Feb. 17, 1953 J. H. RAMAGE ET AL 2,628,926

MANUFACTURE OF MACHINEABLE MOLYBDENUM

Filed June 21, 1949 2 SHEETS—SHEET 1

Fig. 1.

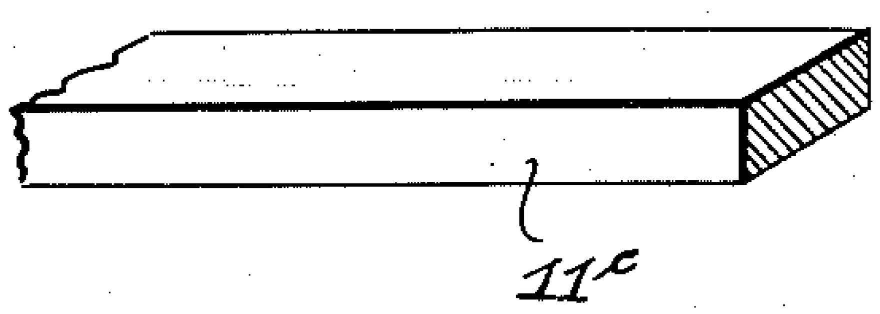


Fig. 2.

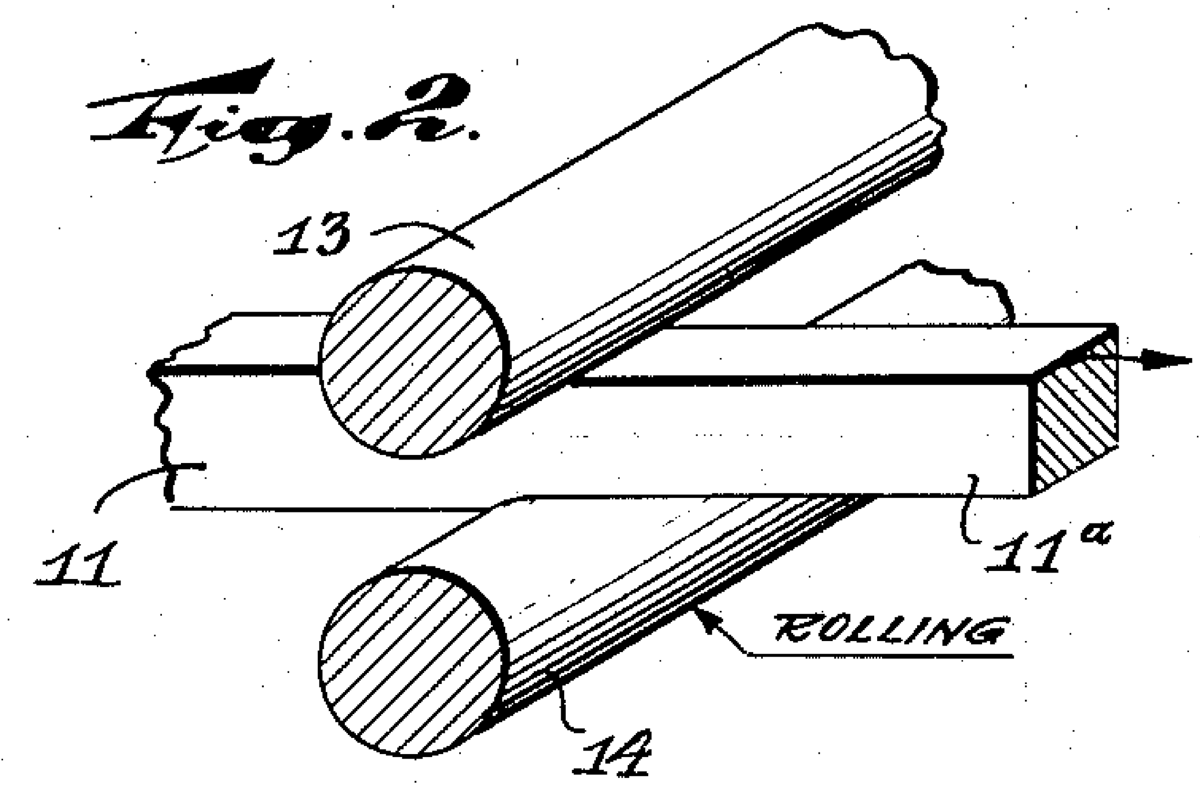


Fig. 3.

