1961

A preferred orientation study of sheet formed by rolling copper, nickel, titanium and tungsten metal powders

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Department:

A PREFERRED ORIENTATION STUDY OF SHEET FORMED BY ROLLING COPPER, NICKEL, TITANIUM, AND TUNGSTEN METAL POWDERS

BY

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A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE, METALLURGICAL ENGINEERING

Rolla, Missouri

1961

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ABSTRACT

Pole figure scans of strip rolled from copper, nickel, titanium, and tungsten metal powders were made on samples taken across the width of the strip, and with various amounts of surface material removed, to determine if any preferred orientation existed. No preferred orientation was found in any of the samples examined.

The method used was a modification of the Schultz technique, using the Norelco Pole Figure Fixture, whose geometry and operation is described, in conjunction with the Norelco diffractometer.

Defocusing resulting during the run of a pole figure is discussed and a method for reducing this defocusing is described.

The rolling of metal powders is briefly discussed and the method used to roll the strip used in the investigation is described.

It was found that it was desirable to prepare the pole figure specimen so that its surface is as flat and smooth as possible, preferably by a polish-etch procedure, in order to prevent spurious intensity recordings.

Examination of the microstructures of the various sheets indicated that the larger the particle size, the

greater the deformation of the particles in the green strip.

ACKNOWLEDGEMENTS

The author would like to express his gratitude to the P. R. Mallory Company of Indianapolis, Indiana, for supplying the material and apparatus used to produce the strip for this investigation and to Mr. Allen G. Wehr, Mallory Research Fellow, who graciously gave his time to prepare the samples. The author is also grateful to various professors in the Metallurgical and Ceramic Engineering Departments for their help and encouragement.

The author is indebted to Dr. D. S. Eppelsheimer, Professor of Metallurgical Engineering, for his encouragement, assistance, and support during the course of this investigation.

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I. INTRODUCTION

In recent years, much interest in the rolling of sheet from metal powders has been developed. This interest has precipitated for a number of reasons, such as a reduction in the number of rolling and annealing operations to produce this strip, the production of strip composed of non-alloyable metals for special properties, the production of sheet from high-melting point powders, and the freedom from preferred orientation of the finished sheet.

The purpose of this investigation was to determine if a tendency toward preferred orientation existed in metal sheet rolled from various powders at the Mallory plant at Indianapolis, Indiana, and to determine if such a tendency could be attributed to the powder characteristics or to the method of rolling, as well as to the usual mechanisms attributed to the formation of preferred orientations. The specimens used were examined in the "as rolled" and "as sintered" conditions, with no further rolling after the initial forming by rolling having been done.

Preferred orientation is objectionable in sheet that is to be used in deep drawing applications and other applications where isotropic physical properties are desired.

Although it is generally accepted in the literature that there is no preferred orientation in sheet rolled from metal powder, there is no report of a systematic investigation of the effect that particle characteristics and rolling variables may have in producing texture in sheet produced by this method.

II. REVIEW OF THE LITERATURE

Sheet has been rolled from many different metal powders, most of the published work being done on copper and nickel powders. Various studies on the effect of roll variables and powder characteristics on the mechanical properties of the finished product have been made, as well as experimentation with methods of rolling the powders.

In 1953, J. Bach[1] reported on the rolling of copper and nickel powders at Sylvania, but did not mention any studies of preferred orientation. P. E. Evans, and G. C. Smith[2] reported (1954) the effects of rolling and sintering variables on physical properties, but made no preferred orientation studies.

In his presentation at the Tenth Annual Meeting of the Metal Powder Association, W. D. Jones[3] reviewed the history of metal rolling and described the basic principles involved, but made no mention of preferred orientation or texture. However, in the discussion following the presentation, Dr. H. H. Hausner (Sylvania Electric Products, Inc., Bayside, N. Y.), reported that rolling metal powders to a density of about 90% or more does not result in any preferred orientation.

Storckheim, Nylin, and Sprissler[4] of Sylvania reported (1955) practically no preferred orientation for a powder-rolled specimen of 18-8 stainless steel which was re-rolled five times. Worn and Perks[5], (1959) report that pure nickel strip rolled by them possessed a substantially random crystal orientation.

Evans and Smith[6] (1959) report no marked orientation before or after sintering in copper strip rolled with a roll gap of zero (0) mm, but that after a 75% reduction by cold rolling, a high degree of preferred orientation exists in the center of the strip. This preferred orientation disappeared after an anneal at 450°C for one hour in vacuum.

In their review of the rolling of sheet from metal powders, Nasser and Zirm[7] state "A noteworthy point which may have technical importance in some cases is the fact that, in contrast to ordinary sheets, strip made by powder metallurgy shows hardly any rolling texture."

III. THE ROLLING OF METAL POWDERS

A. General Methods Used. There are various modifications in the apparatus used to roll metal powders, but the equipment basically consists of (1) a feed hopper to provide a constant feed of powder to the rolls, (2) the rolls, arranged with their axes in the same horizontal or vertical plane, or, more rarely, at some inclined plane, (3) a guide to carry the strip as it emerges from the rolls, (4) sintering furnaces, and furnaces for annealing after cold reductions, (5) cold mills, for further densification of the strip after sintering.

Generally, three cold rollings and annealings are sufficient to give the strip near-theoretical density. Hot rolling may be used and will reduce the number of rollings necessary for densification.

The size of rolling equipment necessary for producing strip of a given thickness from powder is considerably larger than the equipment for producing strip of the same thickness by conventional rolling processes. The strip thickness to roll diameter varies, but may be as high as (1:300) for some powders.

Bimetallic strip may be produced by using one metal strip as a carrier for the powder of the other metal.

B. Method Used For This Investigation. A schematic drawing of the apparatus used to roll the material used in this investigation is shown in Figure 1. The eight inch diameter rolls are arranged with their axes in the same horizontal plane. The hopper feeds the metal powder into the rolls and acts as a powder reservoir. The retaining side sheets fit closely to the rolls and serve to regulate the width of the strip.

Since only relatively small amounts of powder were used in rolling most of the samples it was not practical to use the hopper to feed the powder into the rolls. Instead, with the rolls stopped, cotton was stuffed into the roll gap to prevent loss of the powder, which was then poured into the roll gap in sufficient amount and rolled. The incoherent, fragile edges of the green strip were trimmed off.

Only one sample was obtained which was rolled using the hopper as a powder feed. This was Mallory 3000, a tungsten base alloy.

None of the samples were further rolled to increase their density after initial forming or sintering.

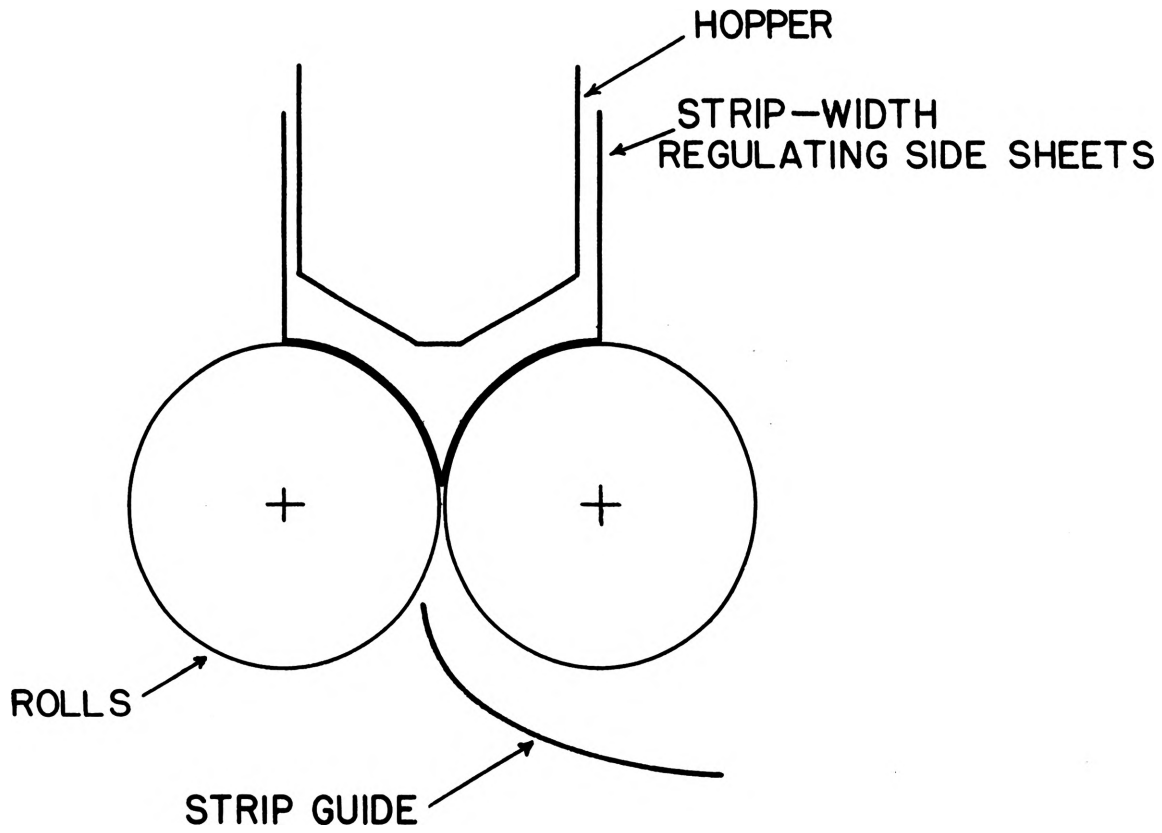


FIG.—1

SCHEMATIC OF POWDER ROLLING APPARATUS

IV. X-RAY TECHNIQUE FOR DETERMINATION OF PREFERRED ORIENTATION

A. The Stereographic Projection. The most satisfactory method for showing preferred orientation in polycrystalline material is by the use of the stereographic projection to obtain pole figures. The relationships existing between orientation in crystalline aggregates and their stereographic projection, and the methods of obtaining pole figures are explained in Barrett[8] and Cullity[9].

The method used in this investigation is a modification of that described by Schultz[10]. The elements composing the stereographic projection and its arrangement used in this investigation are shown in Figure 2. The axes NS, WE, and AB are mutually perpendicular. The plane whose position or orientation is to be defined is located at the center C of the reference sphere. A normal to the plane is constructed, extending from the center of the sphere to its intersection with the surface of the sphere at point P. This normal is called the pole of the plane.

Any circle formed on the surface of the sphere by the intersection of a plane passing through the center of the sphere is called a great circle. Two great circles in

Figure 2 are NWSE and AEBW. If the plane does not pass through the center of the sphere, then the resulting circle is known as a small circle.

The point P may be located on the surface of the sphere by a method similar to that used to locate points on the surface of the earth. By measuring the angles α and ϕ along the surface of the sphere to point P (see insert of Figure 2) the position or orientation of the plane with respect to the reference point (or plane) from which α and ϕ are measured is defined. The angle of "rotation", α , (longitude), is measured from the great circle NWSE along a circle on the surface of the sphere formed by the intersection on the sphere of any plane parallel to the plane of the circle AEBW. The "tilting" angle ϕ (latitude), is measured from the point N along any great circle (meridian) formed by a plane passing through the line NCS.

