This invention relates to molybdenum and its alloys, and more particularly to such of exceptionally uniform grain structure.

An object of our invention, generally considered, is to manufacture molybdenum, and alloys thereof with small proportions of other metals such as cobalt, nickel, iron and tungsten, in such a way that improved grain structure is obtained.

Another object of our invention is to obtain in refractory metals, by an improved working schedule, exceptionally uniform grain structure, high hardness, augmented ultimate strength, and a higher "as worked" elongation with minimum porosity.

A further object of our invention is to produce molybdenum, and alloys thereof with small proportions of cobalt, nickel, iron or tungsten, by a swaging process involving first a treatment above recrystallization temperature, followed by a treatment below recrystallization temperature, the first treatment taking place during a single swaging pass, and the subsequent treatment occurring during a plurality of such passes.

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

The properties of molybdenum, and alloys thereof with small proportions of metal selected from the group consisting of cobalt, nickel, iron and tungsten, are primarily dependent from the grain structure of the swaged, rolled or otherwise worked ingot. By the expression "alloys of molybdenum with small proportions of metal selected from the group consisting of cobalt, nickel, iron and tungsten," we mean such in which the proportion of cobalt is not greater than .3%, in which the proportion of either of the alloying nickel or iron is not greater than 1/2%, an in which the proportion of the alloying tungsten is not greater than 25%, all proportions being by weight. Although cobalt is preferred as the alloying metal, nickel and iron have similar effects. Tungsten has a similar effect as to properties, but the working temperatures for such alloys are preferably slightly increased, depending on the proportion of tungsten in the alloy.

In accordance with our invention, we propose to work the metal first at a temperature above that of recrystallization, such as at about 1400° C. to 1450° C., and then work at a temperature below recrystallization, such as at about 950° C. to 1000° C. The working above recrystallization temperature is desirably confined to a single swaging, rolling, or other deforming pass, while the swaging or other working below recrystallization temperature is desirably carried on during two or more passes, with soaking or annealing in a hydrogen atmosphere at temperatures between about 950° and 1000° C. between passes. By such treatment, we propose to secure exceptionally uniform grain structure, high hardness, augmented ultimate strength, and a higher "as worked" elongation than has been obtained previously when working such materials. We have also found that such material processed in this way has minimum porosity.

As an example of what has been done, we desirably start with a hydrostatically pressed cylindrical slug or bar, conveniently about 1.375" in diameter. Such a bar after pressing is sintered in a furnace in accordance with conventional practice to about .950" diameter. We then propose to swage such a bar down to a diameter of about .500" in accordance with the following schedule:

First pass, down to about .710" diameter, while at a temperature of about 1450° C.
Second pass, swage from .710" diameter to .600" diameter, while at a temperature of about 1000° C.
Third pass, swage from .600" diameter to .500" diameter, while at a temperature of about 1000° C.

After the first pass, the bar was air-quenched to avoid recrystallization, and then soaked in hydrogen for about 15 minutes at a temperature of about 1000° C. before taking the second pass. The material was returned to the furnace after the second pass and soaked in hydrogen for about 15 minutes at about 1000° C. before taking the third or final pass. After the third pass the bar was air-quenched to room temperature.

From the foregoing, it will be seen that the bar treated in accordance with our invention is desirably reduced in area about 44% during a first pass at a temperature above recrystallization, about 28% during a second pass at a temperature below recrystallization, and about 30% during a third and final pass of a temperature below recrystallization. It will also be seen that the total area reduction from initial size to final size is about 72%, and critical temperatures are specified above.

Upon testing material as above treated and which contained .1% of cobalt, we found the following "as worked" properties, meaning the properties without further heat treatment or annealing:

Hardness .......................... V.P.N. 236
Yield point ........................ p.s.i. 107,000
Ultimate strength .................. p.s.i. 117,000
Elongation ........................ El. 39
Grain count ........................ A.S.T.M. 9-10

Although the foregoing example is specific to molybdenum alloyed with .1% of cobalt by weight, yet similar temperatures, which are also critical for the best results, may be employed in operating on molybdenum alloyed with proportions of cobalt within the range of .05% to .3% and proportions of either nickel or iron, within the comparable range of 1% to 5%. In operating on pure molybdenum, or such containing alloying material less than the minima above specified or less than 1% of tungsten, the first swaging or rolling temperatures are desirably slightly higher, or approximately 1450° to 1475° C., as compared with 1400° to 1450° C. for the molybdenum-cobalt alloys.