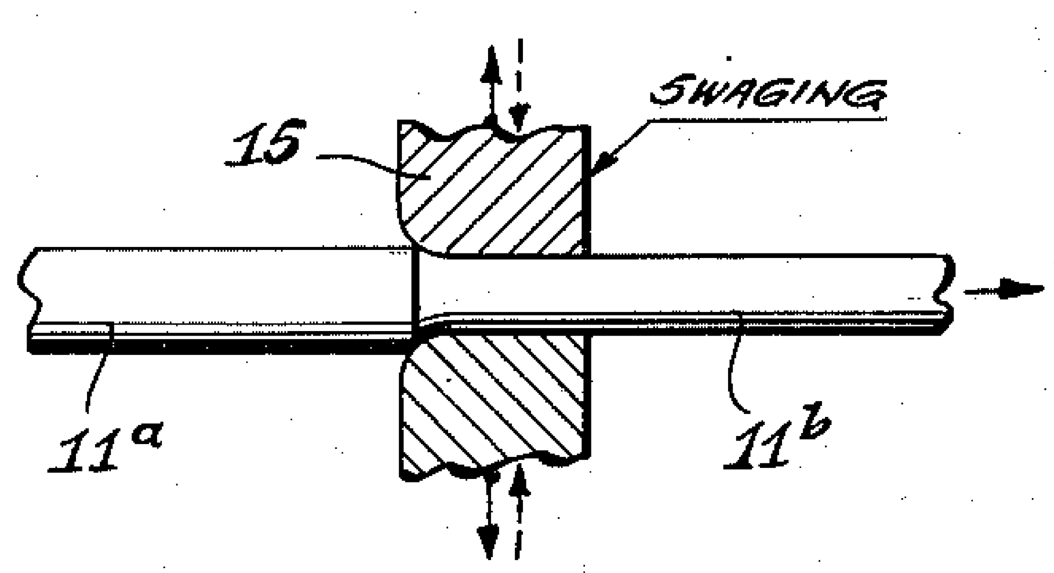
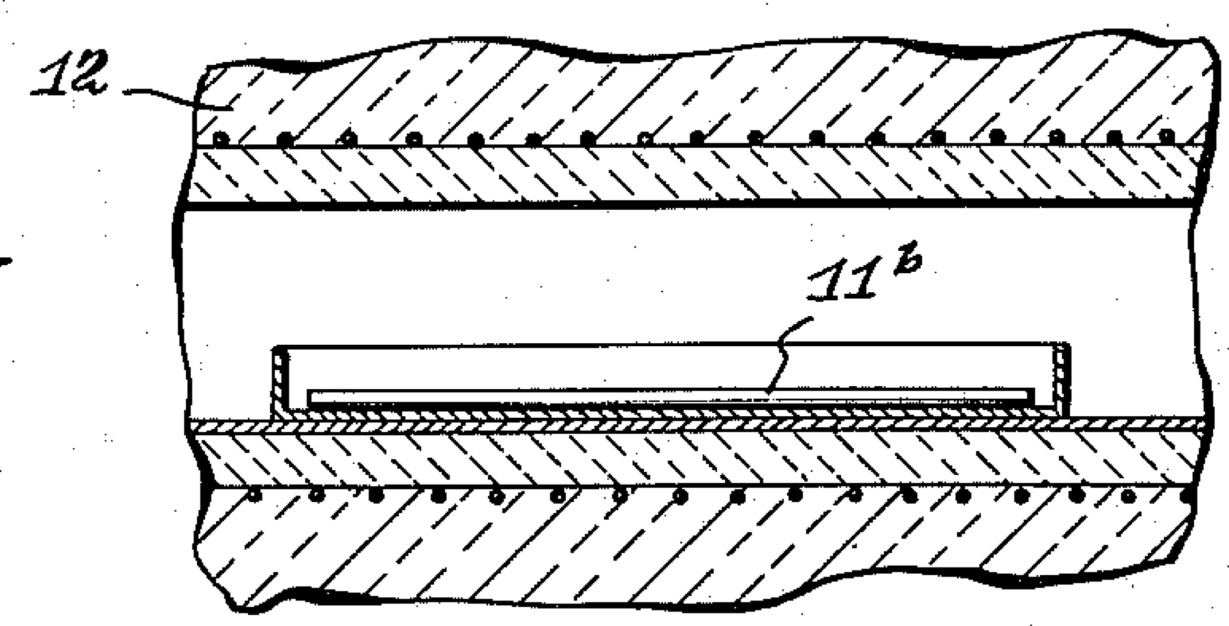


Fig. 4.