To transfer this three-dimensional information to a two dimensional flat sheet, a plane known as the projection plane is constructed tangent to the reference sphere at N, and perpendicular to NCS. Point S is made the point of projection, from which a straight line is made to pass through the point P, extending until it intersects the projection plane at point P'. The point

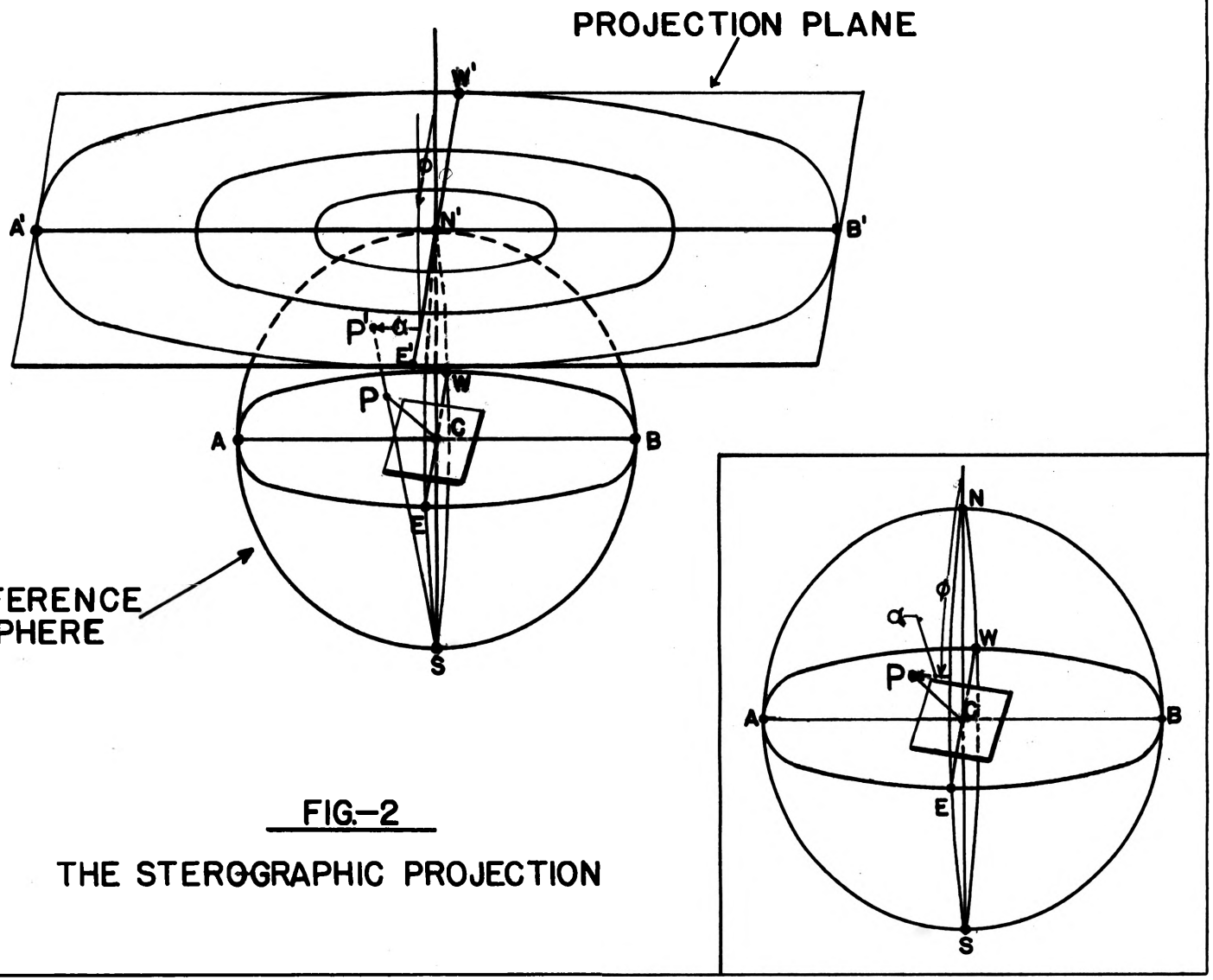


FIG.–2

THE STEREOGRAPHIC PROJECTION

P' on the projection plane may be called the "pole" of the plane, and describes the orientation of the plane with respect to any of the other "standard" or fixed points on the reference sphere and their projections, that is, points N, A, E, B, W, and their projections N', A', E', B', and W'.

The stereographic projection as applied to the germetry of the apparatus used in this investigation is illustrated in Figure 3. This illustration shows the relations between the stereographic projection, incident and diffracted X-ray beams, and sample position.

In plotting a pole figure of polycrystalline material, the poles of only one (hkl) set of planes are used. The position of the X-ray counter is held constant (θ=constant) so that only the Bragg diffractions from the particular (hkl) plane being studied will be recorded. Each pole will show the orientation of one grain in the specimen, since all the (hkl) planes in each grain will have the same orientation. All of the grains in the sheet are considered to lie in the center of the reference sphere. Only reflections from those (hkl) planes lying in the plane AEBW will be recorded by the counter, and the number of these planes will determine the intensity of

the diffracted beam. For the sample position shown ($\alpha = 0°$, $\phi = 0°$), only those planes parallel with the surface of the sheet would be recorded, their poles corresponding to the sheet normal. Their projection would be N'. In order to record planes of other orientations in the sheet, they must be brought into the plane AEBW by rotation of the specimen about the axes Y (ϕ rotation) and X (α rotation). It should be noticed that the α and ϕ rotations required to bring a plane into recording position are made in opposite directions from the α and ϕ measurements made on the pole figure to locate the plane.

In the case of rolled material, the rolling direction (R.D.) is generally taken as the reference direction, and the surface of the sheet is described as lying in plane AEBW at the center of the reference sphere, parallel to the projection plane. The corresponding reference points on the sphere and their projections are the intersection of the specimen normal with the surface of the sphere (point N), its projection (point N'), and the intersection (B) of the line of rolling direction with the sphere, its projection being B'. The rolling direction is usually indicated on the pole figure by an arrow as shown. Each

point or pole plotted on the projection plane then shows the orientation of a plane (hkl) with respect to the sheet surface and rolling direction. The plot of many such planes (the plot of the intensities of their diffracted X-ray beams) is known as a pole figure and gives a quantitative picture of the orientation of a large number of grains in the surface of the sample.

The stereographic polar net used to plot the inner 60° portion of pole figures using this method is shown in Figure 4.

In practice, the points at which intensity readings of fixed magnitude (e. g. 10, 20, 30, etc.) occur are plotted on the pole figure. These points are then connected to give the picture or "map" (pole figure) of the crystallographic orientation of grains in the surface of the sample.

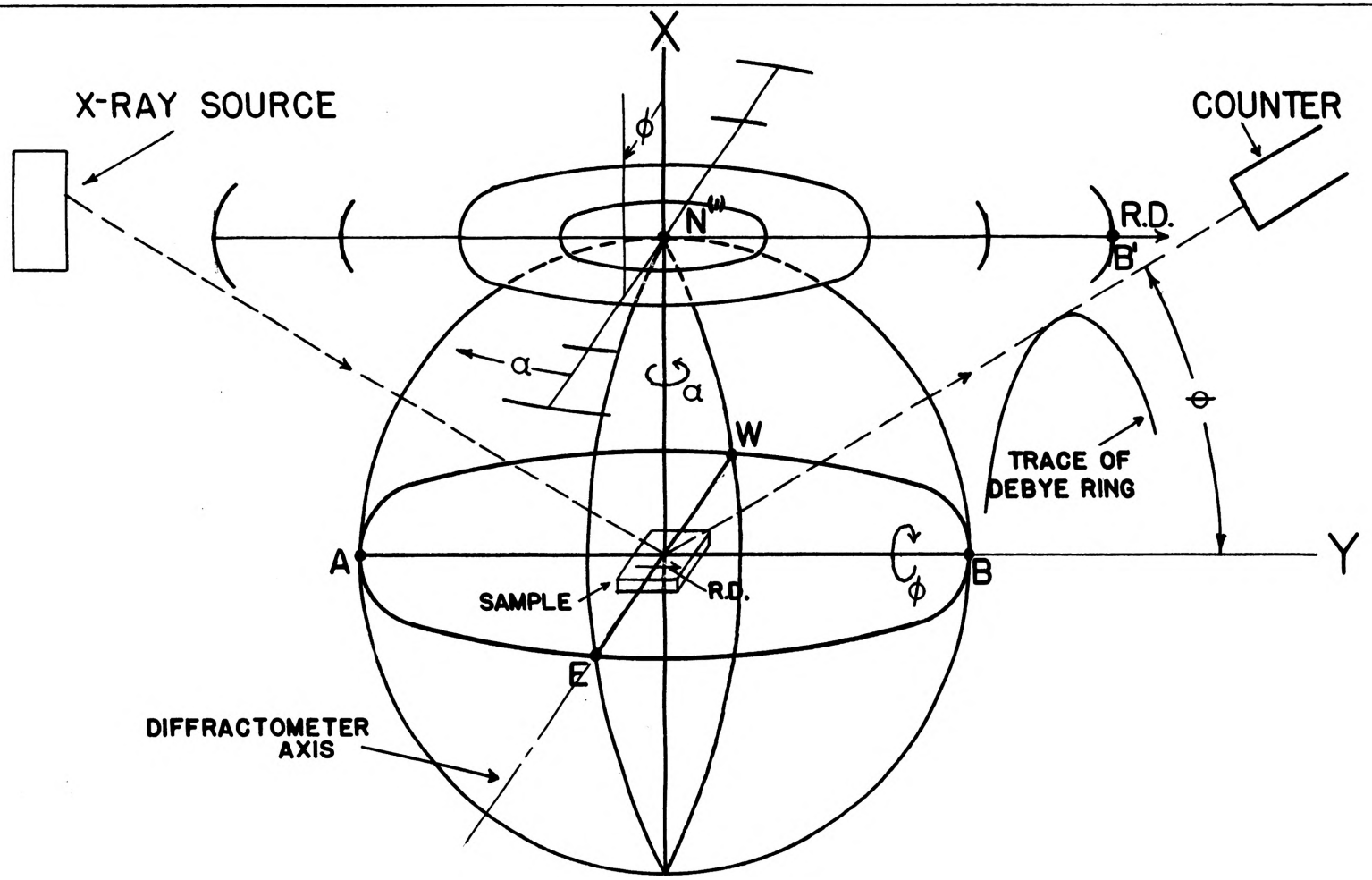


FIG.—3

RELATIONSHIP BETWEEN SAMPLE, DIFFRACTED BEAM, AND STEROGRAPHIC PROJECTION

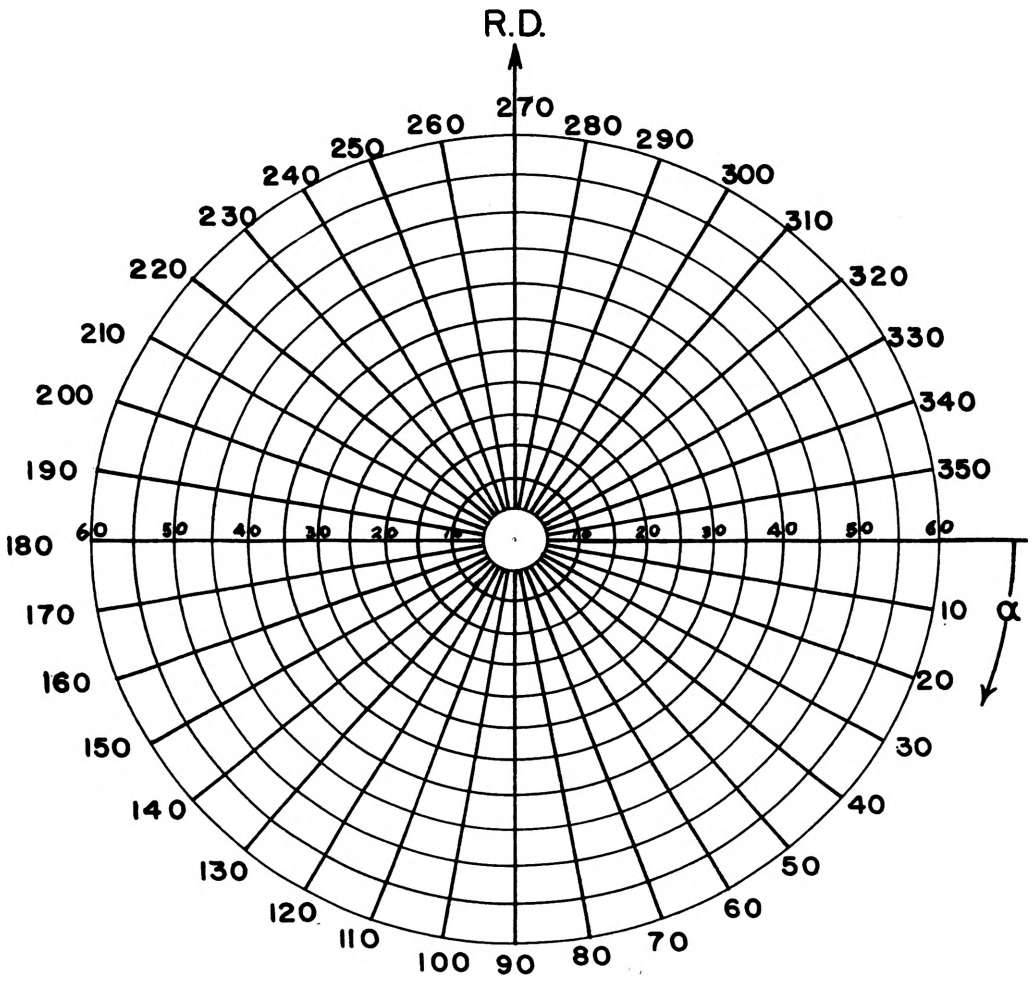


FIG.—4

POLAR NET

B. The Norelco Pole Figure Fixture. The pole figure fixture used in this investigation was developed by the Philips Electronic Division of the North American Philips Company. The fixture is designed to replace the standard specimen holder of the Norelco Diffractometer, and the two may be quickly interchanged.

