

FABRICATION OF DUCTILE INTERMETALLIC SPUTTERING TARGETS

Field of the Invention

[01] The invention is directed to a method of fabricating ductile intermetallic sputtering targets by elemental blending and hot isostatic pressing.

Background of the Invention

[02] Cathodic sputtering processes are widely used for the deposition of thin films of material onto desired substrates. A typical sputtering system includes a plasma source for generating an electron or ion beam, a target that comprises a material to be atomized and a substrate onto which the sputtered material is deposited. The process basically involves bombarding the target material with an electron or ion beam at an angle that causes the target material to be sputtered or eroded off the target. The sputtered target material is deposited as a thin film or layer on the substrate.

[03] The target materials for use in sputtering processes have developed from pure metals to ever more complicated alloys. The use of complex 3 to 6 element compounds and extremely brittle intermetallic alloys such as NiAl, NiAl, RuAl, CoAl, TiAl and NiNb are common in the sputtering industry. Alloying additions such as Cr, B, Zr, Ta, Hf, Pt, SiO₂, Ti₂O₃, and so on are frequently added to B2 (i.e. NiAl, CoAl, RuAl, ..) and other intermetallic alloys to modify characteristics such as deposited film grain-size or surface energy.

[04] Most intermetallic alloys are intrinsically hard and brittle, and some of them are less thermal conductive than metals. Therefore, these intermetallic alloys, once consolidated into solid forms pose daunting challenges associated with machinability into targets and service ductility during cathodic sputtering. These materials typically exhibit very limited

mechanical shock resistance during machining and thermal shock resistance during sputtering.

Summary of the Invention

[05] The present invention relates to a novel method of fabricating sputtering targets that have an intermetallic stoichiometry, that renders them ductile enough for machining and sputtering. The process employs elemental blending of the prescribed species that constitute the intermetallic alloy and low-temperature hot isostatic press (HIP) consolidation at high pressure to prevent and control the formation of the intermetallic phases in the target material. The fact that the target does not contain the nominal intermetallic phase is not an issue in the application since cathodic sputtering is an atom-by-atom deposition process where the different atomic species recombine on the substrate to form the equilibrium and desired intermetallic phase. Another object of the present invention is to reduce the cost of target materials because the manufacture of intermetallic powders prior to HIP consolidation is not a required step. Typically, alloy powders are manufactured using sintering or gas atomization processes which tend to have very high associated batch run costs. These and other objectives of this invention will become apparent from the following detailed description.

Brief Description of the Drawings

[06] Reference is now made to the accompanying drawings wherein:

[07] FIG. 1 is a process flow chart of the invention described herein; and

[08] FIG. 2a to 2h show the microstructures of some of the alloys represented in the Table.

Detailed Description of the Invention

[09] FIG. 1 shows the process flow scheme for making the targets of the invention. The first step is the selection of raw material powders like Al, Ti, Ru, Ni, Nb, etc. at 10. It must be pointed out here that at least one of the powders involved must be a very fine powder such as -400 mesh because of densification requirements. For instance, Al powder has an average

particle size of 30 microns in all X-Al-Y, where X can be represented by elements such as Ru, Ti, Co and Ni, and Y can be represented by elements such as Cr, B, Zr, Ta, Hf, Pt, SiO₂, Ti₂O₃. The specific alloy compositions are those typically associated with crystal structures such as B2, L1₂, D0₁₉, L1₀, etc. Theoretically, the finer the powder is, the better it is for processing, but ultrafine Al powder is very difficult to handle due to its explosive nature. Therefore Al powder of 30 microns mean particle size is typically selected for all Al containing materials. The same considerations apply to Ni powder in NiNb.

[10] Blending at 20 is also critical for the whole process because the homogeneity of final products depends on this step. In practice, various blending methods can be employed to reach required homogeneity, such as V-blending, Turbular blending, ball mill blending and/or attritor mill blending (wet or dry), all of which are well known in the art.

[11] The blended powder is then compacted if necessary at 30 and then subjected to canning at 40 prior to HIP pressing.