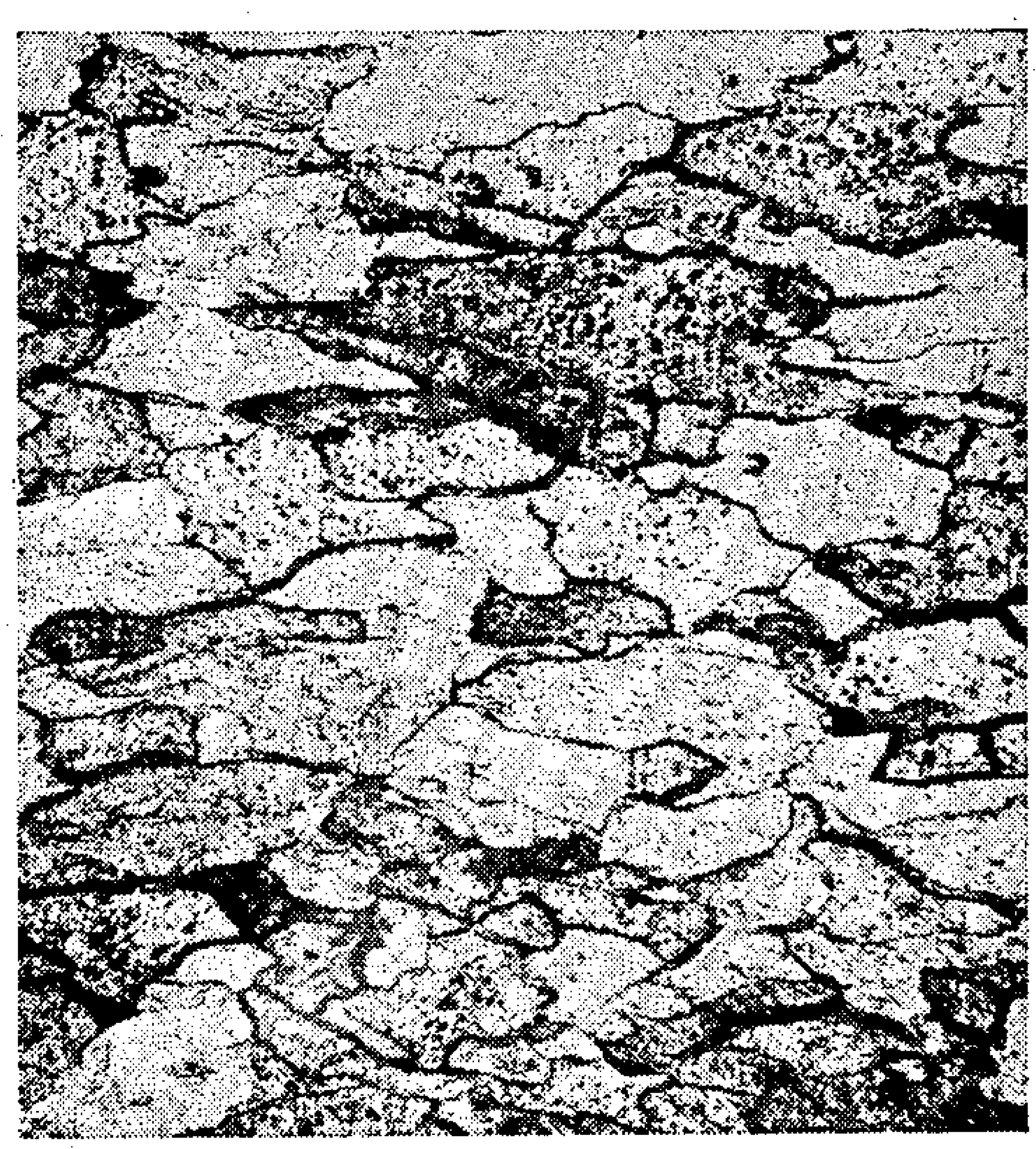


Fig. 5.

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MANUFACTURE OF MACHINEABLE MOLYBDENUM

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Fig. 6.