A drawing of the fixture is shown in Figure 5. The X, Y, and diffractometer axes correspond to those of Figure 3 and show the indentity of geometry of the two figures.

The frame consists of an outer ring and shaft for mounting in the diffractometer. An inner ring rotating within the frame provides ϕ rotation. The specimen table rotates within its own plane to provide α rotation. Both of these rotations may be individually coupled by flexible drive shafts with motors in an external cabinet. The tilting angle, ϕ, may be continuously varied in either direction around Y at the rate of 1° per minute, while α may be varied in one direction around X at either 10° or 20° per minute. The specimen table also is fitted with a drive that provides oscillation in its own plane at the rate of 1° per minute. The oscillation may be adjusted to fit the size of the sample with interchangeable cams providing oscillation strokes of 1/16", 1/4", or 3/8". The fixture

is provided with three interchangeable vertical slits with widths of 0.010, 0.020, and 0.040 inch.

Other accessories include the sample height adjustment bar, shown in place by the dotted lines in Figure 5, and a removable knob for raising and lowering the specimen stage, shown in place with the specimen alignment bar in Figure 10. The specimen stage is locked in elevation with a set screw. Specimens up to 5/16" thick and up to about 1 1/4" in diameter or diagonal may be accommodated on the fixture.

The Norelco Pole Figure Fixture is shown separately in Figure 6 and mounted, ready for operation, in Figure 7.

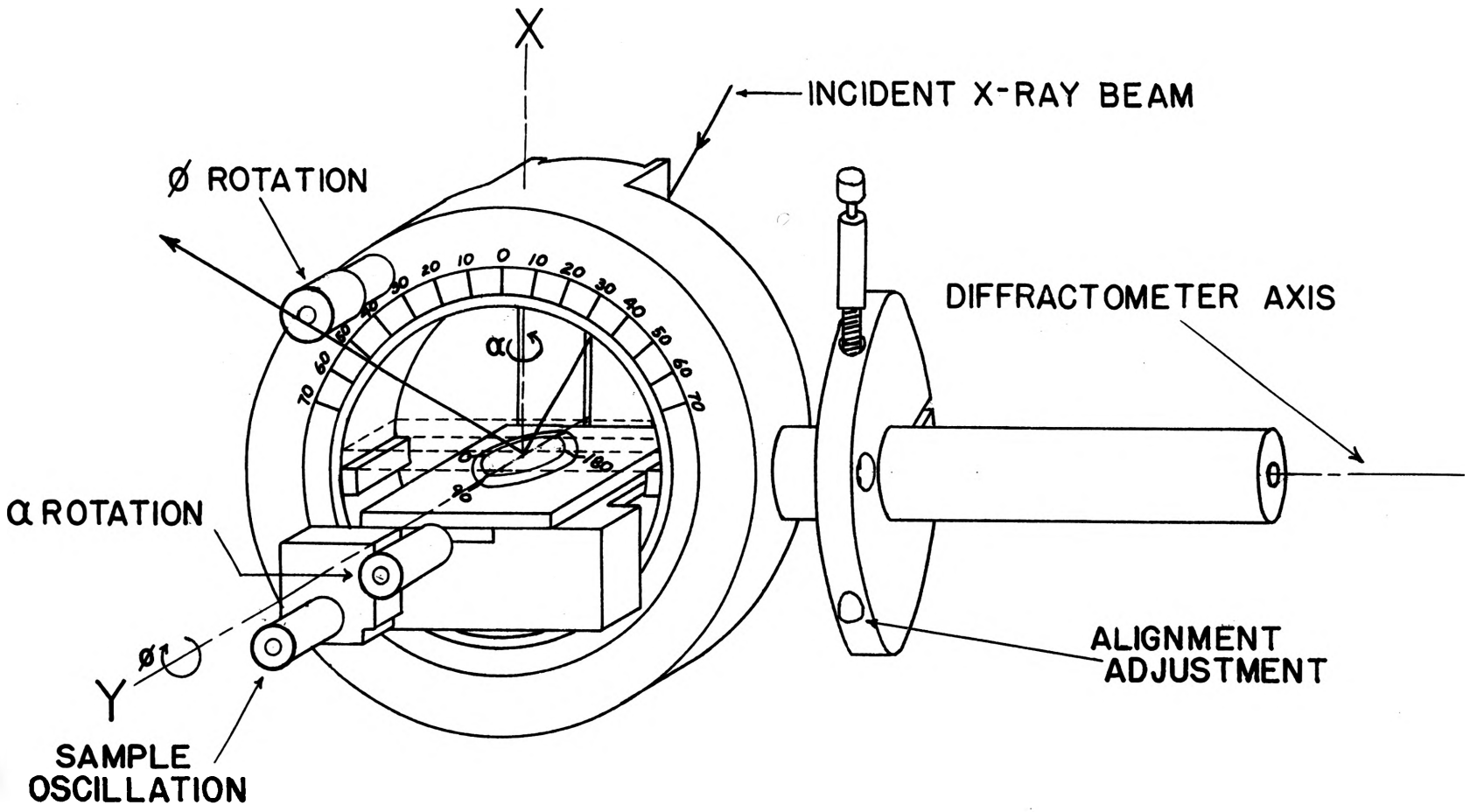


FIG.—5

NORELCO POLE FIGURE FIXTURE

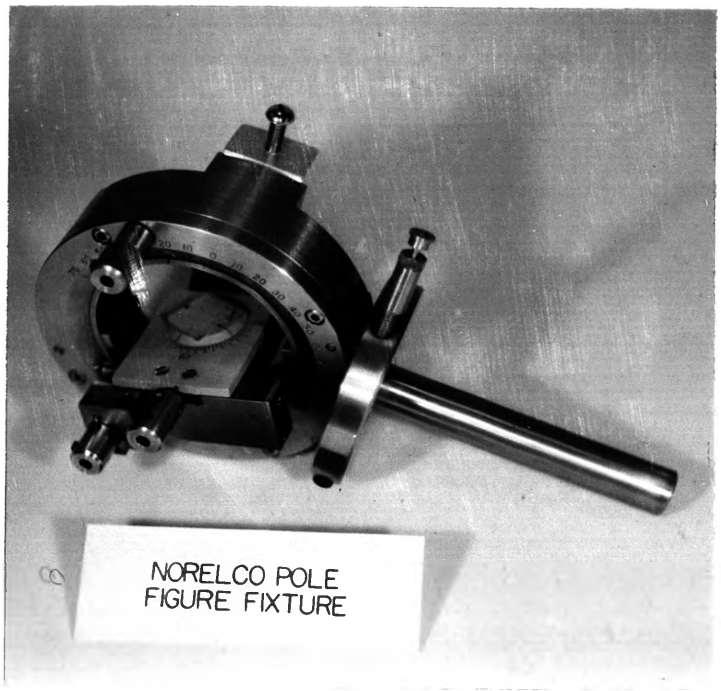
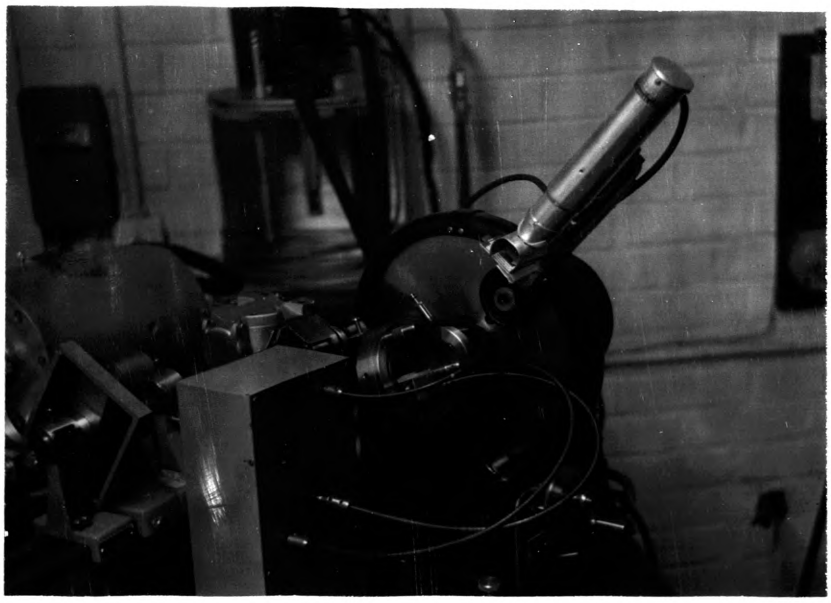


FIG.—6

NORELCO POLE
FIGURE FIXTURE



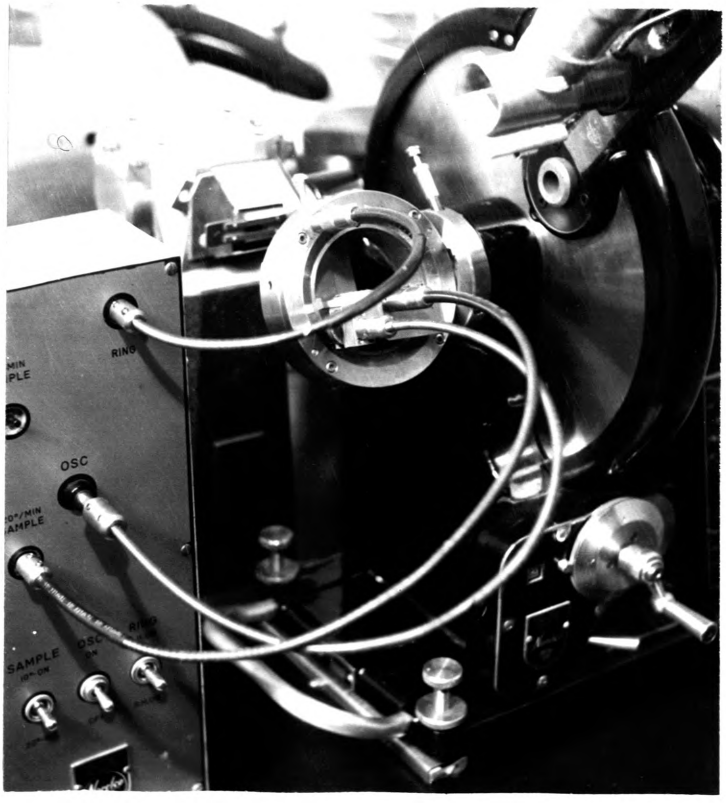


FIG.—7

MOUNTED NORELCO POLE

FIGURE FIXTURE

C. Alignment Of The Pole Figure Fixture. The procedure for alignment of the pole figure fixture for optimum X-ray optics is similar to that for the standard specimen holder as described in the Norelco Instruction and Operating Manual.[11]

The diffractometer is first aligned following the procedure outlined in the manual, using the standard specimen holder. With the diffractometer correctly aligned, the pole figure fixture is then interchanged with the standard specimen holder and aligned by the following procedure:

1. Set the tilting angle, ϕ, at zero.

2. With the fixture slits removed and the sample height adjustment bar in place, raise the specimen stage until the two just touch, and tighten the locking screw. This will place the surface of the stage on the axis of the diffractometer.

