United States Patent [19]

Guidotti

[11] **3,925,068**

[45] **Dec. 9, 1975**

[54] **PREPARATION OF ALLOYS**

[75] Inventor: **Ronald A. Guidotti,** Reno, Nev.

[73] Assignee: **The United States of America as represented by the Secretary of the Interior,** Washington, D.C.

[22] Filed: **Sept. 5, 1974**

[21] Appl. No.: **503,536**

[52] **U.S. Cl.** **75/174;** 75/84; 75/135

[51] **Int. Cl.**[2] **C22C 27/02;** C22B 34/22; C22B 34/24

[58] **Field of Search** 75/84, 129, 135, 174

[56] **References Cited**

UNITED STATES PATENTS

839,585	12/1906	Heany	75/174 X
842,546	1/1907	Heany	75/174 X
1,355,532	10/1920	Brace	75/174 X
3,597,192	8/1971	Wilhelm	75/174 X
3,775,096	11/1973	Guidotti et al.	75/84

Primary Examiner—C. Lovell

Attorney, Agent, or Firm—William S. Brown; Donald R. Fraser

[57] **ABSTRACT**

Alloys of tantalum, niobium and vanadium are prepared by a process comprising (1) admixing Ta, Nb, or V nitride with an alloying metal, such as Ni, Co or Fe, (2) compacting the admixture and (3) heating the compact under vacuum at a temperature above the liquidus temperature of the desired alloy.

1 Claim, No Drawings

PREPARATION OF ALLOYS

Alloys of Ta, Nb or V have previously been prepared by processes such as hydrogen-reduction of a mixture of an oxide of the metal and the alloying metal or oxide, arc melting of pressed compacts of the alloy components that are prepared by aluminothermic reduction of the oxides, electric arc furnace treatment of mixtures of oxides, steel scrap, coke and a flux to form ferroalloys, etc. However, these processes all suffer from disadvantages such as limits as to the types of alloys that can be prepared, excessive energy requirements, undesired side reactions, low yields, etc.

It has now been found, according to the present invention, that the disadvantages of the prior art processes can be largely overcome by means of a process for preparing the alloys comprising (1) admixing Ta, Nb or V nitride with an alloying metal, (2) compacting the admixture and (3) heating the admixture under vacuum at a temperature above the liquidus temperature of the alloy.

Ta, Nb or V nitrides are most conveniently prepared by reaction of the corresponding oxide with ammonia, as disclosed in U.S. Pat. No. 3,775,096. They may, however, also be prepared by other known processes such as ammonolysis of the metal halides.

The process of the invention has been found to be particularly effective where the alloying metal is nickel, cobalt or iron, with the product alloys being useful as steel additives. However, other alloying metals, such as tin, manganese, molybdenum, tungsten and copper may also be used.

Both the Ta, Nb or V nitride, and the alloying metals, are preferably employed in finely divided form, i.e., having about −200 to −300 mesh particle size. However, particles up to about −50 mesh may be used if care is taken to ensure adequate mixing. Even larger particles may be used if sufficient attrition occurs during mixing. These particle sizes are readily obtained by size reduction of the nitrides and metals, individually or admixed, by conventional means such as ball and rod milling. Optimum proportions of nitride and alloying metal will depend on the specific nitride and alloying metal and on the intended use of the alloy. Generally, however, the ratio of the alloying metal to the nitride may range from about 0.1 to 5.0, preferably about 0.3 to 3.0.

The finely divided nitride and alloying metal are intimately admixed by conventional means, such as a rod mill, and the mixture is compacted by application of a pressure of about 25,000 to 45,000 psi for a period of 5 to 10 sec.

Prior to compaction, a lubricant-binder is also preferably added to, and admixed with, the nitride and alloying metal for the purpose of minimizing any galling tendency in the die during compaction. A 1% solution of camphor in a solvent such as acetone, methyl alcohol, benzene, acetic acid or chloroform has been found to be particularly effective for this purpose. However, other lubricant-binder materials may be used provided they are not present in sufficient amounts to cause high residual carbon contamination during heating. Generally, amounts of about 0.5 to 2 percent, preferably no more than about 1 percent, of the lubricant-binder are satisfactory. Other suitable materials are high molecular weight polyethylene glycols, e.g., the Carbowaxes, in solution in acetone; stearic acid or butyl stearate in a hydrocarbon solvent; dextrose in water and a saturated aqueous solution of ammonium carbonate. Compaction may be by any conventional means such as cold-pressing in a die at the required pressure, for a period of about 5 seconds. Other suitable means include hydrostatic pressing or hot pressing.

The compact is then heated at a temperature above the liquidus temperature of the desired alloy, which will generally range from about 1,200° to 1,800°C, under a vacuum of about 1,000 to 10μ Hg, for a period of about 2 to 12 hours. Nitrogen is thereby driven off, leaving a high-purity molten alloy behind. By adjustment of the temperature and composition of compact, i.e., the ratio of nitride to metal, solid alloys containing significant amounts of nitrogen, e.g., about 1 to 9%, can also be prepared. These are particularly suitable for use as steel additives where strengthening by nitrogen is desired. The reaction may be carried out in any conventional reactor capable of providing the required temperature and vacuum. E.g., the reactants may be contained in an inert container, such as an alumina crucible, and the reaction conducted in an oven fitted with a vacuum system. Resistive heating, external or direct, or inductive heating, may be employed. Or, the compact can be arc melted under vacuum. Optimum total heating time will depend on temperature, which in turn depends on the composition and melting point of the alloy, pumping capacity of the vacuum system, size of sample and degree of nitrogen removal desired, and is best determined empirically.

The invention will be more specifically illustrated by the following examples.

EXAMPLE 1

A compact containing 5g of NbN and 13.8g of Ni was prepared by admixing −200 mesh NbN and Ni with 25 ml of a 1% solution of camphor in acetone in a rod mill for 10 minutes, followed by cold-pressing in a die at a pressure of 45,000 psi for a period of about 5 seconds.

The compact was heated under vacuum in an Al_2O_3 crucible in a closed system at 1300°C in a Globar-heated furnace. The vacuum pump was rated at 33 liters per minute. After a period of 2 hours, the system vacuum had dropped to under 30μ Hg, at which time heating was stopped.

The resulting alloy analyzed < 0.003% N, 0.20% O and 78–80% Ni, with the balance being Nb.

EXAMPLE 2

A compact containing 5 g of NbN and 4 g of Ni was prepared and heated as in Example 1, heating being at a temperature of 1270°C. The resulting alloy analyzed 2.6% N, 0.17% O and 44% Ni.

I claim:

1. A process for preparation of an alloy useful as a steel additive comprising (1) admixing a nitride of tantalum, niobium or vanadium with an alloying metal consisting of nickel, cobalt or iron, the particle size of the nitride and the alloying metal being reduced to about −50 to −300 mesh prior to admixing, with the ratio of the alloying metal to the nitride ranging from about 0.3 to 3.0, (2) compacting the admixture at a pressure of about 25,000 to 45,000 psi and (3) heating the resulting compact under a vacuum of about 1000 to 10μ Hg at a temperature of about 1200° to 1800°C, but above the liquidus temperature of the desired alloy, for a period of about 2 to 12 hours.

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