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METHOD OF MAKING FINE-GRAIN CHROMIUM

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The present invention relates to a method of pro-
ducing very fine-grain solid chromium bodies, particu-
larly 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 pro-
cesses 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 chro-
mium powder as herein taught with the benefits result-
ing 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 in-
hherent deficiencies in that they are both time consum-
ing 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 tempera-
ture controlled atmosphere to considerably enhance
the properties of solid chromium resulting therefrom.

Other objects, features and advantages of our inven-
tion will become apparent to those skilled in this par-
ticular 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 45, therefore unobtainable, may be fabri-
cated from chromium powders which rods and the like
are characterized by a considerable increase and opti-
mization of the mechanical properties thereof. In addi-
tion to the improved mechanical properties, our process
makes chromium rod available far more economically
than presently known methods. Furthermore, our pro-
cess 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 pow-
der 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 stainless 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 ob-
tained. 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 under-
stood by those skilled in this particular art, that the
recrystallization temperature of the particular chromium
powder used will be influenced by the degree of pure-
neurities in the powder. And once the recrystallization tem-
perature of a particular powder used is determined, it is
only necessary in the practice of this process to main-
tain 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. Chro-
mium deforms irregularly when hot compacted, there-
fore, 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 con-
tainer may be heated in graphite.

Following this, the container was extruded under a
3000 lbs. 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 1/2 inch rod appeared to be of
good quality, with a density of 99.9% that of pure chro-
mium as determined by displacement comparison (Arch-
medes' Method) tests.

The high temperature strength of this fine grain chro-
mium is more than twice that of typical forged chro-
mium. Furthermore, after extrusion, it was found that
the chromium had a much higher recrystallization tem-
perature 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 fol-
lowing data is quoted:

| MECHANICAL PROPERTIES AT 700-750° C. | Yield Strength, p.s.i. | TYS, p.s.i. | Elong., percent | Reduc-
of Area, percent |
|-------------------------------------|-----------------------|-------------|-----------------|------------------|
| Fine grain extruded powdered chrome
| 46,000 | 53,000 | 44 | 45 |
| Forged chromium | 13,200 | 22,000 | 42 | 70 |
| Extruded and forged chrome
| 21,000 | 26,000 | 50 | |

<table>
<thead>
<tr>
<th>MECHANICAL PROPERTIES AT 900-999° C.</th>
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<tbody>
<tr>
<td>Fine grain extruded powdered chrome</td>
</tr>
<tr>
<td>24,600</td>
</tr>
<tr>
<td>Extruded and forged chrome</td>
</tr>
<tr>
<td>13,700</td>
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The elongation was computed by the following equation:

Percentage elongation = \( \frac{L_f - L_o}{L_o} \times 100 \)

wherein, "L₀" is the original length and "Lₐ" is the
length before rupture. The reduction of area was com-
puted by the following equation:

Percentage reduction of area = \( \frac{A_{o} - A}{A_{o}} \times 100 \)
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 under load is extruded through a reduced opening at an end thereof; and removing the second container.

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