3. Loosen the knurled knob at the rear of the diffractometer and the clamp screw (not shown in Figure 5.) located in the fixture collar.

4. Set the diffractometer at the approximate 2θ position.

5. With only one half of the narrowest slit assembly in place, and holding the diffractometer aligning bar flat

on the specimen stage against this slit half, adjust the position of the fixture on the diffractometer axis until the edge of the diffractometer aligning bar touching the half-slit bisects both parallel slit assemblies (Soller slits). Make sure that in this position the fixture collar is fitting snugly against the diffractometer hub.

6. Tighten the collar clamp screw only.

7. Set the diffractometer at the exact (0°) 2θ position.

8. With the fixture slits removed and the narrowest convergent and receiving slit in place, hold the diffractometer aligning bar flat on the fixture stage and turn the alignment adjustment screw (Figure 5) until the bottom edges of the alignment bar bisect the two slits. The knurled knob at the rear of the diffractometer may now be tightened.

D. Errors In Method. One advantage in the Schultz reflection method is that no tilting angle correction is necessary to compensate for changes in volume of the material being irradiated. However, defocusing does occur as $\emptyset$ is varied, as explained by Chernock and Beck[12], resulting in a decrease in intensity for samples with random crystallographic orientation as $\emptyset$ is varied from 0 toward 90°. At $\emptyset$=90° the incident X-ray beam would be absorbed in the surface of the specimen, since at this position the incident beam and the specimen surface would both lie in the same plane.

The defocusing is due to the fact that as the specimen is tilted ($\emptyset$ rotation), certain parts of the irradiated area of the specimen are moved off the focusing circle described by the source, the specimen surface, and the receiving slit. This defocusing may be considered in two separate parts, illustrated in Figures 8 and 9.

Figure 8A shows the pole figure device slit system as viewed from the direction of the incident X-ray beam. The width of the slit is exaggerated for clarity. The line ACB represents the surface of the specimen at $\emptyset$=0° ($\emptyset_o$),while the dotted line A_1CB_1 represents the specimen surface at an arbitrary tilting angle $\emptyset_1$. The side view,

including the source and receiving slit, is shown in Figure 8B. This view shows that the parts of the surface of the specimen lying at A_1 and B_1 (points in Figure 8A, lines in Figure 8B) will not lie on the required focusing circle and hence the portion of the X-ray beam diffracted from these surfaces will not be recorded at the receiving slit.

This defocusing has the effect of reducing the recorded intensity as the tilting angle is increased. This reduction in intensity would be superimposed on any intensity variation due to preferred orientation. Figure 8C shows the sharp, narrow peak and intensity I_o that would be obtained at $\phi=0°$. Figure 8D shows the relative positions of the peaks diffracted from A_1, C, and B_1 at $\phi=\phi_1$, and the reduced intensity I_1, recorded at 2θ. This decrease in intensity with increasing ϕ may be compensated for by determining the relation between the intensity decrease and ϕ and applying a correction factor. This information is obtained by recording intensity versus ϕ for a randomly oriented powder sample of the material being studied. The correction factor is that necessary to make the intensity at any ϕ value equal to that at $\phi=0°$. The broadening effect on the

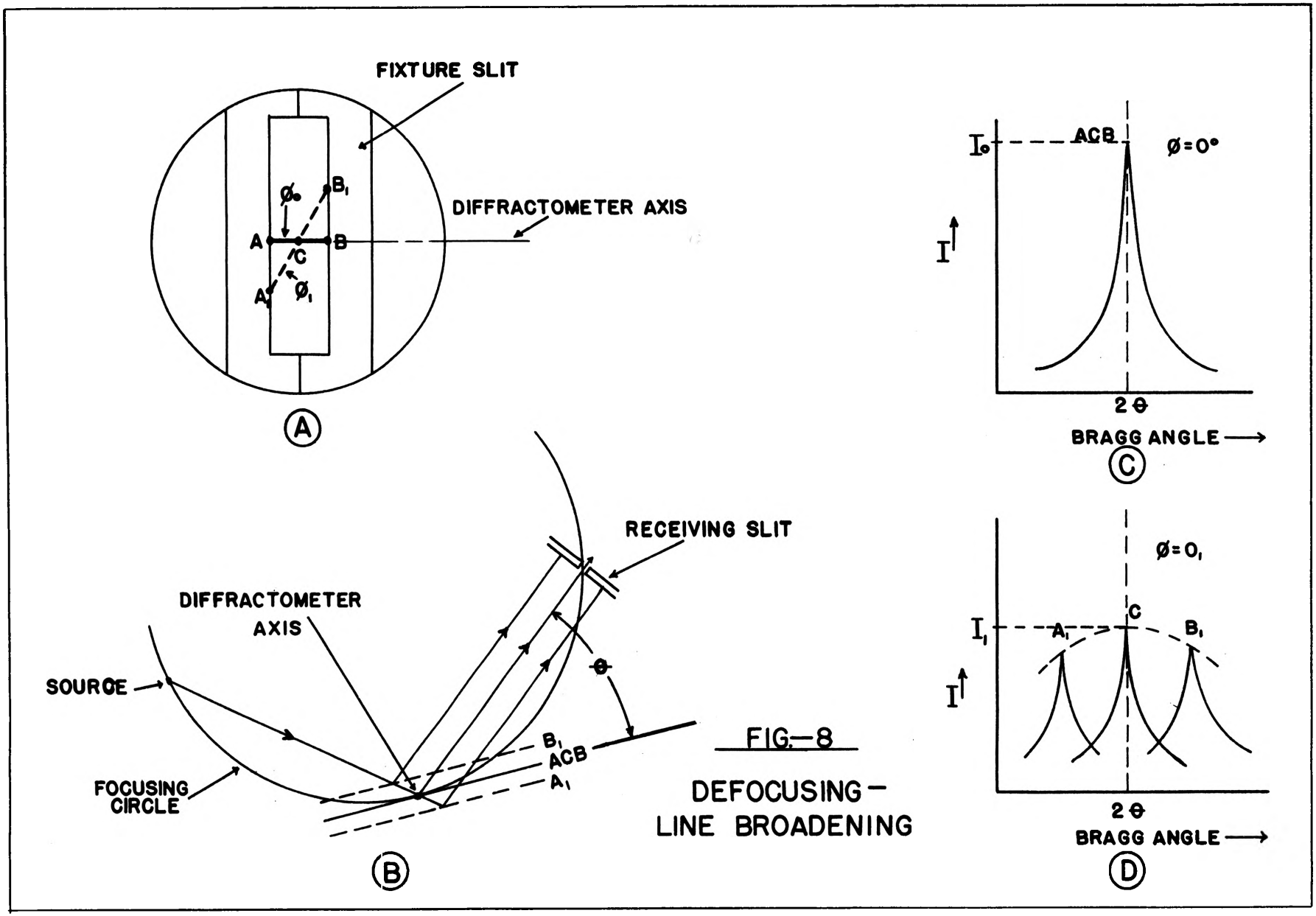


FIG.—8

DEFOCUSING—
LINE BROADENING

composite peak when all points between A_1 and B_1 are considered is shown by the dotted line in Figure 8D.

Two methods of minimizing this effect are (1) to keep the width of the pole figure fixture slit as narrow as possible while retaining sufficient diffraction intensity, and (2) to widen the receiving slit as much as is practicable. Both methods were used in this investigation.

The second defocusing effect is that due to misalignment of the specimen during mounting, which will result in a movement of the diffraction peak. This effect is illustrated in Figure 9. If care is not taken when aligning the specimen, the surface of the irradiated portion of the specimen may not pass through the center (C) of rotation of the inner ring of the pole figure fixture. Also, in this case, the specimen surface would not contain the diffractometer axis, and the diffraction peak would not occur at the true 2θ angle. Although the full intensity of the diffracted beam could be recorded by setting the receiving slit position for the peak, defocusing would occur as ϕ is increased, resulting in a decrease in recorded intensity.

In Figure 9A and Figure 9B, C' represents the center of the irradiated portion of the specimen at $\phi=0°$, and C'_1

represents the center of the irradiated portion at some tilting angle ϕ_1. As the tilting angle is increased from 0°, the relative position of the irradiated surface of the specimen will move further away from the true center (C). Therefore, even though initially the full intensity of the diffracted beam may be recorded by setting the slit for the peak (C in Figure 9C), the intensity I_1 at some angle ϕ_1 will be less because the position of the Bragg reflection will change, as shown in Figure 8D.

Chernock and Beck[12] report that the change in the apparent 2θ value with a change of 45° in ϕ may be as large as 0.5° or as small as 0.03°, depending on the care taken in aligning the specimen. They give a variation of 0.05° in 2θ between the positions $\phi=+45°$, $\phi=0°$, and $\phi=-45°$ as the maximum allowable deviation for specimen alignment.

The best method for minimizing this defocusing error is to carefully align the specimen when mounting it. Increasing the width of the receiving slit will allow more of the widened peak to be recorded, thereby reducing the seriousness of this defocusing effect.

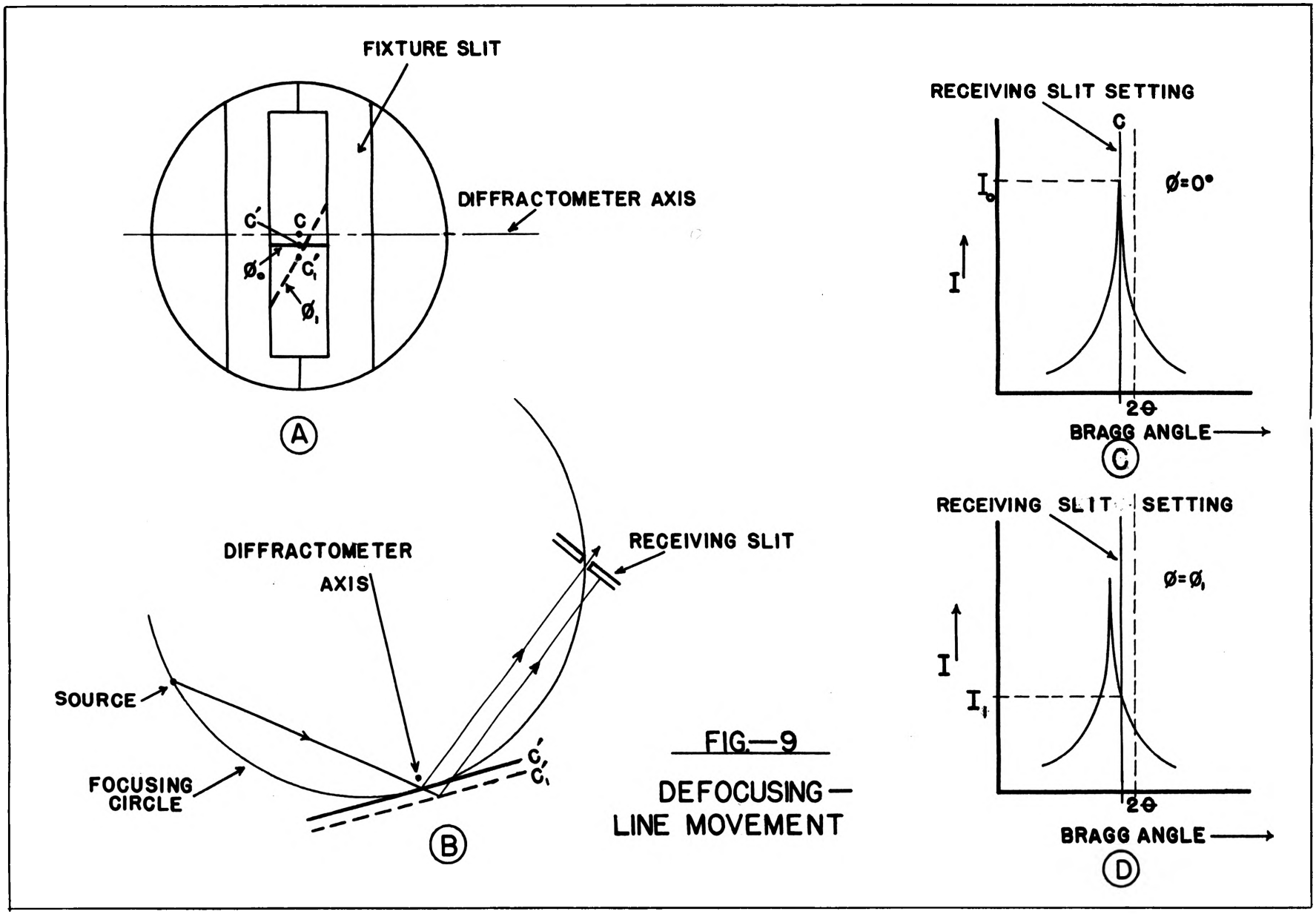


FIG.—9

DEFOCUSING—
LINE MOVEMENT

E. Experimental Procedure. Square specimens were cut from the sheets and mounted on the fixture either "as rolled" or with a determined amount of surface metal removed by a grind-polish-etch or etching-only procedure. The surface to be used must be as flat and smooth and as free from etching products and pits as possible. The samples were mounted in thin bakelite mounts for the grind-etch procedure. These mounts could then be mounted directly on the fixture stage.