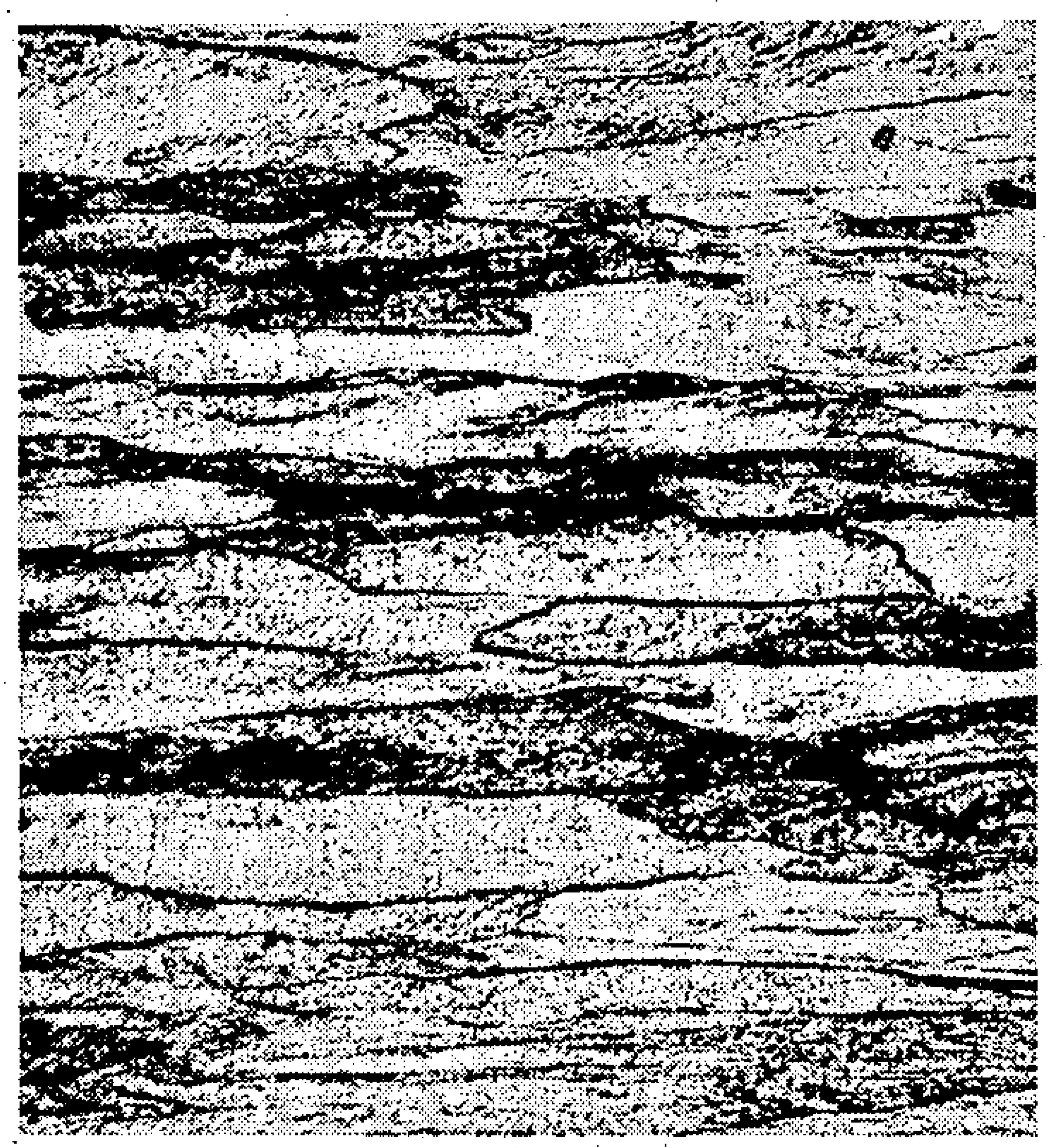
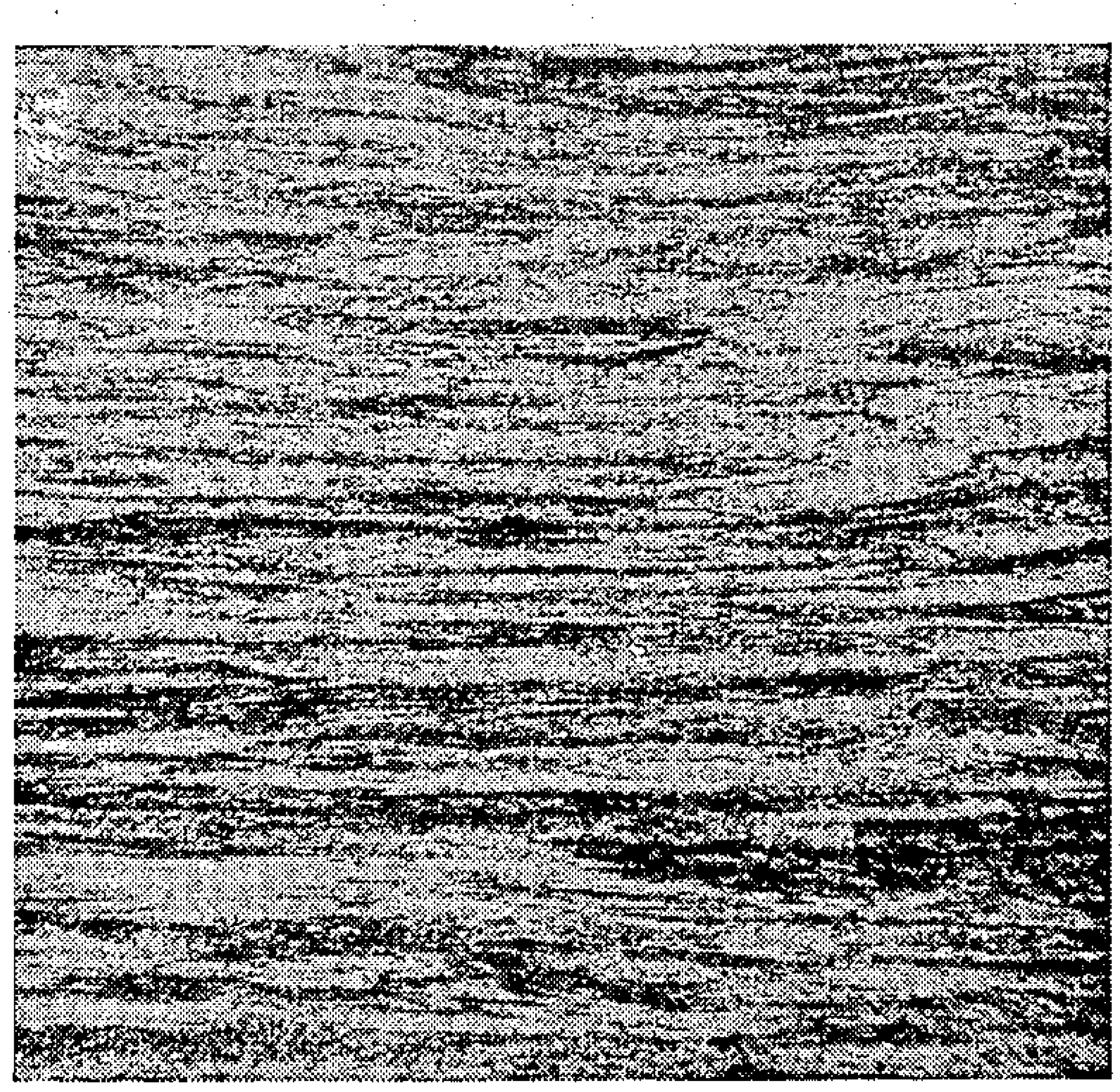


