

## SHORT COMMUNICATIONS

## The Sintering of Colloidal Titanium

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It is said that, in powder metallurgy, the finer the particle size of metallic powder, the more simplified conditions can be used in sintering it. So far as we can determine, however, nothing has been reported about the sintering of any metallic powder as colloidal as pyrophoric.

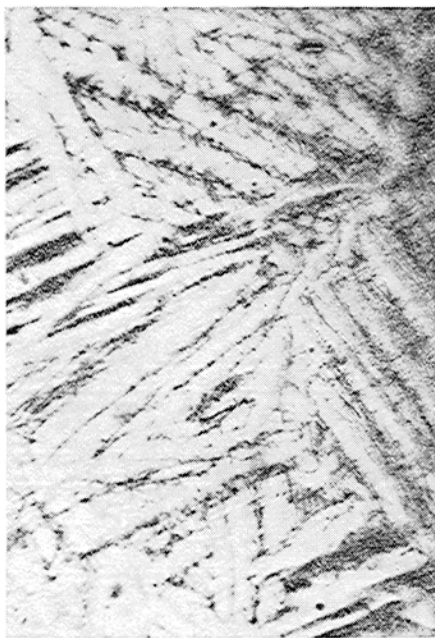
Recently, pure colloidal titanium was obtained through the dehydrogenation of titanium hydride, the composition of which was exactly stoichiometric ( $\text{TiH}_{2.00}$ ). This dehydrogenated titanium was compacted at a pressure of 5 ton/cm<sup>2</sup>, and then sintered for two hours at 1400°C. By this process a non-porous product was obtained; the density was found to be 4.54 g./cm<sup>3</sup>, which corresponds to the full density of metallic titanium.

First of all, a mass of sponge titanium (purity 99.9% by ASTM standard) was subjected to reaction with pure hydrogen gas in a vessel at a red hot temperature; the hydrogen was carefully kept in the vessel, and prevented from being degraded lower than 99.999% in purity by passing it through a membrane of palladium alloy. A titanium hydride material with a composition exactly equal to  $\text{TiH}_{2.00}$  was obtained by this process.

The titanium hydride obtained above was then milled for 40 hr. with a rotary mill made of sintered alumina. When the hydride powder thus pulverized was observed by electronmicroscopy, it was seen that the particles are of sizes between 60 Å and 300 Å, plus aggregated thereof. The hydride was then thermally dehydrogenated at 450°C in a tube furnace evacuated at  $2 \times 10^{-5}$  mmHg. A colloidal titanium was obtained, the specific surface of which was found to be 16 m<sup>2</sup>/g. by the B.E.T. method; its mean particle size was therefrom calculated to be 0.08 μ. The residual hydrogen of 27.7 p. p. m. was found by the fusion method. The colloidal titanium was kept in an air-tight jar filled with bomb nitrogen, as it is considerably pyrophoric in air.

The colloidal titanium obtained was then compacted in nitrogen atmosphere, by applying a pressure of 5 ton/cm<sup>2</sup>, into a disk, with a diameter

of 11.3 mm., a thickness of approximately 2 to 3 mm., and a green density of 3.1 g./cm<sup>3</sup>. The disk thus obtained, after impregnation with liquid paraffin in order to protect it from air, was sintered at 1400°C for two hours in a vacuum furnace kept at  $5 \times 10^{-5}$  mmHg. The density of this product was 4.54 g./cm<sup>3</sup>, as measured by a picnometer, while a nonporous structure was observed by means of microscopy (Mag.  $\times 400$ ).



Structure of non-porous titanium sintered at 1400°C for two hours. (Mag.  $\times 400$ )

In contrast to the common idea that some porosity is unavoidable in a product obtained through the sintering of metallic powder, it may be seen that, if a colloidal particle of titanium is taken as the starting material, a substantially-full density of metallic titanium can be obtained by means of sintering alone, using a powder compacted at a sufficiently high pressure and kept away from air.