The specimens were mounted on the specimen stage with either rubber cement or clay. The latter was found to be satisfactory and was the more rapid method. The sample is mounted so that the rolling and transverse directions coincide with the marks on the specimen stage.

With the fixture slits removed and the sample height adjustment bar firmly held in place, the stage is elevated until the surface of the specimen and bar touch. This alignment is shown in Figure 10. If clay is used to mount the specimen, the stage may be slightly further elevated to position the specimen surface parallel with the stage surface. In any case, at the final setting, care is taken that the stage and specimen touch and that the height adjustment bar rests against its supports. The stage is

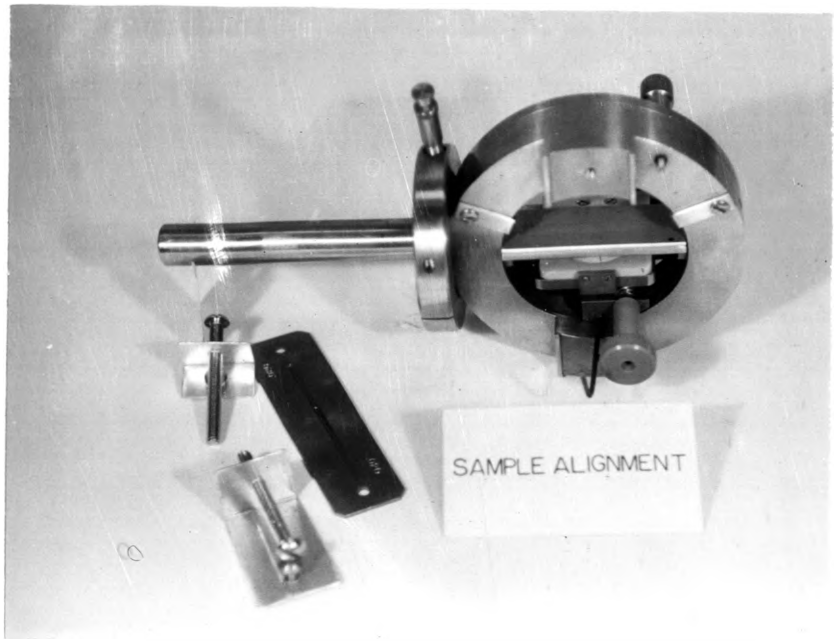


FIG.—10

SAMPLE ALIGNMENT

then locked in elevation with the locking set screw.

The whole incident beam must fall on the specimen at all times. It may be desirable to use a "stop off" mechanism such as the simple screw arrangement shown disassembled in Figure 10, so that the maximum incident-beam length may be obtained for any particular sample size and diffractometer setting. This adjustment is made with a fluorescent screen the size of the sample centered and aligned on the stage. Using a soft X-ray beam, the length of the incident beam is adjusted with the screws until the maximum beam length for any α angle and oscillation position is obtained, keeping in mind that the whole incident beam must fall on the specimen at all positions. The diffractometer must be set at the correct 2θ angle during this adjustment.

After mounting and aligning of the specimen, the specimen surface should contain the diffractometer axis. To check for this condition, 2θ scans with the standard horizontal receiving and scatter slits in place may be made at ϕ values of +45°, 0°, and -45°. The sample is considered correctly aligned if the deviation of the diffraction peak between these three ϕ positions is 0.05° or less. The 2θ value is then set and held constant as

the pole figure is scanned by varying the sample through the angles ϕ and α. This is the method used when peaks adjacent to that being studied are only about three degrees 2θ or less away. A vertical slit (described below) may be used in conjunction with the standard receiving slits to increase α and ϕ resolution if its use will not reduce the recorded intensity by too great an amount.

When adjacent peaks are far enough apart that there is no danger of recording two peaks or parts of two peaks simultaneously at any ϕ setting, as is the case with cubic metals, the width of the horizontal receiving and scatter slits may be increased, and a vertical slit may be used.

Depending on the width of the horizontal slits, the intensity correction factor for tilting angle is reduced, and the precision of sample alignment is not as critical. The maximum slit width would correspond to the effective slit width obtained by using no horizontal slits at all. This was the procedure followed in this investigation. No correction factor was necessary for ϕ values up to 40° and amounted to only about 1.3 at $\phi = 60°$, as compared to a factor of about 4.0 necessary when the standard 0.006" horizontal receiving slit is used.

The procedure followed is essentially the same as that outlined above except that, since the precision of sample alignment is not as critical, if reasonable care is taken in adjusting the sample height, the 2θ scans at Ø values of -45°, 0°, and +45° need not be made. After determining the 2θ value of the diffraction peak for Ø=0°, the horizontal receiving and scatter slits are removed and the vertical slit is mounted, centered as nearly as possible on the Soller slit assembly.

Since there is no provision for mounting vertical slits on the standard Norelco diffractometer, it was necessary to fashion such an accessory. The vertical slit and filter holder were made from aluminum sheet. It is shown disassembled in Figure 11 and assembled in Figure 12. It was designed to fit over the receiving Soller slit housing and could be easily slid on or off. It is shown in place in Figure 7.

The two methods used in this investigation to scan the pole figure were: (1) runs in which α was varied continuously from 0° through 360° at constant Ø values taken at 5 or 10 degree intervals from 0° to 60° Ø, (2) runs in which α and Ø were varied simultaneously to make spiral scans of the pole figure. The first method is the

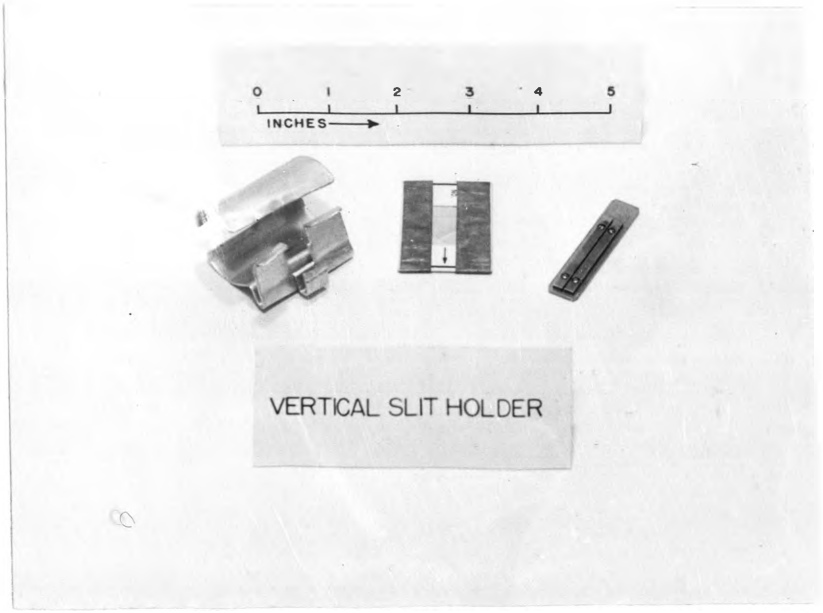


FIG:—11

VERTICAL SLIT HOLDER

DISASSEMBLED

FIG.—12

VERTICAL SLIT HOLDER
ASSEMBLED

slowest but the easiest to plot, and is used for specimens showing any tendency for orientation. The second method is a rapid, satisfactory method to determine if any orientation exists.

V. EXPERIMENTAL RESULTS

The results and information for each material are presented separately. The density measurements were made by mensuration. Dimensions were measured by use of micrometer and vernier calipers; mass was determined by weighing to one milligram.

The specimens were examined "as rolled" and/or with some of the surface material removed to determine the effect, if any, that distance from the surface of the specimen might have in orientation characteristics. Surface material was removed by grind-polish-etch and etch-only procedures. The grind-polish-etch method was found to give the smoothest surface and the best results for pole figure runs.

TABLE I

THICKNESS AND DENSITY CHARACTERISTICS OF STRIP

ROLLED FROM COPPER, NICKEL, AND TITANIUM POWDERS

Strip Material	Strip Designation	Roll Opening in Mils	Green Strip Thickness in Mils		%-Theoretical Density of Green Strip		Sintered Strip Thickness in Mils		%-Theoretical Density of Sintered Strip	
			Edge	Center	Edge	Center	Edge	Center	Edge	Center
Copper	C1	10	16	16	63	45	16	16	95	90
	C2	20	26	26	63	42	26	26	91	58
	C3	5	13	13	77	65	13	13	89	93
Nickel	N1	10	26	26	70	73	24	24	76	70
	N2	20	33	33		75	27	27		63
	N3	5	24	26	70	75	24	26	71	83
Titanium	10 rpm T1	25		26		85		25		85
	10 rpm T2	30		28		83		24		84
	10 tmp T3	35		45		56		42		74
	13 1/3 rpm T1	25		27		83		25		89
	13 1/3 rpm T2	30		29		76		23		90
	13 1/3 rpm T3	35		41		54		41		75

A. Copper Strip.

1. Copper Powder Specifications.

Producer: American Metal Climax, Inc.

Apparent Density: 2.1 to 2.5 gm/cc

Screen Analysis:

+100 mesh	0.1%
-100+150	0.5 max.
-150+200	4.0 max.
-200+250	1.5 max.
-250+325	2-7
-325	90 min.

Chemical Analysis:

Copper	99.00%
Carbon	0.03-0.05
Grease	0.01-0.025
HNO_3 insol.	nil-0.02
Sulfur	0.01-0.018
Arsenic	0.0003-0.002
Antimony	0.0015-0.002
Iron	0.001-0.002
Tin	0.0002-0.0003
Nickel	0.0002-0.0003
Lead	0.00-0.15

2. Discussion of Strip Characteristics and Experimental Results. Three strips were rolled at a roll speed of 10 rpm with three different roll openings. The sample designations, roll openings used, thickness and density of the sintered and green strip are given in Table I.

A photomicrograph of the loose copper powder is shown in Figure 13. The rounded appearance of the particles indicates the powder was produced by atomization.

The strip as rolled and trimmed was about six inches long by two and one-half inches wide. Sintering was at 900°C for one hour on a graphite boat in a cracked ammonia atmosphere.

A representative photo of the green and sintered strip is shown in Figure 14. Frilling of strip edges is evident in the sintered strip, indicating an uneven distribution of powder feed across the width of the strip, which is to be expected from the method used to roll the samples.

Longitudinal and transverse metallographic samples were prepared from across the width of the rolled strip. None of the samples examined showed the marked deformation of powder particles as was reported by Evans and Smith[13].

This was probably due to the fact that the powder used to roll the strip studied in this investigation was of a finer particle size than that used by them.

Transverse and longitudinal sections of the unsintered strip C1, that strip rolled with the narrowest roll opening, are shown in Figure 15. The large void areas are caused by particles that were pulled out during polishing. Visual microscopic examinations made on the sintered strip indicated a decrease in porosity of the strip as the roll gap was decreased. Sample C3 showed a decrease in porosity from center to edge of the strip.

The recrystallized copper strip is shown in Figure 16 in the transverse view of strip C3 which was taken from the edge of the sintered strip.

