A sputter target comprises a plurality of materials. The plurality of materials includes at least a first material and a second material. The first material is comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe). The second material is comprised of carbon (C), a carbide, a nitrogen (N)-containing material, a nitride, a silicide, an oxygen (O)-containing material, an oxide, boron (B), or a boride. The second material has an average size of between 0.01 microns and 50 microns or is not more than 50 microns in size. In one embodiment, the first material comprises at least 15 atomic percent or greater.
Raw Material Preparation or Selection

Blending

Compacting
When needed

Canning

HIP

Machining to final dimension

Final QA clean and inspect

Figure 1
SPUTTER TARGET AND METHOD FOR FABRICATING SPUTTER TARGET INCLUDING A PLURALITY OF MATERIALS

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application is a divisional application of U.S. patent application Ser. No. 10/739,401, filed Dec. 19, 2003, which is a continuation-in-part of U.S. patent application Ser. No. 10/200,590, filed Jul. 23, 2002, all of which are hereby incorporated by reference in their entirety for all purposes.

FIELD OF THE INVENTION

[0002] The invention is directed to sputtering targets and methods of fabricating sputtering targets and, in particular examples, to sputtering targets and methods of fabricating sputtering targets doped with non-metal additions including boron, carbon, nitrogen, or oxygen preferably in the form of borides, carbides, nitrides or oxides of transition or refractory elements, by using atomized or crushed alloy powder or ultra fine boride, carbide, nitride or oxide, by hot pressing or hot isostatic pressing, and products produced thereby.

BACKGROUND OF THE INVENTION

[0003] 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 involves bombarding the target material with an electron or ion beam at an angle that causes the target material to be sputtered or eroded. The sputtered target material is deposited as a thin film or layer on the substrate.

[0004] The target materials for sputtering process range from pure metals to ever more complicated alloys. The use of complex 3 to 6 element alloys is common in the sputtering industry. Alloying additions such as boron, carbon, nitrogen, oxygen, silicon and so on are frequently added to Cr-, Co-, Fe-based alloys and other intermetallic alloys to modify characteristics such as deposited film grain-size, surface energy and magnetic properties.

[0005] The presence of non-metal additions like boron, carbon, nitrogen, oxygen and silicon to target materials is either in the form of pure elements, e.g. boron and carbon, or in the form of compounds like nitride and oxide. The pure element phases such as boron and carbon and the compound phases like boride, carbide, nitride, oxide, and silicide, however cause spitting problems during sputtering. The present invention provides a solution to this problem.

SUMMARY OF THE INVENTION

[0006] According to one embodiment, the present invention relates to a novel method of fabricating sputtering targets that are doped with non-metals such as boron, carbon, nitrogen, oxygen and silicon or mixtures thereof or compounds of non-metals and products produced by these processes. According to one embodiment, a process comprises preparation of a pre-alloyed powder or selection of ultra fine compound powder of about 0.01 to 50 microns, preferably 0.1 to 10 microns, more preferably 1.0 to 5.0 microns average particle size and most preferably less than 2 microns. It has been discovered that spitting will not occur when the above phases are in form of ultra fine particles of less than 50 microns, preferably less than 10 microns in size. After the ultra fine powders are blended together, the powder blend is canned, followed by a hot isostatic press (HIP) consolidation. Powder processing as above is employed to make the target materials because of unique advantages over the prior art’s melting process, both technically and economically.

[0007] According to one embodiment of the present invention, a sputter target comprises a plurality of materials. The plurality of materials includes at least a first material and a second material. The first material is comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe). The second material is comprised of carbon (C), a nitrogen (N)-containing material, a carbide, a nitride, or a silicide. The second material has an average size of between 0.01 microns and 50 microns, and the first material comprises at least 15 atomic percent or greater.

[0008] According to one embodiment of the present invention, a sputter target comprises a plurality of materials. The plurality of materials includes at least a first material and a second material. The first material is comprised of cobalt (Co), ruthenium (Ru), nickel (Ni), or iron (Fe). The second material is comprised of an oxygen (O)-containing material or an oxide. The second material has an average size of between 0.01 microns and 50 microns.