Likewise, the forging or other working temperature below recrystallization for pure molybdenum, and such including the minima in alloying ingredients above specified, is desirably increased to about 1000° C. to 1050° C. as compared with 950° C. to 1000° C. for the molybdenum-cobalt alloys. The working temperatures for molybdenum-tungsten alloys, within the specified range of up to 25% tungsten (or from 1% to 25%), are desirably about the same as for pure molybdenum. Likewise, the annealing or soaking temperature after forging is desirably increased to about 1000° C. to 1050° C. for pure molybdenum and such including the minima in alloying ingredients above specified, as well as for molybdenum alloyed with tungsten within the specified range.

Although preferred embodiments have been disclosed, it will be understood that modifications may be made within the spirit and scope of the invention.
We claim:

1. The method of manufacturing ductile metal from a pressed and sintered article of powdered particles of the group consisting of molybdenum and alloys thereof with small proportions of metal selected from the group consisting of cobalt, nickel, iron and tungsten, comprising working said article to reduce its cross-sectional area while at a temperature of between about 1400° C. and 1475° C., air-quenching said worked article to a temperature below its recrystallization temperature, soaking said quenched article in a protective atmosphere for about 15 minutes at a temperature of about 950° C. to 1050° C., working said soaked article to reduce its cross-sectional area while at a temperature of about 950° C. to 1050° C., soaking said worked article in a protective atmosphere for about 15 minutes at a temperature of about 950° C. to 1050° C., and finally air-quenching said worked article to room temperature.

2. The method of manufacturing ductile metal from powdered particles of the group consisting of molybdenum and alloys thereof with small proportions of metal selected from the group consisting of cobalt, nickel, iron and tungsten which have been pressed and sintered to form a generally cylindrical article about .95" in diameter, comprising swelling said article to reduce its diameter to about .71" while at a temperature of between about 1400° C. and 1475° C., air-quenching said swollen article to a temperature below its recrystallization temperature, soaking said quenched article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1050° C., swelling said soaked article to reduce its diameter to about .60" while at a temperature of about 950° C. to 1050° C., soaking said swollen article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1050° C., swelling said soaked article to further reduce its diameter to about .50" while at a temperature of about 950° C. to 1050° C., and finally air-quenching said swollen article to room temperature.

3. The method of manufacturing ductile metal from a pressed and sintered article of powdered particles of molybdenum alloyed with from .05% to .3% of cobalt, comprising swelling said article to reduce its cross-sectional area while at a temperature of about 1400° C. to 1450° C., air-quenching said swollen article to a temperature below its recrystallization temperature, soaking said quenched article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1000° C., swelling said soaked article to reduce its cross-sectional area while at a temperature of about 950° C. to 1000° C., soaking said article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1000° C., and finally air-quenching said quenched article to further reduce its cross-sectional area while at a temperature of about 950° C. to 1000° C., and finally air-quenching said swaged article to room temperature.

4. The method of manufacturing ductile metal from a pressed and sintered article of powdered particles of the group consisting of molybdenum alloyed with from .1% to .5% of metal of the group consisting of nickel and iron, comprising swelling said article to reduce its cross-sectional area while at a temperature of about 1400° C. to 1450° C., air-quenching said swaged article to a temperature below its recrystallization temperature, soaking said quenched article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1000° C., swelling said soaked article to reduce its cross-sectional area while at a temperature of about 950° C. to 1000° C., soaking said article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1000° C., swelling said soaked article to further reduce its cross-sectional area while at a temperature of about 950° C. to 1000° C., and finally air-quenching said swaged article to room temperature.

5. The method of manufacturing ductile metal from a pressed and sintered article of powdered particles of the group consisting of molybdenum alloyed with from .1% to .5% of metal of the group consisting of nickel and iron, comprising swelling said article to reduce its cross-sectional area while at a temperature of about 1400° C. to 1450° C., air-quenching said swaged article to a temperature below its recrystallization temperature, soaking said quenched article in a hydrogen atmosphere for about 15 minutes at a temperature of about 950° C. to 1000° C., swelling said soaked article to further reduce its cross-sectional area while at a temperature of about 950° C. to 1000° C., and finally air-quenching said swaged article to room temperature.

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