Spiral pole figure scans of the (111) planes were made of 0.7" square samples taken from the whole width of both sintered and unsintered strip rolled at each of the three roll settings. To determine if distance below the surface of the sample could make any difference in orientation, each sample was examined (1) as rolled, (2) with about 0.001 inch of surface removed, and (3) with about 0.005 inch of surface material removed. Material was removed by polish-etch and etch-only procedures. The

etchant used was 50% nitric acid. The polish-etch method was found to give the smoothest surface and the best results for pole figure runs. It was found that the etching-only method produced a heavily pitted surface, especially when 0.005 inch of material was removed. This pitted surface caused spurious variations in intensity recordings for some samples. The spurious intensity variations disappeared when these heavily pitted samples were polished and lightly etched.

None of the thirty copper specimens examined showed any preferred orientation.

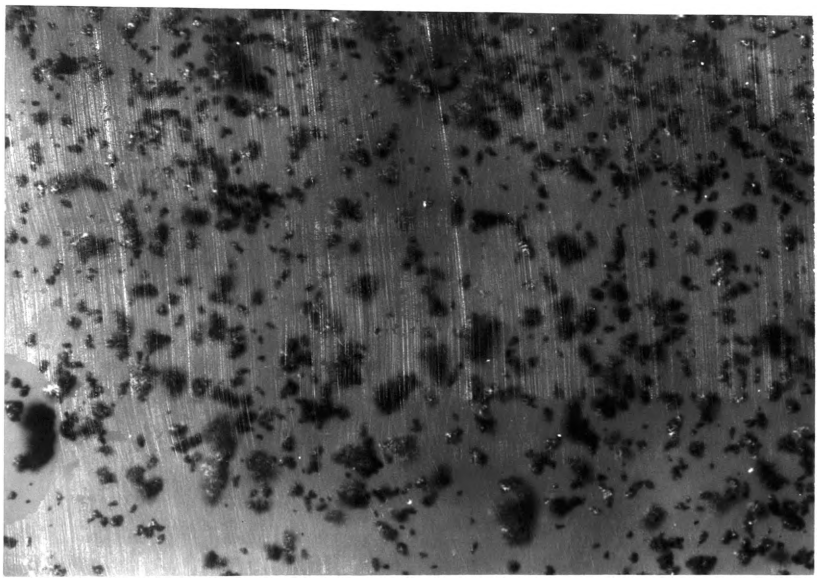


FIG.—13

COPPER POWDER

100 X

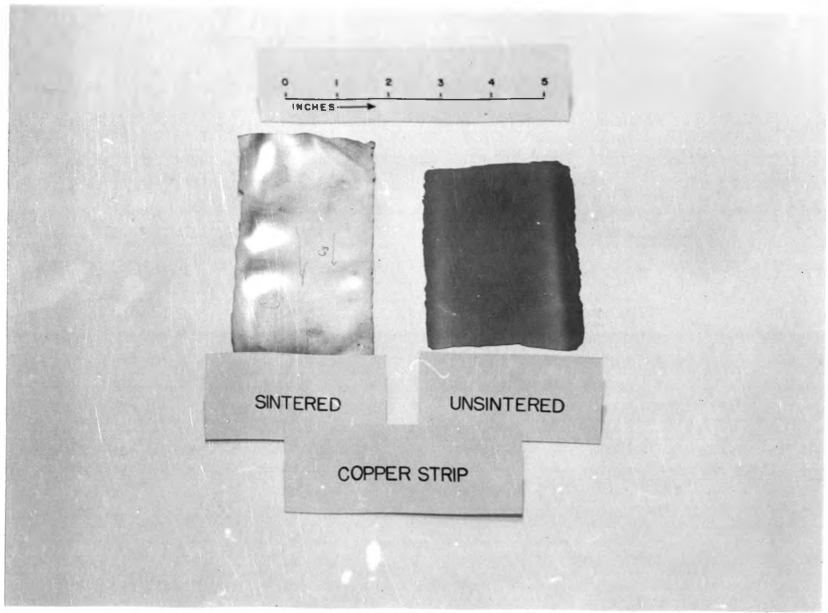
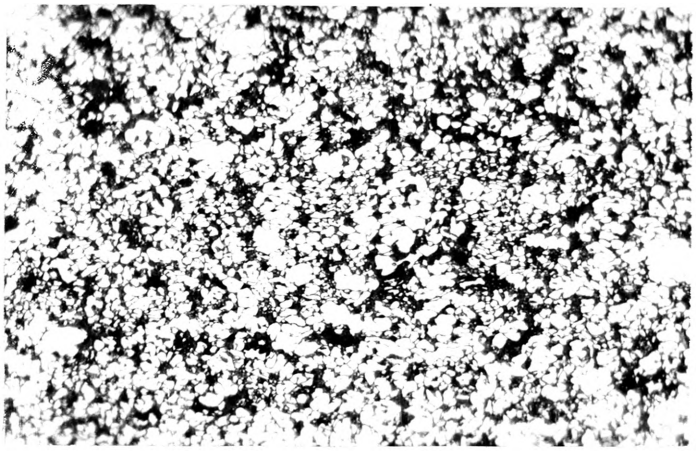
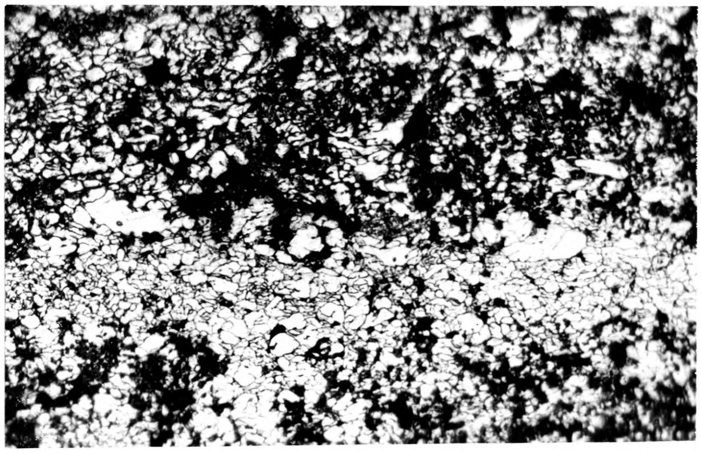


FIG.—14

ROLLED COPPER STRIP



TRANSVERSE



LONGITUDINAL

FIG.—15

GREEN COPPER STRIP MICROSTRUCTURES

250 X

ETCHANT—50% NITRIC ACID

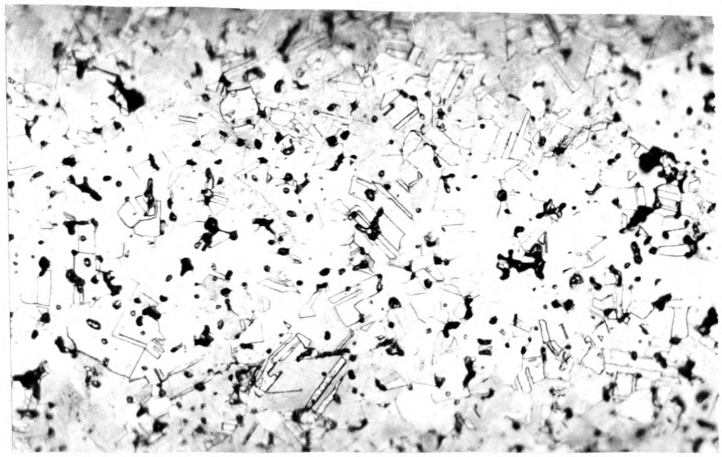


FIG.—16

SINTERED COPPER STRIP MICROSTRUCTURE

TRANSVERSE 250X

ETCHANT—50% NITRIC ACID

B. Nickel Strip.

1. Nickel Powder Specifications.

Producer: Sherritt Gordon Mines Limited

Grade: F100

Method of Production: Atomized

Fisher Average Particle Size: 20 microns

Porosity: 0.578

Apparent Density: 56.435% of theoretical

Screen Analysis:

+150 mesh	0.6%
+200	5.4
+270	5.0
+325	4.4
-325	84.6

Chemical Analysis:

Not known

2. Discussion of Strip Characteristics and Experimental Results. Strips were rolled at a roll speed of 10 rpm at three different roll openings. The strip data is given in Table I.

Specimen N1 was sintered at 1000°C for one hour, one piece being resintered at 1300°C for one-half hour. Specimen N2 was sintered at 1100°C for one hour, and specimen N3 was sintered at 1300°C for one half hour. All sintering was done in a cracked ammonia atmosphere.

A photomicrograph of the loose powder is shown in Figure 17. The rounded appearance of the powder indicates its method of manufacture was apparently by atomization. The strip as rolled and trimmed was about five inches in length by three and one-half inches wide. Representative samples are shown in Figure 18. Frilling of the edges of the sintered strip was not so evident in the nickel strip as in the copper strip.

Metallographic studies of samples taken across the width of the nickel strip failed to reveal any marked elongation or flattening of grains. Representative microstructures of the unsintered strip are shown in Figure 19. These samples were taken from near the center of the strip rolled with the smallest roll opening.

The recrystallized sintered strip is shown in Figure 20. The microstructure of all three strips showed a fairly uniform amount of porosity across the width of the strip. There was a marked reduction in porosity as the roll gap was decreased, estimated from the microstructure of the unetched sintered specimens as varying from 50% maximum porosity for the widest roll gap to 30% maximum for the narrowest roll gap.

Pole figure scans were made of 0.7" square samples taken from the width of both sintered and unsintered strip rolled at each of the three roll openings. All of these specimens were examined with about 0.001 inch of surface material removed by the polish-etch procedure, using an etch of 50% nitric acid. Of the twenty-two runs made, none showed any tendency toward preferred orientation.