[0009] According to one embodiment of the present invention, a sputter target comprises a plurality of materials. The plurality of materials includes at least a first material and a second material. The first material is comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe). The second material is comprised of an oxygen (O)-containing material or an oxide. The second material has a size of between 0.01 microns and 50 microns. If the sputter target consists of chromium (Cr) and the oxygen (O)-containing material other than simply chromium oxide. If the sputter target consists of chromium (Cr) and the oxide only, the oxide is an oxide other than simply chromium oxide.

[0010] According to one embodiment of the present invention, a sputter target comprises a plurality of materials. The plurality of materials includes at least a first material and a second material. The first material is comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe). The second material is comprised of boron (B) or a boride. The second material is not more than 50 microns in size.

[0011] These and other objectives of this invention will become apparent from the following detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] Reference is now made to the accompanying drawing wherein:

[0013] FIG. 1 shows the process flow chart of the invention described herein.

DETAILED DESCRIPTION OF THE INVENTION

[0014] The alloy powders of the present invention include alloys and intermetallic alloys composed of 2 to 6 elements,
including but not limited to Cr-, Co-, Ru-, Ni-, or Fe-based alloys. The alloy powders contain Cr, Co, Ru, Ni, or Fe, optionally alloyed with Pt or Ta, and include at least one inorganic compound selected from a boride, carbide, nitride, or oxide of elements from the Periodical Table of elements shown in columns II to VIIIA, and I to IB.

[0015] In preferred embodiments, the non-metallic additive is in combined form such as an inorganic compound of a non-metal although elemental additions may be used if desired. Preferred doping non-metals are compounds of boron, carbon and nitrogen. In still other embodiments compounds of oxygen or silica are included as dopants. Preferred doping compounds are borides, carbides, and nitrides, as well as oxides and silicides. Especially preferred compounds are Mo3B, AlN (Aluminum Nitride), and B4C, as well as Al2O3, Cr2O3, SiO2, and mixtures thereof. The amount of dopant may range from about 1 to 15 atomic percent (at. %), and preferably from 1 to 12 at. %.

[0016] In further embodiments, the step of forming the doped elemental powders or alloys is carried out by mechanical mixing to achieve substantially uniform blending of the materials. In further embodiments, the canning step is carried out so as to avoid segregation of the doped element or alloy.

[0017] FIG. 1 shows the process flow for making the targets. The first step is the preparation of raw material powders like atomized alloy powders of Ni—Al—B, Fe—B, Fe—C, Fe—Si and so on or the selection of commercially available ultra fine compound powders such as Al2O3, AlN, Mo3B and Cr2O3 of 10 microns or less. Atomized powders have very fine microstructure because of extremely quick cooling and solidification, therefore it is the first choice as raw materials. In some cases powders of fine microstructures can also be made by melting and mechanically crushing ingots much more economically than by atomization, especially for small quantities of powder. Some ultra fine compound powders like Al2O3, AlN, Mo3B, Cr2O3, B4C and so on are also commercially available, and therefore save both time and money for new product development. Blending of various powders together is necessary because segregation occurs quite often, especially when powders of differing particle size and gravity are combined. Special blending and homogenizing methods include ball milling, v-blending, tubular blending, and attritor milling and/or wet blending. Therefore, it is preferred that the alloy powders and/or mixture be substantially homogeneous for best results.

[0018] Proper canning techniques are needed to avoid segregation during canning. Hot pressing in a graphite die could be used as well to consolidate the powder. The powders are canned in preparation for consolidation. In canning 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. In vacuum hot pressing, the chamber is continuously evacuated prior to and during load application. 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.

[0019] The encapsulate material from the canning step is then consolidated preferably via Hot-Isostatic-Pressing (HIP), a procedure known in the art. A HIP unit is typically a cylindrical pressure vessel large enough to house one or more containers. The inner walls of the vessel can be lined with resistance heating elements, and the pressure can be controlled by the introduction of inert gas within the container. HIP parameters including temperature, pressure and hold time will be minimized to prevent the growth of compound phases and grain size, as well as to save energy and to protect the environment. Pressures of about 5 to about 60 ksi (preferably 10-20 ksi) at temperatures between about 500°C, to about 1500°C, are typically employed to achieve appropriate densities. Depending upon the complexity of the cycle, the hold times during isostatic pressing typically vary from about 0.5 to about 12 hours. Pressure during vacuum hot pressing is varied from 0.5 to 5 ksi (preferably 1.5 to 2.5 ksi) at temperatures ranging from about 500°C to 1500°C (preferably 800-1000°C). 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.

