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PREPARATION OF TITANIUM NITRIDE OF HIGH PURITY

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1 Claim. (Cl. 23—191)

This invention relates to the preparation of titanium nitride of high purity from titanium-containing metal scrap, such as scrap of titanium metal or scrap alloys of titanium with aluminum, vanadium, silicon, chromium, molybdenum, tungsten, manganese, iron, cobalt, nickel, indium, boron, copper or beryllium.

The present invention is based upon the discovery that titanium nitride is resistant to the action of strong acids, such as aqua regia, whereas such acids readily attack the elements present in titanium metal as alloying components or impurities and also readily attack the nitrides of such elements. Consequently, it is possible to prepare substantially pure titanium nitride from such titanium-containing materials by converting the titanium content thereof to nitride and then subjecting the nitrided material to the action of a strong acid, such as aqua regia, to convert the impurities to salts. The salts then may be removed in any desired manner such as by leaching the material with water or other suitable solvent.

Bulky pieces of titanium, such as pieces of sheet titanium, cannot be converted completely to titanium nitride by subjecting the pieces to the action of nitrogen or ammonia even at an elevated temperature. However, such pieces of titanium can be converted to hydride by subjecting the pieces to the action of hydrogen at an elevated temperature. The resulting hydrided pieces are brittle and can be broken up readily to finely divided form. The finely divided titanium hydride then can be converted to nitride by being subjected to the action of nitrogen or ammonia at an elevated temperature.

In accordance with the present preferred practice of the invention a plurality of pieces of titanium-containing metal scrap are placed in a confined reaction zone and the reaction zone is evacuated to remove air and moisture by subjecting the reaction zone to vacuum while being heated at a temperature between about 100° C. and 200° C. Hydrogen then is introduced into the reaction zone to maintain a small positive pressure therein, such as about five pounds per square inch, and the charge is heated to a suitable hydriding temperature, such as between about 900° C. and 1000° C. The reaction zone then is cooled and the hydrided pieces removed. The pieces then are broken up and converted to a finely divided form as by crushing.

The finely divided titanium hydride-containing material is placed in a confined reaction zone and the reaction zone is evacuated to remove air and moisture as previously described. Nitrogen or ammonia gas then is introduced into the reaction zone to maintain a small positive pressure therein, such as about five pounds per square inch, and the charge is heated to a temperature sufficiently high to start the nitriding reaction. If the nitriding gas used is ammonia, the latter is dissociated to nitrogen and hydrogen gases. Since the nitriding reaction is exothermic, the reaction proceeds to completion without additional heating. If ammonia is used as the nitriding gas, the produced hydrogen may be released periodically to maintain the desired pressure in the reaction zone to prevent leakage of air into the reaction zone. In order to prevent excessive rise in temperature, the rate of introduction of the nitriding gas into the reaction zone may be suitably controlled. An inert gas, such as helium or argon, may be introduced into the reaction zone to maintain the desired pressure therein, this being especially desirable when the nitriding gas used is nitrogen. When the nitriding reaction has gone to completion, the charge is cooled to about room temperature while maintaining the reaction zone under a slight positive pressure of ammonia, nitrogen or inert gas. The impure titanium nitride powder then is removed from the reaction zone and is subjected to the action of a strong acid, such as aqua regia, to convert the impurities to salts which are subsequently removed by leaching with water or other suitable solvent.

In place of aqua regia, other acids may be used for treating the impure titanium nitride to convert the impurities to salts. Thus, when the elements present in the titanium metal as alloying components or impurities are known, a particular acid may be selected for this purpose which is known to react with these elements or their corresponding nitrides to form salts. It is preferable to use for this purpose an acid which forms water soluble salts with the elements or their nitrides which are present in the impure titanium nitride.

I claim:

The method for recovering titanium in the form of high purity titanium nitride from scrap alloy of titanium with a metal selected from the group consisting of aluminum, vanadium, silicon, chromium, molybdenum, tungsten, manganese, iron, cobalt, indium, boron, copper, beryllium and mixtures thereof which comprises subjecting the alloy to a hydriding operation to convert the titanium content of the alloy to titanium hydride, reducing the titanium hydride in the thus treated material to finely divided form, subjecting the thus reduced material to a nitriding operation and thereby form a titanium nitride-containing mixture, subjecting said mixture to the action of aqua regia at about room temperature to form a mixture of titanium nitride and a compound of the selected metal, and removing said compound from the titanium nitride.

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