Fig. 7.



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2,628,926

MANUFACTURE OF MACHINABLE MOLYBDENUM

John H. Ramage, deceased, late of Bloomfield, N. J., by The Bloomfield Bank and Trust Co., executor, Bloomfield, N. J., and Robert D. Malin, Park Forest, Ill., assignors to Westinghouse Electric Corporation, East Pittsburgh, Pa., a corporation of Pennsylvania

Application June 21, 1949, Serial No. 100,392

10 Claims. (Cl. 148—11.5)

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This application is a continuation in part of the application, Serial No. 722,306, filed January 16, 1947, now abandoned.

This invention relates to molybdenum and, more particularly, to such that is easily machineable.

The principal object of our invention, generally considered, is to produce molybdenum in which, on an average, the length of each grain is no less than two and no more than four, or at the utmost for certain purposes, six times the width or diameter, whereby the material is easily machineable and, at the same time, the crystal cohesion is good and the material not too weak.

Another object of our invention is to produce molybdenum in which the grains are at least slightly elongated beyond a one to one ratio between lengths and widths, say on the average, to no less than two and generally no more than four times their transverse dimensions.

A further object of our invention is to produce molybdenum in which the grain size is between 10 and 250 per square millimeter, while the grain length is increased from an unworked or recrystallized condition, of a one to one ratio between the length and transverse dimensions, to avoid poor crystal cohesion and weak material, to not less than a grain length of two, and generally not more than a grain length of four, times the transverse dimension, thereby also avoiding long fibrous material which will not machine.

Other objects and advantages of the invention will become apparent as the description proceeds.

Referring to the drawing:

Figure 1 is a perspective view of a piece of molybdenum embodying our invention.

Figure 2 is a perspective view of an ingot of molybdenum undergoing a rolling operation.

Figure 3 is a sectional view of a swaging machine through which a piece of molybdenum is being passed.

Figure 4 is a longitudinal sectional view of a furnace in which molybdenum may be sintered and/or recrystallized.

Figure 5 is a reproduction of a photomicrograph, at a magnification of 100, in the drawing accompanying the application, of an etched surface of a piece of readily-machineable molybdenum, embodying our invention.