[0020] After consolidation, the solid material form (billet) is removed from the encapsulation can, and a slice of the billet can then be sent to be tested as to various properties of the billet. If desired, the billet can be subjected to additional thermo-mechanical processing to further manipulate the microstructural and macro-magnetic properties of the target. Also, the final shape and size of the sputter targets can be formed, for example, by processes such as wire EDM, saw, waterjet, lathe, grinder, mill, etc. In these steps, the target can be cleaned and subjected to a final inspection.

### TABLE 1

<table>
<thead>
<tr>
<th>Materials</th>
<th>Typical Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co—Cr—Pt—B</td>
<td>Co61at%Cr65at%Pt12at%Bi2at%</td>
</tr>
<tr>
<td>Co—Cr—Pt—O—Si</td>
<td>Co56at%Cr18at%Pt16at%SiO2 (0.5—10) mol %</td>
</tr>
<tr>
<td>Co—Ta—N—B</td>
<td>Co60at%Ta5at%B10at% doped with nitrogen of 1—4 at.%</td>
</tr>
<tr>
<td>Co—Ta—Zr—O—Si</td>
<td>Co85at%Ta5at%Zr5at%SiO2 (0.5—10) mol %</td>
</tr>
<tr>
<td>Cr—Ti—Pt—B</td>
<td>Cr60at%Ti16at%Pt12at%B2at%</td>
</tr>
<tr>
<td>Cr—B—</td>
<td>Cr70at%B30at%</td>
</tr>
<tr>
<td>Cr—Mo—B</td>
<td>Cr80at%Mo10at%B5at%</td>
</tr>
<tr>
<td>Cr—Mo—O</td>
<td>Cr80at%Mo20at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Cr—O</td>
<td>Cr doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Cr—Ti—B</td>
<td>Cr80at%Ti16at%B4at%</td>
</tr>
<tr>
<td>Cr—V—O</td>
<td>Cr80at%V20at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Cr—V—Zr—O</td>
<td>Cr75at%V20at%Zr10at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Cr—W—O</td>
<td>Cr90at%W10at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Cr—Zr—O</td>
<td>Cr90at%Zr10at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Fe—Co—B</td>
<td>Fe55at%Co31at%Bi12at%</td>
</tr>
<tr>
<td>Fe—Si—Al</td>
<td>Fe75at%Si17at%Al10at%</td>
</tr>
<tr>
<td>Fe—Ta—C</td>
<td>Fe80at%Ta8at%C12at%</td>
</tr>
<tr>
<td>Ni—Al—B</td>
<td>Ni50at%Al10at%doped with boron of 1—4 at.%</td>
</tr>
<tr>
<td>Ni—Al—N</td>
<td>Ni40at%Al40at%doped with nitrogen of 4 at.%</td>
</tr>
<tr>
<td>Ni—Al—O</td>
<td>Ni50at%Al10at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Ru—Al—O</td>
<td>Ru50at%Al50at%doped with oxygen of 1—4 at.%</td>
</tr>
<tr>
<td>Ru—Al—N</td>
<td>Ru50at%Al50at%doped with nitrogen of 1—4 at.%</td>
</tr>
</tbody>
</table>
EXAMPLES

[0021] The following examples demonstrate the present invention further, but should not be construed as a limitation of the present invention. The processes for all materials are similar with each other as shown in FIG. 1, and the main differences are various combinations of raw materials (powders).