[12] In step 40 following the blending process, the powders are canned prior to HIP processing. For example, a container is filled with the powder, evacuated under heat to ensure the removal of any moisture or trapped gasses present, and then sealed. Although the geometry of the container is not limited in any manner, the container can possess a near-net shape geometry with respect to the final material configuration.

[13] As mentioned above, low-temperature/high-pressure hot isostatic pressing (HIP) at 50 is a requisite part of the process. The low temperature mitigates the formation of embrittling intermetallic reaction zones between the elemental particles and high pressure ensures complete densification of the powder composite. As described herein, a temperature in the range of 200 to 1000 °C and pressure in the range of 5 ksi to 60 ksi are employed for isostatic pressing. The holding time at the designated temperature and pressure ranges from 0.5 to 12 hours. After HIP consolidation, the solid billet can be machined at 60 to final desired dimensions using a variety of techniques including wire EDM, saw, waterjet, lathe, grinder, etc. any of which are well known in the art. It is noteworthy that other powder consolidation techniques such as hot pressing and cold pressing can also be employed independently or in

conjunction with HIP processing, depending on desired results. After machining, the product is cleaned and subjected to a final inspection at 70.

[14] The following table depicts some alloys manufactured using the invention described herein. Figures 2a to 2h depict the microstructures associated with these alloys and demonstrate the minimized intermetallic reaction zones between the individual elemental phases.

[15] Table: Summary of alloys investigated.

Materials	Typical Chemistry
Al-Ni-B	Al60at%-Ni30at%-Cr10at%
Ru-Al	Ru50at%-Al50at%
Co-Al	Co50at%-Al50at%
Ti-Al	Ti50at%-Al50at%
Ni-Al	Ni50at%-Al50at%
Ni-Nb	Ni50at%-Nb50at%

In the Table and Figures 2a-2f, Figures 2a-2b depict the Al-Ni-B alloy as an overview and in detail; Figures 2c-2d depict the Ni-Nb alloy in an overview and in detail; Figures 2e-2f depict the Ru-Al alloy in an overview and in detail; Figure 2g depicts the microstructure of the Co-Al alloy, and Figure 2h depicts the microstructure of the Ti-Al alloy.

[16] While this invention has been described with reference to several preferred embodiments, it is contemplated that various alterations and modifications thereof will become apparent to those skilled in the art upon a reading of the detailed description contained herein. It is therefore intended that the following claims are interpreted as including all such alterations and modifications as fall within the true spirit and scope of this invention.

What is Claimed Is:

1. A method of fabricating ductile sputter targets that have an intermetallic chemistry, the method comprising the steps of:

selecting raw elemental or alloy powders;

blending the blended powders;

canning the blended powders;

hot isostatic pressing the canned powders at low temperatures and at high pressures to mitigate the formation of intermetallic reaction zones;

forming a billet; and

machining the billet to form a target.

2. A method according to claim 1, wherein at least one of the powders or alloys selected has a particle size of at least as small as -400 mesh.

3. A method according the claim 1 wherein the powders are selected so as to form an alloy of the formula X-Al-Y, wherein Al is aluminum, X is Ru, Ti, Co or Ni, and Y is Cr, B, Zr, Ta, Hf, Pt, SiO₂, or Ti₂O₃.

4. A method according to claim 3 wherein the resulting alloy composition is associated with a crystal structure selected from the group consisting of B2, L1₂, DO₁₉ or L1₀.

5. A method according to claim 3, wherein the Al powder has a mean particle size of about 30 microns.

6. A method according to claim 1, wherein the parameters of hot isostatic pressing comprise a temperature of 200 to 1000°C, a pressure of 5 to 60 ksi, and a time period of 0.5 to 12 hours.

7. A method according to claim 1, wherein the target contains RuAl.
8. A method according to claim 1, wherein the target contains CoAl.
9. A method according to claim 1, wherein the target contains TiAl.
10. A method according to claim 1, wherein the target contains Al-30at%Ni-10at%Cr.
11. A method according to claim 1, wherein the target contains NiAl and/or NiNb.