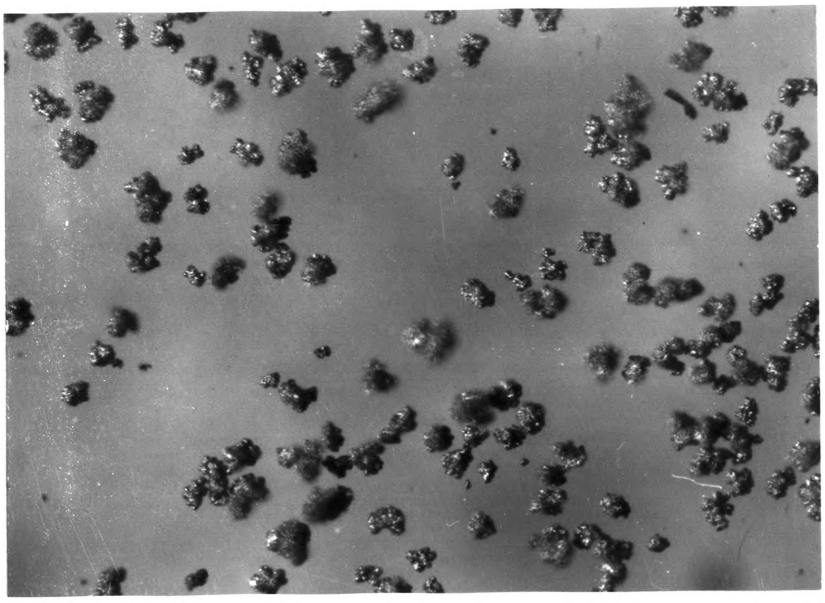


FIG.—17

NICKEL POWDER

100X

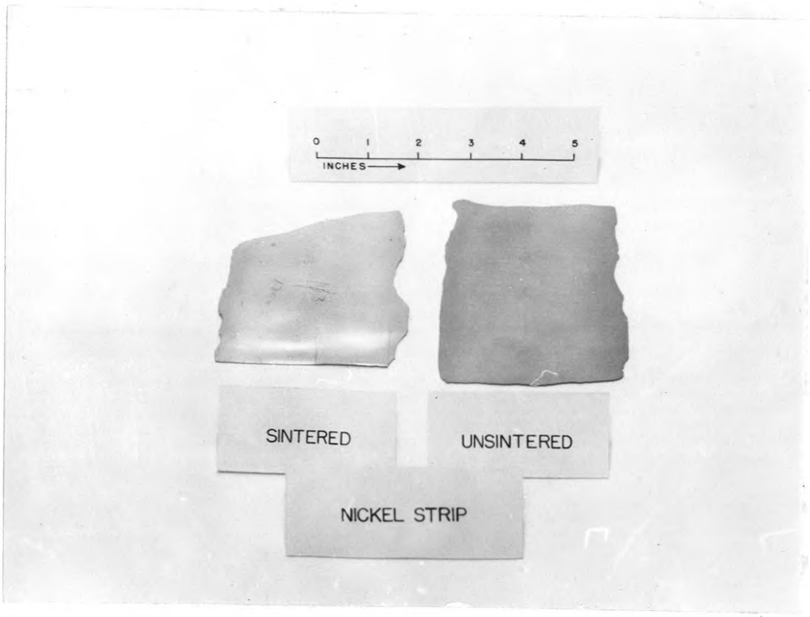
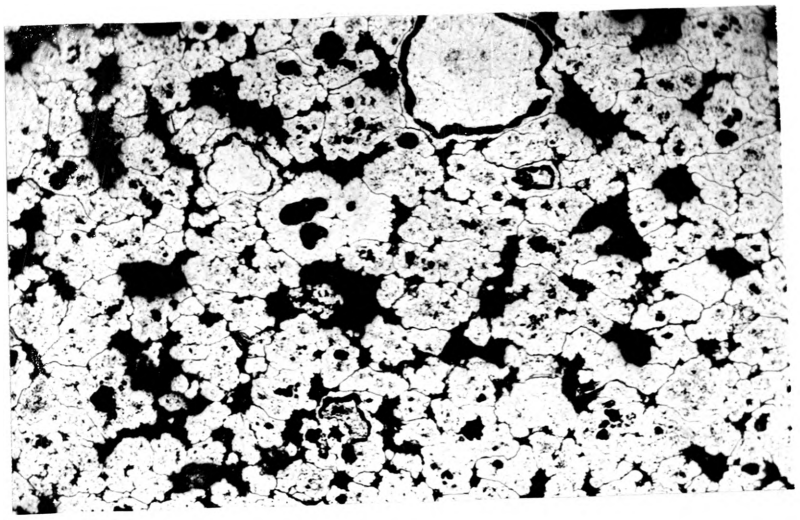
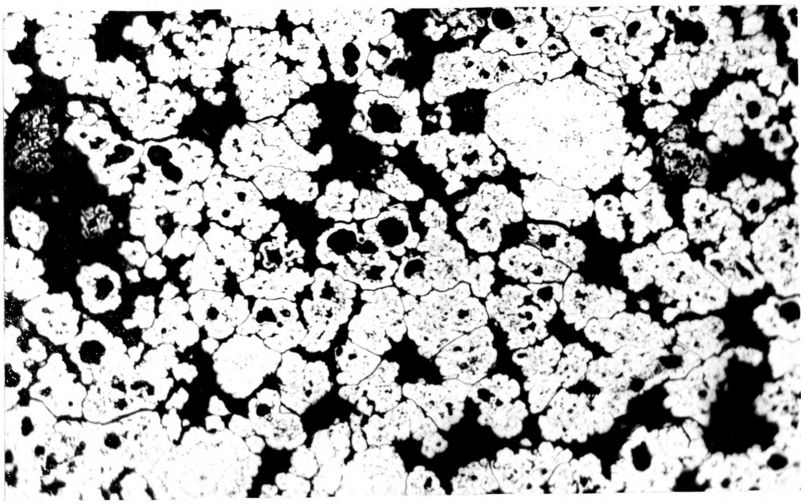


FIG.—18

ROLLED NICKEL STRIP



TRANSVERSE



LONGITUDINAL

FIG.—19

GREEN NICKEL STRIP MICROSTRUCTURES

250 X

ETCHANT-50% NITRIC ACID

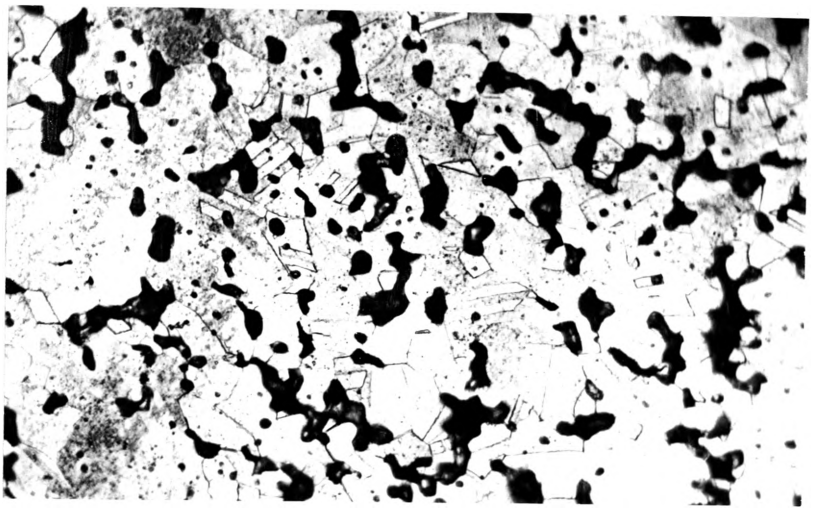


FIG.—20

SINTERED NICKEL STRIP MICROSTRUCTURE

TRANSVERSE 250 X

ETCHANT - 50% NITRIC ACID

C. Titanium Strip.

1. Titanium Powder Specifications

Producer: Union Carbide Metals

Method of Production: Mg reduction of TiO_2

Screen Analysis:

+200 mesh	6%
+325	50%
-325	40%

Chemical Analysis:

Commercial Grade Purity

2. Discussion of Strip Characteristics and Experimental Results. The loose powder is shown in Figure 21. Three roll openings were used to roll the titanium powder, and two different speeds, 10 rpm and 13 1/3 rpm, were used at each roll setting. The strip data is given in Table I.

The titanium strip as rolled and trimmed was about one inch wide by 3 1/2 inches long. Sintering was done at 1350 to 1400°C for one hour in a vacuum.

A representative photo of green and sintered strip is shown in Figure 22.

Roll speed made no apparent difference in the microstructure of strip rolled at the same roll opening. Porosity versus roll opening as estimated from the microstructure of the sintered strip was as follows: 0.025 inch, 20% porosity; 0.030 inch, 30% porosity; and 0.035 inch, 50% porosity.

The particles of the green titanium strip exhibited greater cohesion than did the particles of the green strip rolled from the other metals in that the tendency to break away from the strip during polishing was less. This was probably due at least in part to the larger particle size of the titanium powder.

The microstructures of the green titanium strip showed some deformation of grains, especially in that strip rolled at the narrowest roll opening. This is shown in the microstructures in Figure 23 of the strip rolled at 13 1/3 rpm with a roll opening of 0.025 inch.

The recrystallized microstructure shown in Figure 24 is that of the strip rolled at 10 rpm roll speed and 0.030 inch roll opening. This microstructure is typical of that of all the sintered strip.

Spiral pole figure scans of the (011) planes were made of 0.7" square specimens taken from each of the green and sintered specimens. The metallographic and X-ray specimens were prepared by grinding and polishing followed with an etch of the following composition: 60 cc glycerine, 20 cc hydrofluoric acid, 20 cc nitric acid. The six sintered specimens were also examined as rolled. None of the eighteen samples showed any tendency toward preferred orientation.

FIG.—21

TITANIUM POWDER

100 X

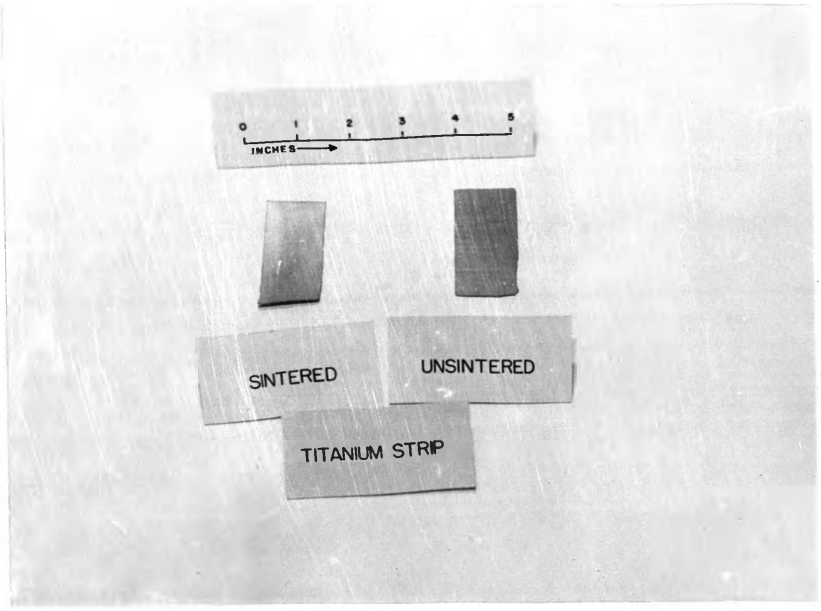
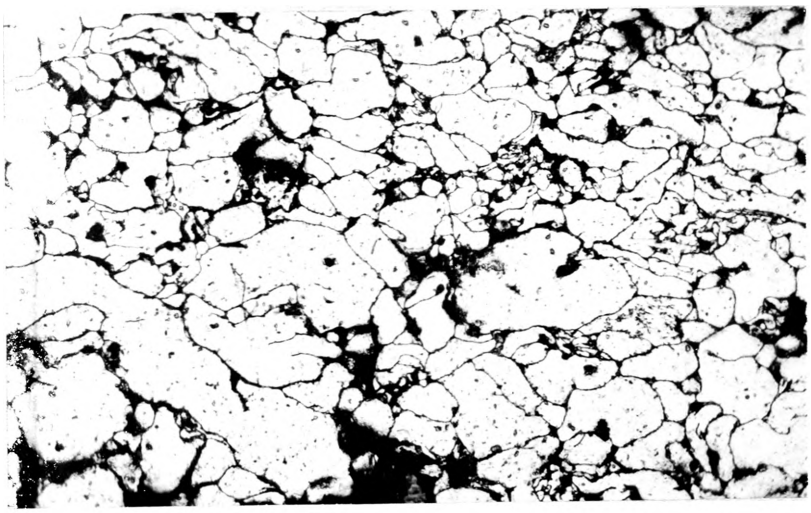
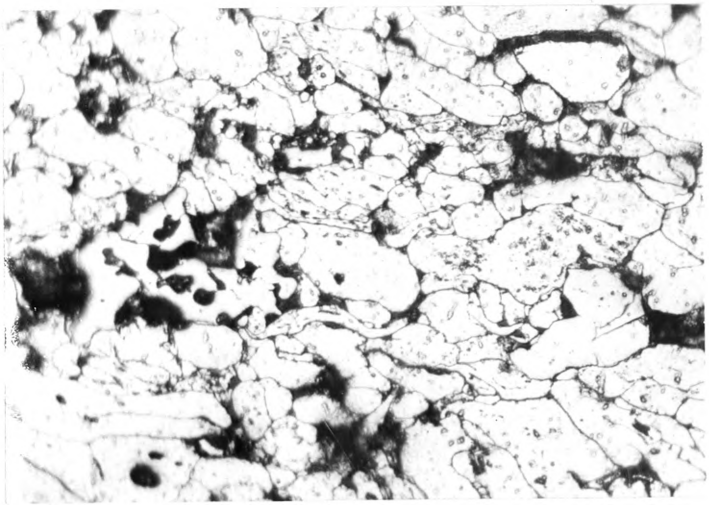


FIG.—22

ROLLED TITANIUM STRIP



TRANSVERSE



LONGITUDINAL

FIG.—23

GREEN TITANIUM STRIP MICROSTRUCTURES

250 X

ETCHANT: 60 CC GLYCERINE

20 CC NITRIC ACID

20 CC HYDROFLUORIC ACID

FIG.—24

SINTERED TITANIUM STRIP MICROSTRUCTURE

TRANSVERSE 250 X

ETCHANT: 60 CC GLYCERINE

20 CC NITRIC ACID

20 CC HYDROFLUORIC ACID

D. Tungsten Strip.

1. Tungsten Powder Specifications.

Producer: Produced at Mallory by hydrogen reduction from General Electric WO_3.