Example 1

Production of Cr—Mo Based Sputtering Target With Boron Content—Cr80 at %-Mo15 at %-B5 at %

[0022] The above alloy is made with the following powder blends, (1) Cr, Mo and ultra fine MoB compound powder, and (2) Cr, Mo and pre-alloyed Cr-3.1 wt % B powder that is made with a vacuum induction melter at 1730°C and mechanically crushing cast ingots into powder at room temperature. The pre-alloyed Cr-3.1 wt % B powder can also be made by gas atomization. Special attention must be paid to mixing all powders together when ultra fine compound powder like MoB is used, otherwise segregation may occur. Here with an attritor mill or a ball mill must be used for blending from 2 to 24 hours. The HIP parameters for this kind of alloy include the temperature ranging from about 1000-1400°C, at a pressure from about 5-40 ksi and a hold time from about 1-12 hours. The cooling rate must be controlled too, otherwise the HIPed billet may crack during cooling down. A cooling rate of 3°C/min and a hold plate at 800°C for 6 hours is introduced to cooling phase.

Example 2

Production of Co—Cr—Pt Based Sputtering Target With SiO2 Content—Co56 at %-Cr18 at %-Pt16 at %-SiO3.33 at %-SiH6.67 at %

[0023] Two different combinations of starting powders are employed herein. The first is the combination of Co, Cr, Pt and ultra fine SiO2 powder and the second is the combination of Co, Cr, Pt, atomized Co—Si pre-alloy and ultra fine CrO3 powder. The suicides are ultra fine and well dispersed in Co matrix of original gas-atomized Co—Si particles. Special mixing methods using an attritor mill or a ball mill for 2 to 24 hours must be employed here to mix all powders together homogeneously when ultra fine compound powders like SiO2 and CrO3 are used, otherwise segregation may occur. The HIP parameters for this kind of alloy include the temperature ranging from about 600-1400°C, at a pressure from about 5-40 ksi and a hold time from about 1-12 hours.

Example 3

Production of Cr—X (wherein X is Boride, Carbide, Nitride or Oxide, or Mixtures Thereof) Sputtering Target Doped with Oxygen

[0024] Cr80 at %-Mo20 at % doped with oxygen of 1-4 at omic % (at. %).

[0025] Regular Cr, Mo and partly oxidized Cr powder of oxygen level 15000 ppm are used to make the targets. The Cr powder of high oxygen is produced by oxidizing Cr flakes at high temperature and then subjected to mechanical crushing. In this case, only a part of the surface area of Cr powder is covered with oxides. Special attention must be paid to Cr powder of high oxygen level and mixing all powders together in this case, otherwise segregation may occur. Here with an attritor mill or a ball mill may be used for blending from 2 to 24 hours. The HIP parameters for this kind of alloy include the temperature ranging from about 800-1400°C, at a pressure from about 5-40 ksi and a hold time from about 1-12 hours. The cooling rate must be controlled too, otherwise the HIPed billet may crack during cooling down. A cooling rate of 3°C/min and a hold plate at 800°C for 6 hours is introduced to cooling phase.

Example 4

Production of NiAl Sputtering Target Doped with Boron, Oxygen or Nitrogen—Ni50 at % Al50 at % doped with boron of 1-4 at. %

[0026] Gas-atomized NiAl intermetallic powder and ultra fine Al2O3 and AlN powder of less than 5 microns in diameter were taken for making NiAl sputtering targets doped with oxygen or nitrogen. Besides gas-atomized NiAl powder, boron-doped gas-atomized NiAl powder was also taken for making NiAl sputtering targets doped with boron and borides are ultra fine and well dispersed in the matrix. Conventional gas atomization methods are used to manufacture the powders. Special attention must be paid to mixing all powders together when ultra fine compound powders like Al2O3 and AlN are used, otherwise segregation may occur. Here with an attritor mill or a ball mill may be used for blending from 2 to 24 hours. The HIP parameters for this kind of alloy include the temperature ranging from about 900-1400°C, at a pressure from about 5-40 ksi, and a hold time from about 1-12 hours. The cooling rate must be controlled too, otherwise the HIPed billet may crack during cooling down. A power-off furnace cooling and a hold plate at 700°C for 4 hours is introduced to cooling phase.

[0027] 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 sputter target comprising a plurality of materials, the plurality of materials including at least a first material and a second material, the first material comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe), the second material comprised of carbon (C), a nitrogen (N)-containing material, a carbide, a nitride, or a silicide, the second material having an average size of between 0.01 microns and 50 microns, the first material comprising at least 15 atomic percent or greater.

