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METHOD OF MAKING FINE-GRAIN CHROMIUM

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The present invention relates to a method of producing very fine-grain solid chromium bodies, particularly rods, and more particularly, relates to a suitable heat treatment and extrusion process whereby chromium characterized by considerably improved physical and mechanical properties than that achievable by the processes of the prior art is obtained.

Up to the present time there have been many methods for the preparation of chromium barstock, but to our knowledge none of these methods have utilized chromium powder as herein taught with the benefits resulting therefrom. An example of such methods comprises the separate steps of: melting the cast material; forging; warm working; and recrystallization in order to obtain a stock of fine grain size. These methods exhibit inherent deficiencies in that they are both time consuming and costly. However, by our method of preparing fine grain chromium powder a much finer grain size is obtained. Furthermore, such size is obtained without the steps of melting and forging.

It is accordingly a primary object of our invention to provide a novel method of treating chromium powder to considerably enhance the properties of solid chromium resulting therefrom.

It is a further object of our invention to provide a novel method of treating chromium powder in a temperature controlled atmosphere to considerably enhance the properties of solid chromium resulting therefrom.

Other objects, features and advantages of our invention will become apparent to those skilled in this particular art from the following detailed disclosure thereof.

We have found that by a suitable heat treatment and extrusion process as hereinafter described in considerable detail that chromium rods, as for example, a grain size of ASTM 13, heretofore unobtainable, may be fabricated from chromium powders which rods and the like are characterized by a considerable increase and optimization of the mechanical properties thereof. In addition to the improved mechanical properties, our process makes chromium rod available far more economically than presently known methods. Furthermore, our process offers a means for producing such rods of a fine grain size.

In one particular example of our process we started with commercially available —325 mesh chromium powder which was analyzed as containing 0.3% iron and 0.5% oxygen. The first step was to reduce the iron content to 0.1% by agitating such chromium powder for five (5) minutes in a 2% solution of concentrated nitric acid in water followed by filtration and drying of the chromium residue.

Following this, the powder was placed in an 11 gauge, 2 inch outside diameter seamless steel tube with a plug welded to the bottom thereof. Then the powder was subjected to a pressure, applied to the cross-sectional area corresponding to the inner diameter of the tube, of 150 tons (about 125,000 p.s.i.). Thus, by cold compacting, a more dense chromium powder was obtained. The container was then evacuated, in order to minimize the possibilities of oxidation, sealed and heated to 1000° C. for ninety (90) minutes; after which the powder was hot compacted by a 300 ton load (about

250,000 p.s.i.) applied to the above mentioned area. The temperature was selected in order to negate the possibilities of producing a coarse grain chromium. That is, by maintaining the temperature below the material's recrystallization temperature the crystalline structure does not reform into a larger more coarse grain. The recrystallization temperature of the particular material used occurred at 1050° C. Of course, it will be understood by those skilled in this particular art, that the recrystallization temperature of the particular chromium powder used will be influenced by the degree of impurities in the powder. And once the recrystallization temperature of a particular powder used is determined, it is only necessary in the practice of this process to maintain the heats just below such temperature.

Following this, the container was removed by pickling in a 20% nitric acid solution in water and recontained in a cold rolled steel can 7 inches long, 2.7 inches in outer diameter, and 1.8 inches in inner diameter. Chromium deforms irregularly when hot compacted, therefore, it was necessary to machine the rod in order to properly insert it into a new container of cold rolled steel 7 inches long, 2.7 inches in outer diameter and 1.8 inches in inner diameter.

Thereafter, the container was evacuated, sealed and heated to 1000° C. for ninety (90) minutes. In order to further reduce the possibilities of oxidation the container may be heated in graphite.

Following this, the container was extruded under a press load, applied to the total cross-sectional area of the container, of 370 to 400 tons, through a 0.750 inch die in a 2.80 inch liner. The rod produced thereby, was then stripped by pickling in a 20% nitric acid solution in water. The resultant ½ inch rod appeared to be of good quality, with a density 99.9% that of pure chromium as determined by displacement comparison (Archimedes' Method) tests.

