

[54] **ELECTRICALLY CONDUCTIVE
REFRACTORY BODY**

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[58] Field of Search **252/518, 520; 106/55;
264/61, 65**

[56] **References Cited**

UNITED STATES PATENTS

3,544,486 12/1970 Passmore 252/518 X

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[57] **ABSTRACT**

An electrically conductive refractory body suitable for use as a boat for the evaporation of metals consists essentially of TiB₂, BN and TiH₂. AlN may be optionally included.

1 Claim, No Drawings

ELECTRICALLY CONDUCTIVE REFRACTORY BODY

THE INVENTION

Electrically conductive boats are commonly used in the vacuum deposition of metals, e.g. aluminum, onto suitable substrates, such as paper or plastic film. Electric current is passed through the boat in order to heat it to a temperature at which metal will evaporate therefrom. Examples of materials commonly used in the manufacture of such boats are graphite, composites of aluminum nitride and titanium boride, and composites of boron nitride and titanium boride.

The useful life of such boats is generally quite short because of, among other things, the high temperature of operation. For example, in the vacuum deposition of aluminum, the boats are resistively heated to temperatures of at least about 1450°C. The combination of high temperature, corrosiveness of the metal being evaporated and thermal cycling that may occur during the life of the boat can cause cracks to occur in the boat. Such cracking is generally a major cause of failure of such boats.

It is a purpose of this invention to provide a composite boat that is more resistant to cracking than prior art compositions.

A boat in accordance with this invention consists of a composition of titanium diboride, boron nitride and titanium dihydride, and may also contain aluminum nitride. Improved resistance to cracking results when the boat composition is within about the following ranges: 5 to 65 volume percent of BN; 20 to 50 volume percent of TiB₂; 1 to 5 volume percent of TiH₂; 0 to 30 volume percent of AlN.

In one example of a boat in accordance with this invention, 254 grams (41 volume %) of TiB₂, 178 grams (57 volume %) of BN and 14 grams (2 volume %) of TiH₂ were thoroughly mixed together and then hot pressed in a vacuum chamber inside a graphite die-mold at 2,050°C and 4,000 psi for 4 hours to yield a disc 4 3/16 inches in diameter by 5/8 inch thick. The disc had a density of 3.06 grams/cc, which is 97.7% of the absolute theoretical density of 3.235 grams/cc.

A similar disc was prepared of a prior art composition using the same hot pressing process, the composition consisting of 267 grams TiB₂, 169 grams BN and 9 grams of H₃BO₃. Two evaporation boats having overall dimensions of 0.375 by 0.750 by 3.00 inches were machined from each composition. Cavities were machined in the boats to hold the metal evaporant. The boats were then tested under identical conditions by self-resistance heating to the evaporation temperature range for Al (1,400° to 1,700°C) in a vacuum chamber, and 0.060-inch diameter Al wire was fed thereinto. 5 gms of Al were so evaporated at a rate of 1.0 gm/minute, after which the rate was increased successively to 2.0, 3.0, 4.0, 5.0, and 6.0 gms/min. with the evaporation of 50 gms at each rate. Thus, the test consisted of the evaporation of a total of 300 gms of Al at successively increasing rates from 1 to 6 gms/min. After the test, the boats were examined for evidence of cracking. In the two boats made in accordance with this invention, one had no cracks and the other had only very slight cracks. However, the two prior art boats were both cracked to a substantial extent.

It is believed that the increased resistance to cracking may be due to the reduced porosity, as shown by the higher percentage of theoretical density, that results from the use of TiH₂. The composition of this invention had a density that was 97.7% of theoretical absolute density, while the prior art boat's density was only 94.5% of theoretical absolute density.

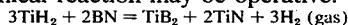
In order to determine whether TiH₂ also increased the density of composites containing AlN, a second pair of composites was made in a similar manner to that described above. A composite in accordance with this invention was made by mixing together 165 grams (31.5 volume %) TiB₂, 113.5 grams (43.2 volume %) BN, 88.5 grams (23.3 volume %) AlN and 12 grams (2 volume %) TiH₂ and hot pressing for 4 hours at 2,050°C under 4,000 psi pressure.

Simultaneously, a prior art composite consisting of 176 gms TiB₂, 113.5 gms BN, and 88.5 gms of AlN was also hot pressed under the same conditions. Again, the composite of this invention had a significantly higher percentage of absolute theoretical density than the prior art composite, 97.0% versus 94.5%.

These tests show that the enhanced density conferred by the addition of TiH₂ is attained whether or not AlN is included as a constituent of the composite.

In fact, the benefits conferred by the addition of TiH₂ are also not limited to composites containing TiB₂, but are specifically applicable to any composite containing BN or to the latter alone. An example of the use of TiH₂ in the fabrication of BN is provided by the following example.

636 gms of a BN powder which had been previously shown to be incapable of consolidation into a sound, dense refractory body by hot pressing (or any other means) was mixed thoroughly with 328 gms of TiH₂ powder. This composition equals 80 volume % BN, 20 volume % TiH₂. The mixed powders were hot pressed as described above at 2,050°C and 2,500 psi for 4 hours into a sound refractory body 5.03 inches diameter by 1.09 inch thick. The density was determined to be 2.53 gms/cu cm, corresponding to 93% of the absolute theoretical density of 2.72 gms/cu cm. Although the mechanism whereby the TiH₂ facilitates consolidation of the BN is not known, it is suggested that the following chemical reaction may be operative.



Attempts to make sound BN bodies using lesser TiH₂ additions were not successful. One of these comprised the equivalent of 10 vol % TiH₂ (half that of the above example) and exhibited extensive cracks normal to the hot pressing direction after simultaneous consolidation under the same conditions used above. Crucibles were machined from both composites, and the latter showed much thermal shock cracking in tests involving external heating and evaporation of Al. In contrast, the former (20 vol % TiH₂) composite showed no cracking. A third composite comprising 5 vol % TiH₂ was also made at the same time under the same conditions, and completely fell apart (disintegrated) after removal from the graphite die. Thus, it is evident that the TiH₂ addition is the bonding agent, and that it must be present in sufficient amount (more than 10 vol % TiH₂ for this grade of BN) in order to attain a sound body. However, lesser amounts of TiH₂ may be adequate to enhance densification with other types of BN powder, which are more capable of being densified by hot pressing because of better particle geometry or other reasons.

The above examples demonstrate the benefits conferred by the addition of TiH_2 , its applicability to a wide range of composites containing BN, and indicate its usefulness in the manufacture of such composites. It may also be used in increasing the size of bodies which can be made with a specific unit of manufacturing equipment, as well as for enhancing the quality of such bodies.

I claim:

1. An electrically conductive refractory boat, for the vacuum deposition of metals, consisting essentially of TiB_2 , BN and TiH_2 , wherein said boat has the following composition: 5 to 65 volume percent of BN; 20 to 50 volume percent of TiB_2 ; 1 to 5 volume percent of TiH_2 ; 0 to 30 volume percent of AlN.

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