Chemical Composition: 99%+ Tungsten, 0.25% Nickel added to improve green strength and sintering behavior.

Screen Analysis: Unknown, no loose powder available.

2. Discussion of Strip Characteristics and Experimental Results. Only one strip each of green and sintered sheet was rolled. Roll opening was zero (0) inch as read on the roll gage, and roll speed was 10 rpm. Sintering was for one hour at 1500°C in a cracked ammonia atmosphere. The green strip is shown in Figure 25.

The sintered and green strip were both very fragile. The thickness of the sintered strip was about 18 mils and that of the green strip about 19 mils.

The microstructure of the green strip is shown in Figure 26, and that of the sintered strip in Figure 27. The green strip shows a great amount of void area caused by particles which were pulled out during polishing. This indicates the fragility of the green strip.

Spiral pole figure scans of the (110) planes were made of the sintered and green strip, (1) with the surface as rolled, and (2) with about 0.001 inch of surface material removed. Two samples were taken from each strip, one from the center portion and one from the edge. None of the samples showed any preferred orientation.

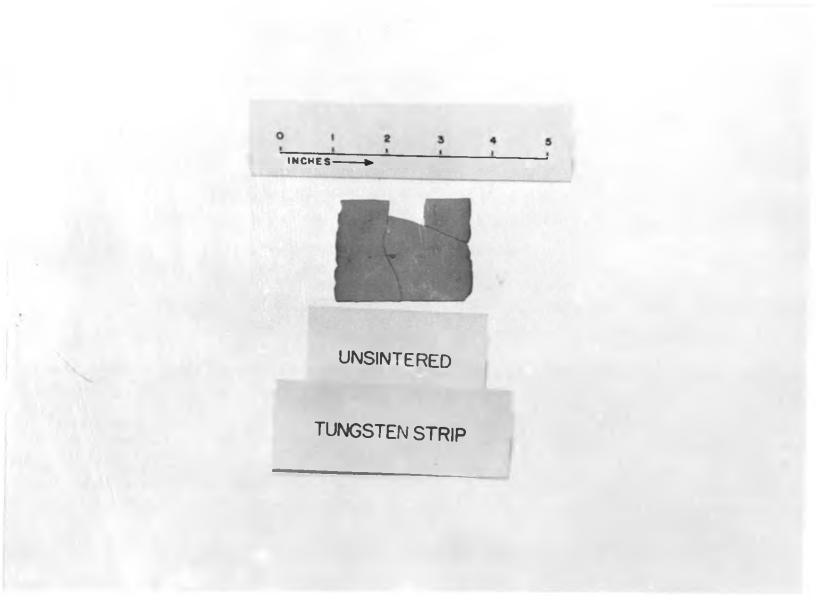


FIG.—25

ROLLED TUNGSTEN STRIP

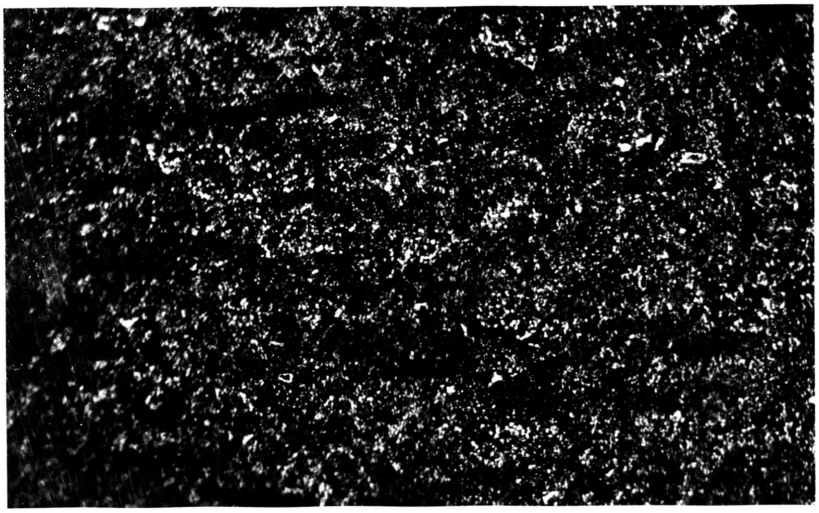


FIG.—26

GREEN TUNGSTEN STRIP MICROSTRUCTURE

TRANSVERSE 250 X

ETCHANT: 3% BOILING SOLUTION

OF HYDROGEN PEROXIDE

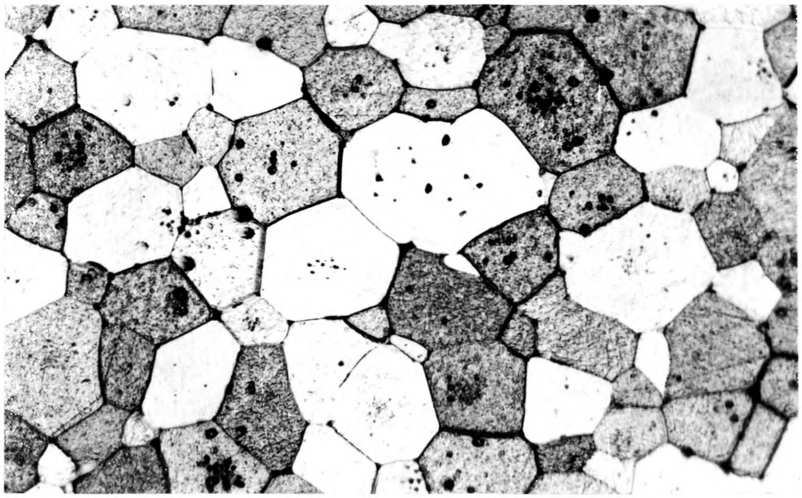


FIG.–27

SINTERED TUNGSTEN STRIP MICROSTRUCTURE

ROLLED SURFACE 250 X

ETCHANT: 3% BOILING SOLUTION

OF HYDROGEN PEROXIDE

E. Mallory 3000 Strip. A sample of commercially produced Mallory 3000 green strip was also available for study. Its analysis was as follows: 90% tungsten, 7% nickel, and 3% iron. It was rolled in rolls with a diameter of six inches with a roll opening of 0.006 inch at a roll speed of 13 1/3 rpm.

No loose powder or powder specifications were available, except for the Fisher average particle size, which was 9.6 microns. The strip which was supplied was about twenty inches long by five inches wide. The strip was very fragile and required care in handling. A photograph of the strip is shown in Figure 28.

The thickness across the width of the strip was uniform at 30 mils. The density measured across the width of the strip varied from 69% of the theoretical in the center to 73% at the edge, as determined by mensuration.

Metallographic preparation was extremely difficult because of the ease with which the particles were dislodged. A photomicrograph is shown in Figure 29. This is a longitudinal section taken from near the center of the strip.

Spiral pole figure scans of the (110) planes were made of one-inch square samples taken across the width of

the strip. The samples were examined as rolled and with about 0.001 inch of surface material removed. None of the samples showed any preferred orientation.

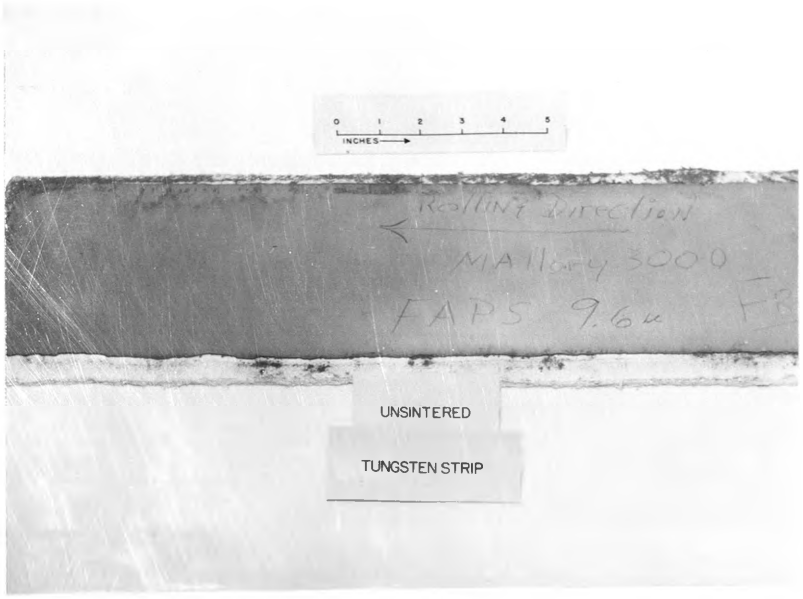


FIG.—28

GREEN MALLORY 3000 STRIP

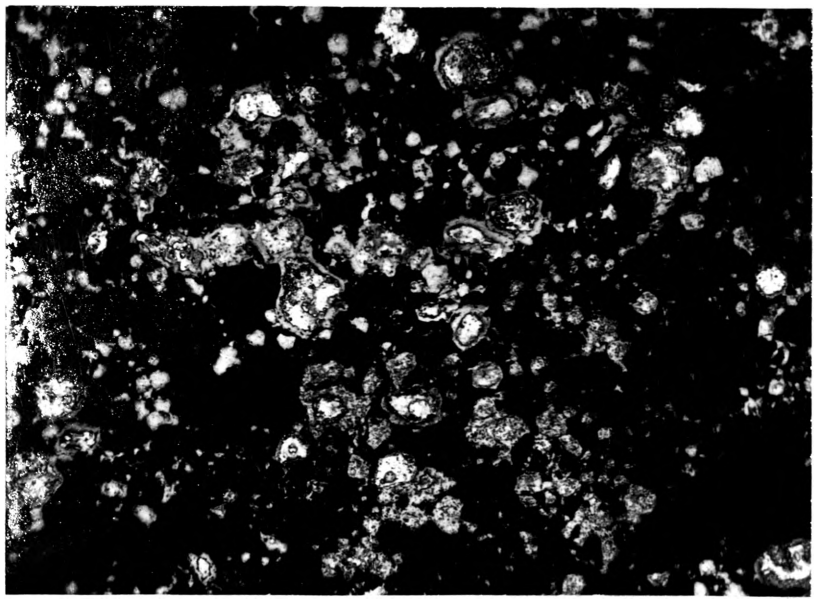


FIG.—29

GREEN MALLORY 3000 STRIP

LONGITUDINAL 250 X

ETCHANT: 3% BOILING SOLUTION

OF HYDROGEN PEROXIDE

VI. CONCLUSIONS

1) Use of a vertical receiving slit in place of the standard horizontal receiving and scatter slits on the Norelco diffractometer greatly reduces the defocusing encountered as the tilting angle is increased from $\phi = 0°$. The use of the vertical receiving slit also eliminated the need for extremely critical sample alignment since any small amount of variation in alignment would not cause the diffracted peak position to move out of the relatively wide (in the vertical direction) receiving slit opening.

2) Although the amount of deformation of powder particles during rolling is probably mainly dependent, all other factors being equal, on the ductility of the metal being rolled, there is some indication from the microstructures of the green strip examined in this investigation that the size of the powder particles rolled will also play an important role. The titanium powder had a comparatively larger particle size with 56% of +325 mesh particles, and in spite of its lesser ductility showed more deformation of its particles than did the nickel and copper strip, and at a larger roll opening.

3) The general effect of decreasing the roll gap used to roll metal powders is to produce a strip of decreasing porosity, and to impart more deformation to the powder particles.

4) The deformation or working of metal powder during the initial forming-by-rolling operation is much less than the amount necessary to produce preferred orientation.

5) Particle shape, at least for the material studied in this investigation, apparently contributes nothing toward the production of preferred orientation of sheet rolled from metal powder. Even if there was a tendency for alignment of particles according to their shape, there would be the further requirement that crystallographic orientation within individual grains would have to be related to particle shape.

6) Preferred orientation in sheet rolled from metal powder is highly unlikely, barring the introduction of some other influencing factor not considered in this investigation, such as a magnetic field applied during the rolling of magnetic powder particles.

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VITA

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