Figure 6 is a reproduction of a photomicrograph, at a similar magnification, of an etched surface of a piece of molybdenum which is hard to machine.

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Figure 7 is a reproduction of a photomicrograph, at a similar magnification, of a piece of molybdenum which is impossible to machine because the grains thereof are fibrous, that is, elongated to too great an extent.

It has been known that molybdenum can be machined only with great difficulty and even in the so-called machineable lots and single bars, a large percentage was found not machineable.

We have made a study of molybdenum which was found to be easily machineable, difficultly machineable, and practically unmachineable, and are able to now recognize the features that render it machineable and fabricate molybdenum that is uniformly machineable throughout.

We have found that it is essential, in easily machineable molybdenum, that the grain structure be such that in no direction is the length of the grains less than two or generally more than four times the width or diameter, on an average. However, the grain size has been found not to be restricted to a narrow range, as we have rendered molybdenum machineable over a range of 10 to 250 grains per square millimeter, which range includes most of the molybdenum used in industry.

Perfectly equiaxed grains, that is those in which the length corresponds with the transverse dimension or diameter, and occur in treated or cast metal, unworked mechanically, are not good because the crystal cohesion is poor so that the machining tool pulls whole grains out of the body of the metal beneath the actual cut, leaving a pitted finish, and the material composed of such grains is too weak, resulting in cracks and breaks due to the stress of the tool.

On the other hand, molybdenum in which the fibres are long, as in a case where the metal has been rolled or drawn sufficiently to make it ductile, will not machine at all. The tenacity of the long fibres breaks the tool tip, blunts the cutting edge, and results in fibres tearing out beyond the point of machining.

One method of producing easily machineable molybdenum in accordance with our invention, and with reference to the drawing in detail, is to press under conventional or standard conditions, the powdered metal into a slug, bar, or ingot 11, which may be of a convenient size, an example being 1" x 1½" in section, and 24" long. Such a bar may then be placed in a furnace, such as represented by the reference character 12 and corresponding with that illustrated in Figure 1 of the Hall et al. Patent No. 2,431,690, dated December 2, 1947. In such a furnace it is desirably treated in accordance with the method disclosed in said application, that is, it is heated to between about 1400° and 1700° C., or between 50% and 70%, or not more than 70% of that of fusion, as measured in degrees C., in a hydrogen-bearing reducing atmosphere, containing enough water vapor so that a reversible oxidation-reduction reaction occurs, for a relatively long period of time, say about two, or from about 1½ to 3 hours, resulting in the desired sintering of the particles to produce a strong coherent article.

Such a bar during the sintering operation shrinks to a coherent metallic condition, the final dimensions being approximately ⅞" x 1¼" x 20" long. The bar 11 produced may then be worked to develop the grains to the extent desired, that is, it may for example be passed between rollers 13 and 14, illustrated in Figure 2, after heating in a furnace at a temperature between about 1450° and 1500° C., and reduced to a bar 11ᵃ approximately $\frac{13}{16}$" square in section, that is about 60% of its original section. The bar 11ᵃ may then be passed through a swaging machine 15, after being taken from a furnace at a temperature of between 1350° and 1450° C. and reduced to a rod 11ᵇ, .535" diameter in cross-section, that is, about 34% of its pre-swaging section and about 21% of its original section. The rod 11ᵇ so produced may then be placed in the furnace 12 or another one and, in a protective or reducing atmosphere, recrystallized by holding 20 minutes or more at a temperature of about 1600° C. It may then be swaged or otherwise worked to the desired size, at which the crystals are elongated on the average to preferably no less than two and no more than four times their transverse dimensions. The scale formed may be cleaned off in boiling caustic soda or potash.

As an alternative, such a pressed and sintered bar or slug of molybdenum may, instead of being reduced to a bar $\frac{13}{16}$" square in cross-section, may be rolled to one ¾" square in cross-section, that is, about 51% of its original section at a furnace temperature of 1450° to 1500° C., then swaged to .636" round section, that is, about 56% of its pre-swaging section, and about 29% of its original section, at a furnace temperature of 1350° and 1400° C., and then recrystallized by heating for about 20 minutes or more at about 1600° C. in a protective atmosphere. It may then be swaged to .550" round, that is, with a reduction in area of about and not more than 25% and cleaned as by washing in boiling caustic.

Another example would be to treat as above by finishing to .550" round section before recrystallizing at 1600° C., after which just enough working is applied to avoid poor crystal cohesion and undesired weakness, not elongating the grains to more than four times their transverse dimensions on the average.

