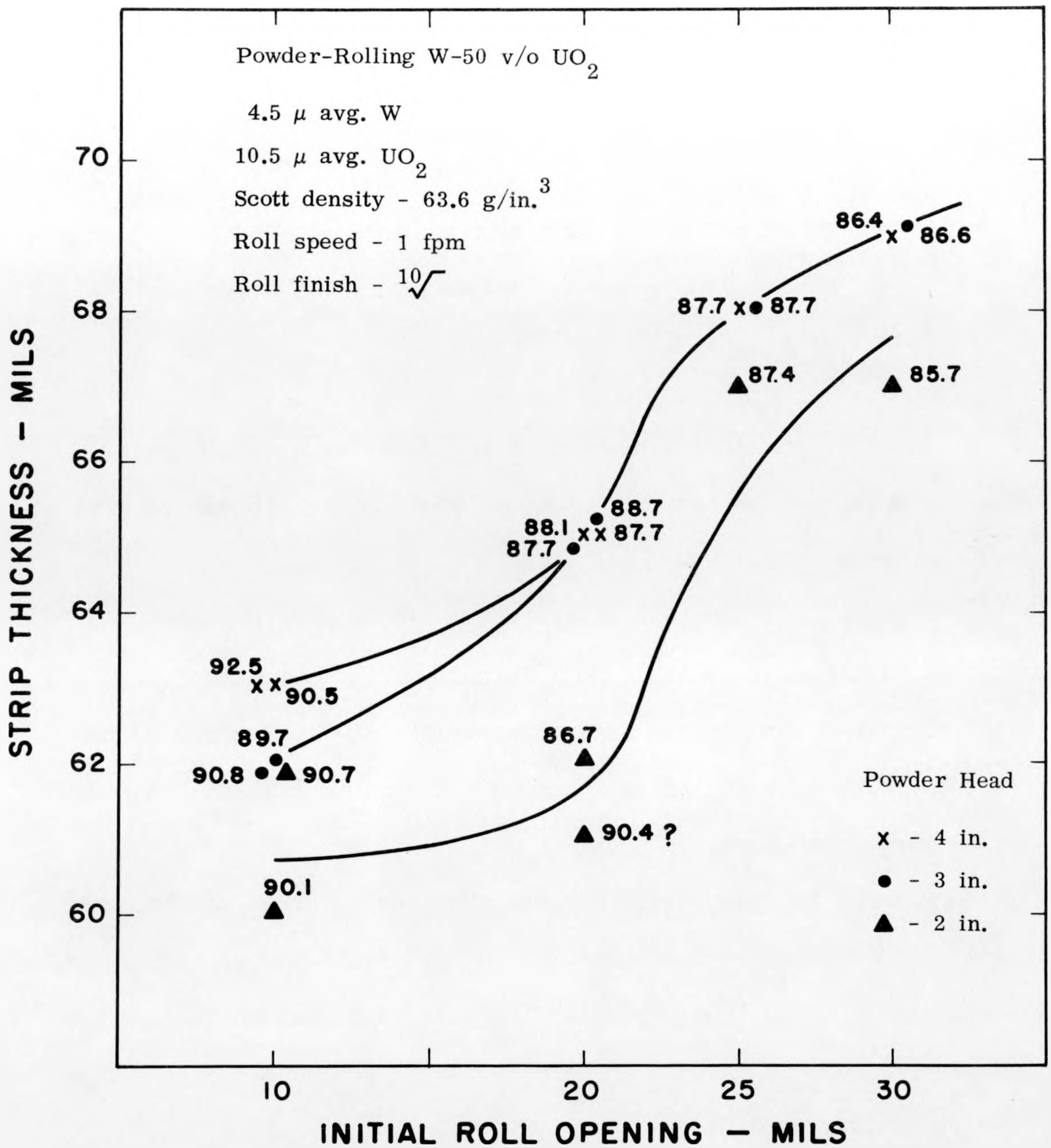


Fig. 14 Variation of strip thickness and density with changing roll opening and powder head. Stanat mill with 10 in. dia x 8 in. rolls. Numbers beside plotted points indicate per cent of theoretical density as sintered at 1700°C (3090°F).

CHAPTER 3

SUMMARY

1. Most commercial H_2-reduced Mo powder appears suitable for powder rolling into strip of about 0.045-0.060 in. thickness with 85% of theoretical density.

2. The main problems in the powder rolling of Mo were: a) maintenance of constant roll friction for intermittent work; b) provision for uniform feed of powder to the rolls; and c) the tendency for Mo strip to laminate as the maximum thickness and densities were approached.

3. The Y-type hopper appeared unsuitable for Mo powder, but such limitations might be overcome with an improved Y-hopper incorporating both precision controls and wide latitude of adjustment. A hopper design called UU, thought to be original, appeared quite promising for Mo or similar powders.

4. The hot rolling of powder-rolled Mo strip was feasible at furnace temperatures near 1500°C (2700°F) for the initial passes. Uniform powder-rolled strip is a necessity for this purpose, and the breakdown

passes are critical. The production of 0.020 in. thick strip on a hot-mill with larger rolls appeared more practical; sufficient ductility was obtained at this thickness to permit the stamping of flat shapes.

5. The powder rolling of W proved to be similar to that of Mo, differing in degree due to the hardness and ductility difference of the two metals. Lamination tendencies were less pronounced than with Mo. Powders finer than 3 microns did not roll well. Mixtures of W with UO_2 could be powder-rolled successfully.

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