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**ARTICLES PRODUCED BY POWDER METALLURGY AND METHOD OF PRODUCING SAME**

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1 Claim. (Cl. 75-226)

This invention relates to articles produced by powder metallurgy technique and methods of producing them. It relates in particular to such articles of titanium and zirconium. It has for its object methods of producing such compacts with greatly improved mechanical properties.

In the known art, powders for producing compacts of titanium and zirconium have been made by a number of methods such as decomposition of titanium hydride, mechanical disintegration of Kroll process sponge and electrolytic production of particulate material. These powders are either made of round particles or irregular spongy masses. They are further characterized by increased hardness and decreased purity as the particle size decreases.

The applicant is not aware of any titanium or zirconium powder finer than 30 mesh having an oxygen content below .10%.

My invention consists in taking a powder preferably through 30 mesh on 60 mesh which powder is composed of individual filamentary particles, from 1.0-23.0 mils diameter and 20.0-250.0 mils length.

The material for my invention is characterized by high purity containing less than .03% oxygen and substantially no other impurities.

It is also characterized by its packing density as determined with a Scott tester, a cubic inch of material compacted by free falling, weighs at least 26 grams and in this compact has substantially all the filamentary particles oriented in the same direction.

If the material is compacted into a steel die by free fall, the particles of the compact will be oriented.

Such an oriented compact may be further compacted cold or hot to produce an article of improved properties as will be illustrated by examples.

The material as described for my invention may be produced in several ways. I prefer the following:

I pass a direct current from a comminuted anode of the pure metal to be produced in powder form to an iron cathode, both immersed in an electrolyte held at 800° C. and composed of sodium chloride in which is dissolved 5% of the metal to be produced in powder form as chloride having an average valence of 2.5 and 0.5% free sodium. The current passed is 50 amperes per pound of comminuted anode material, said anode material being through an 8 mesh screen.

The cathode is an iron rod 3/4" in diameter and spaced evenly 4" from the peripherally positioned comminuted anode.

The product is removed from the cathode, washed with dilute acid and broken up in a mixer provided with titanium surfaces.

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Screening the product prepared in this way provides the following fractions:

|             | Percent Total | Oxygen Content, Percent | Packing Density |
|-------------|---------------|-------------------------|-----------------|
| -60.....    | 5             | .04                     | 20.2            |
| +60-30..... | 82            | .02                     | 27.0            |
| +30.....    | 13            | .05                     | 21.0            |

It may be explained that the greater purity of the desired fraction +60-30 is due to the material containing higher oxygen is slightly more frangible and therefore appears in the finer fraction. The coarser particles are really aggregates of the finer particles.

The procedure and product are identical for titanium and zirconium.

*Example I*

In this example I use titanium. I take the -30+60 fraction produced as described above; I pack this by allowing it to loosely fall into a steel die 4 inches in diameter and 20 inches deep to substantially fill the die; the die is provided with an extrusion orifice 2 inches in diameter; after filling, I insert the plunger and compress to a 5 inch length compact. I then heat the die to 750° C. and extrude a 2 inch round bar about 18 inches long.

This bar has the following properties:

|                   |               |         |
|-------------------|---------------|---------|
| Ultimate strength | -----p. s. i. | 150,000 |
| Yield strength    | -----p. s. i. | 100,000 |
| Elongation        | -----percent  | 30      |

*Example II*

In this example, I use zirconium. I take the -30+60 fraction produced as described above. I pack this into a hollow cylindrical die having a molding cavity one inch in outer diameter providing walls 1/4 inch thick for the compact. The length of the die is 20 inches and the compact is compressed by a suitable punch to a length of 5 inches. This hollow cylinder compact, 1" O. D., 1/2" I. D. and 5" long is sintered in a vacuum of .2 micron at 1000° C. In this condition, it has a hardness of Brinell 82. This cylinder is suitable for swaging and drawing into tubing.

What is claimed is:  
Process of making up into a compact a mass of elongated crystals of a metal selected from the group consisting of titanium and zirconium in alpha form, which crystals vary in screen size between minus 60 mesh and plus 30 mesh including a major portion having a screen size minus 30 mesh plus 60 mesh and consisting of filamentary particles having a purity of less than 0.03% O<sub>2</sub> in which the average diameter of the individual crystals is within the range 1-23 mils and the maximum dimension is within the range 20-250 mils, which comprises screening the crystals to discard the plus 30 mesh content and the minus 60 mesh content, loosely packing the minus 30 plus 60 mesh filamentary crystals in a die under conditions to at least partially orient the crystals in the die and to achieve a packing density of at least 26 grams per cubic inch, and thereafter compacting the crystals in the die under a pressure of from 10 to 200 tons per square inch and at a temperature of from 600° C. to room temperature, the pressure being inversely proportional to the temperature.

**References Cited in the file of this patent**

The Electrochemical Society, Lilliendahl et al., paper presented at the 91st general meeting held at Louisville, Ky. April 1947; preprint 91-16. (Copies in Lib. and Div. 46.)