In the previous examples, we have obtained the desired elongation of the grains by a final working, after a reduction to about 21% in the first, and about 29% in the second example, of the original cross-sectional area (a reduction in area of respectively about 79% and 71%) and heating to restore said grains to an equiaxed condition. It is, however, possible to obtain this result without a final working step, if a final heating step at a temperature above that of recrystallization is accurately controlled as to time and temperature, so that the grains, after undue elongation for easy machineability, are restored, not to an equiaxed condition, but to one in the desired range where easy machineability obtains.

In other words, instead of sintering, working, then recrystallizing at a predetermined size, to an equiaxed condition, as by heating for twenty minutes at a temperature of about 1600° C. and finally working to the finished size and get the desired grain elongation, we may instead conclude the treatment, after sintering the pressed powder and working to finished size, by annealing for one, or approximately one, hour per ¼" of section thickness at a recrystallizing temperature between 1300° and 1350° C., or about 1325° C., in order to effect a formation of the preferred recrystallization pattern so that the elongated grains are not restored entirely to an equiaxed condition, as previously, but only modified toward that condition, stopping when the grains are still elongated to an extent that is not less than twice their transverse dimensions, and not more than four, or at the extreme, six times their transverse dimensions, on the average.

Such treatment is particularly applicable to making molybdenum rods for drilling, and external and internal threading as, for example, in making cathode supports in which $\frac{3}{16}$" diameter holes are drilled $2\frac{7}{32}$" deep, using high speed carbon steel drills. Prior to our invention, a ⅛" drill was first employed followed by a $\frac{3}{16}$" drill. However, when treating the molybdenum in accordance with our invention so that it is easily machineable, we now initially drill the hole with ease, using a $\frac{3}{16}$" drill.

Also we have found that, using light cuts and slow speeds, it is possible to externally thread such a molybdenum rod forming, for example, 30 threads per inch without difficulty by means of a tool bit and chaser. The resulting threads are relatively sharp and clean, as compared with prior practice in which only imperfect threads were obtainable.

Using a 20 thread per inch die, we have obtained sharp clean threads on rods, formed of machineable molybdenum in accordance with our invention, as contrasted with molybdenum not so treated, where only rough and broken threads were obtainable.

We have also tapped machineable molybdenum in accordance with our invention, producing 32 threads per inch with ease, whereas non-machineable molybdenum in accordance with prior practice could not be so tapped.

Figure 5 is a reproduction of a photomicrograph which represents easily machineable molybdenum, that is, in which the grains are slightly elongated to about but not more than four times their transverse dimensions, on the average. Figure 6, on the other hand, represents molybdenum in which the grains are elongated to more than six times their average transverse dimensions, but not to an extreme condition where very fibrous material is obtained. The metal represented by this figure is machineable only with difficulty.

Figure 7 shows, for comparison, molybdenum which has been worked or drawn to an excessive extent to elongate the grains or crystals in the direction of working to make it ductile. This material will not machine at all, because the tenacity of the long fibres breaks the tool tips, blunts the cutting edge, and causes fibres to tear out beyond the point of machining.

From the foregoing disclosure, it will be seen that we have devised a method for producing molybdenum which is readily machineable, and such readily machineable molybdenum. Variations in the method within the scope of our invention, in which the desired final crystal structure is provided, are permissible. In producing thin sections, such for example as represented in Figure 1 by the reference character 11c, it is usually desirable to have some further working, after the recrystallization treatment, to increase the strength and ductility of the metal so that it will resist, without failure, stress as applied by the cutting tool.

It is to be understood that all of the specified rolling and swaging operations elongate the crystals in the direction of the working operation, and that subsequent heating for the next operation must be such as to break up the grains toward the equiaxed condition again until near the finished size, when a slight amount of elongation, usually not to exceed four to one, or at most six to one, on the average, is desired for strength and ductility. This requires that the furnace temperatures for heating prior to working steps be high enough to remove previous elongation, or that a separate recrystallization step be instituted at some point in the process, preferably near the finish. The point this step should be interposed will be determined by the size to which the material is to be finished.

Although a preferred embodiment of our uniformly free machineable type of molybdenum and process of manufacture has been disclosed, it will be understood that modifications may be made within the spirit and scope of the appended claims.

We claim:

1. The method of producing easily machineable molybdenum comprising rolling a bar about ⅞" x 1¼" in cross-section of that material sintered from the pressed powder to about 13/16" square in cross-section at a temperature between about 1450° and 1500° C., swaging said bar to about .535" diameter in cross-section at a temperature between about 1350° and 1400° C., recrystallizing by heating at about 1600° C. for about twenty minutes in a protective atmosphere, and then swaging to a desired size at which the crystals are elongated on the average to no less than two and no more than four times their transverse dimensions.