The high temperature strength of this fine grain chromium is more than twice that of typical forged chromium. Furthermore, after extrusion it was found that the chromium had a much higher recrystallization temperature of over 1200° C. The increase in optimization of the mechanical properties of the chromium rod is best shown by reference to the following table in which data is obtained for chromium fabricated by our process and chromium prepared by prior art processes. The following data is quoted:

MECHANICAL PROPERTIES AT 750–755° C.

	Yield Strength, p.s.i.	UTS, p.s.i.	Elong., percent	Reduction of Area, percent
Fine grain extruded powder chromium	44,800	53,200	44	45
Forged chromium	15,200	22,900	42	70
Extruded and forged chromium	21,000	25,600	50	----------

MECHANICAL PROPERTIES AT 950–960° C.

	Yield Strength, p.s.i.	UTS, p.s.i.	Elong., percent	Reduction of Area, percent
Fine grain extruded powder chromium	24,600	28,300	32	36
Extruded and forged chromium	13,700	14,000	76	----------

The elongation was computed by the following equation:

$$\text{Percentage elongation} = \frac{L_1 - Lo}{Lo} \times 100$$

wherein, "Lo" is the original length and "L_1" is the length before rupture. The reduction of area was computed by the following equation:

$$\text{Percentage reduction of area} = \frac{A - a}{A} \times 100$$

wherein, "A" equals the original unstressed area and "*a*" equals the area of the rod prior to rupture. The data for "Extruded and Forged Chromium" is quoted from "Transactions American Society for Metals," page 1077, volume 50, 1958.

It will thus be seen that we have provided a novel process whereby fine grain chromium is fabricated which exhibits highly increased mechanical properties as well as being less costly and time consuming than any processes heretofore known.

The foregoing detailed description has been given for clearness of understanding only, and no unnecessary limitations should be understood therefrom, for modifications will be obvious to those skilled in the art.

We claim as our invention:

1. In a process for the fabrication of fine grain chromium rod, the steps of: preparing a substantially fine grain chromium powder; cold compacting the powder at a predetermined pressure in a suitable container to increase the density of the powder; evacuating said container; sealing the container; heating the container with the compacted powder contained therein to a temperature of about 1000° C. for a period sufficient to insure a substantially constant temperature of about 1000° C. throughout; subjecting the container to a predetermined load at said temperature to further densify the powder; removing the container; reheating the chromium to a temperature of about 1000° C. in a second evacuated container; subjecting the container with the chromium therein to a press load in a liner whereby the material under load is extruded through a reduced opening at an end thereof; and removing the second container.

2. In a process of the fabrication of improved fine grain chromium rod, the steps of; preparing a substantially fine grain chromium powder; compacting the powder at a pressure sufficient to increase its density in a suitable container; evacuating said container; sealing the container in its evacuated condition; heating the container with the densified powder contained therein to a temperature just below the recrystallization temperature of the powder for a period sufficient to establish a substantially constant temperature throughout; subjecting the container to another pressure load at said temperature to further densify the chromium; removing the container; reheating the chromium to a temperature just below the recrystallization temperature of the powder in a second evacuated container; subjecting the container with the chromium therein to a press load in a liner whereby the material underload is extruded through a reduced opening at an end thereof; and removing said second container.

3. In a process for the fabrication of improved fine grain chromium rod, the steps of; preparing a substantially fine grain chromium powder; placing the powder in a ductile metal container which will not alloy with the chromium powder at temperatures below the recrystallization temperature of the powder; cold compacting the powder at a pressure of approximately 125,000 pounds per square inch to produce a more dense chromium powder; evacuating the container of any gases which would react with the chromium powder at elevated temperatures; sealing the container in its evacuated condition; heating the container with the compacted powder contained therein to a temperature just below about 1050° C., the recrystallization temperature of the powder, for a period sufficient to insure substantially constant temperature throughout; hot compacting the powder at said temperature at a pressure of approximately 250,000 pounds per square inch to further densify the chromium; removing the container; reheating the chromium to a temperature of about 1000° C. in a second evacuated container; subjecting the container with the chromium therein to a press load in a liner sufficient to extrude the container and chromium through a reduced opening at an end of the liner; and thereafter removing the container.

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