2. The method of producing easily machineable molybdenum comprising rolling a bar about ⅞" x 1¼" in cross-section of that material sintered from the pressed powder to about ¾" square in cross-section, at a temperature between about 1450° and 1500° C., swaging to about .636" diameter in cross-section at a temperature between about 1350° and 1400° C., recrystallizing by heating at about 1600° C. for about twenty minutes in a protective atmosphere, and then swaging to a cross-section of about .55" diameter.

3. The method of producing easily machineable molybdenum comprising rolling a bar about ⅞" x 1¼" in cross-section of that material sintered from the pressed powder to about .550" diameter in cross-section at a temperature between about 1450° and 1500° C., recrystallizing by heating at about 1600° C. for about twenty minutes in a protective atmosphere, and then working just enough to avoid poor crystal cohesion and undesired weakness, elongating the grains to no less than two and no more than four times their transverse dimensions on the average.

4. The method of producing molybdenum comprising rolling and swaging a bar thereof sintered from the powder at temperatures between about 1350° C. and 1500° C., to reduce it in section and elongate its grains to produce a fibrous structure, recrystallizing said bar by heating at about 1600° C., and finally swaging to reduce the transverse sectional area of said bar about and not more than 25% at which the grains are elongated so that the ratio between length and transverse dimension lies between only slightly more than one to one and that where the grain lengths are six times their transverse dimensions, on the average, in order to make the material easily machineable, the exact grain length to width ratio depending on the strength desired in the metal, which strength increases with increase in length-width ratio.

5. The method of producing molybdenum comprising rolling and swaging a bar thereof, sintered from the powder, at temperatures between about 1350° C. and 1500° C., to reduce it not more than about 79% in section and elongate its grains to produce a fibrous structure, recrystallizing said bar by heating at about 1600° C., and finally working to reduce the transverse sectional area of said bar about and not more than 25%, thereby elongating the grains, on the average, to between two and six times their transverse dimensions, by said working, in order to make the material easily machineable.

6. The method of producing easily machineable molybdenum, comprising; first, the rolling and swaging of a sintered bar thereof at temperatures between about 1350° C. and 1500° C., to produce a fibrous structure; second, the recrystallization of said bar, as by heating at about 1600° C. and; third, the reduction in transverse sectional area of about and not more than 25%, to produce grains which are, on the average, not less than about twice as long as their transverse dimensions, to overcome poor crystal cohesion and undesired weakness and which are, on the average, not more than four times as long as their transverse dimensions.

7. The method of producing easily machineable molybdenum comprising rolling a bar about ⅞" x 1¼" in cross-section of that material sintered from the pressed powder, at a temperature between about 1450° C. and 1500° C., to about 13/16" square in section, swaging said bar at a slightly lower temperature to about .535" diameter in section, producing a fibrous structure, heat-treating at about 1600° C. to recrystallize the material, and then working to reduce its transverse sectional area about and not more than 25%, thereby elongating the grains to, on the average, no less than two and no more than four times their transverse dimensions.

8. The method of producing easily machineable molybdenum comprising rolling a bar about ⅞" x 1¼" in cross-section of that material sintered from the pressed powder, at a temperature between about 1450° C. and 1500° C. to about ¾" square in cross-section, swaging said bar at a slightly lower temperature to about .636" diameter in cross-section, producing a fibrous structure, recrystallizing at about 1600° C., and swaging to a cross-section of about .550" diameter.

9. The method of producing easily machineable molybdenum comprising rolling a bar of that

material sintered from the pressed powder to a smaller cross-section at a temperature between about 1450° C. and 1500° C., to elongate its grains to more than six times their transverse dimensions to produce a fibrous structure, and heating to a recrystallizing temperature between 1300° C. and 1350° C. for approximately one hour per ¼" of section thickness, to merely modify the grains toward an equiaxed condition, stopping, however, when the grains are still elongated, on the average, to an extent that is not less than twice, and not more than six times their transverse dimensions.

10. The method of producing easily machineable molybdenum comprising working to finished size a bar sintered from the powder to elongate its grains to more than six times their transverse dimensions to a fibrous condition, and concluding the treatment by annealing for approximately one hour per ¼" of section thickness at a temperature between 1300° C. and 1350° C., in order to effect a formation of the preferred recrystallization pattern, where the elongated grains are not restored entirely to an equiaxed condition, but still elongated to an extent, that is, on the average, not less than twice and not more than four times their transverse dimensions.

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By WILLIAM A. ELLIS,

Vice President and Trust Officer, Executor of the Last Will and Testament of John H. Ramage, Deceased.

ROBERT D. MALIN.

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