2. The sputter target of claim 1, wherein the plurality of materials includes a third material, the third material comprised of platinum (Pt) or tantalum (Ta).

3. The sputter target of claim 1, wherein the second material has an average size between 0.1 microns and 10 microns.

4. The sputter target of claim 1, wherein the second material has an average size between 1 micron and 5 microns.
5. The sputter target of claim 1, wherein the second material has an average size less than 2 microns.
6. The sputter target of claim 1, wherein the second material is comprised of a carbide or a nitride of an element from the Periodic Table of elements shown in column VIIA or column IB.
7. The sputter target of claim 1, wherein the plurality of materials further includes a third material comprised of a transition element, a refractory element, a cobalt-transition element alloyed material, or a cobalt-refractory element alloyed material.
8. The sputter target of claim 7, wherein the sputter target comprises grains of the plurality of materials.
9. A sputter target comprising a plurality of materials, the plurality of materials including at least a first material and a second material, the first material comprised of cobalt (Co), ruthenium (Ru), nickel (Ni), or iron (Fe), the second material comprised of an oxygen (O)-containing material or an oxide, the second material having an average size of between 0.01 microns and 50 microns.
10. The sputter target of claim 9, wherein the plurality of materials includes a third material, the third material comprised of platinum (Pt) or tantalum (Ta).
11. The sputter target of claim 9, wherein the second material has an average size between 0.1 microns and 10 microns.
12. The sputter target of claim 9, wherein the second material has an average size between 1 micron and 5 microns.
13. The sputter target of claim 9, wherein the second material has an average size less than 2 microns.
14. The sputter target of claim 9, wherein the second material is comprised of an oxide of an element from the Periodic Table of elements shown in column VIIA or column IB.
15. The sputter target of claim 9, wherein the plurality of materials further includes a third material comprised of a transition element, a refractory element, a cobalt-transition element alloyed material, or a cobalt-refractory element alloyed material.
16. The sputter target of claim 9, wherein the sputter target comprises grains of the plurality of materials.
17. The sputter target of claim 9, wherein the second material is comprised of silicon dioxide (SiO₂).
18. The sputter target of claim 9, wherein the sputter target is comprised of Co, Cr, Pt and SiO₂.
19. A sputter target comprising a plurality of materials, the plurality of materials including at least a first material and a second material, the first material comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe), the second material comprised of an oxygen (O)-containing material or an oxide, the second material having a size of between 0.01 microns and 50 microns,

wherein if the sputter target consists of chromium (Cr) and the oxygen (O)-containing material only, the oxygen (O)-containing material is an oxygen (O)-containing material other than simply chromium oxide, and

wherein if the sputter target consists of chromium (Cr) and the oxide only, the oxide is an oxide other than simply chromium oxide.

20. The sputter target of claim 19, wherein the plurality of materials includes a third material, the third material comprised of platinum (Pt) or tantalum (Ta).
21. The sputter target of claim 19, wherein the second material has an average size between 0.1 microns and 10 microns.
22. The sputter target of claim 19, wherein the second material has an average size between 1 micron and 5 microns.
23. The sputter target of claim 19, wherein the second material has an average size less than 2 microns.
24. The sputter target of claim 19, wherein the second material is comprised of an oxide of an element from the Periodic Table of elements shown in column VIIA or column IB.
25. The sputter target of claim 19, wherein the plurality of materials further includes a third material comprised of a transition element, a refractory element, a cobalt-transition element alloyed material, or a cobalt-refractory element alloyed material.
26. The sputter target of claim 19, wherein the sputter target comprises grains of the plurality of materials.
27. The sputter target of claim 19, wherein the second material is comprised of silicon dioxide (SiO₂).
28. The sputter target of claim 19, wherein if the sputter target consists of chromium (Cr) and the oxygen (O)-containing material only, the oxygen (O)-containing material is an oxygen (O)-containing material other than simply chromium oxide or simply silicon dioxide (SiO₂), and

wherein if the sputter target consists of chromium (Cr) and the oxide only, the oxide is an oxide other than simply chromium oxide or simply silicon dioxide (SiO₂).
29. A sputter target comprising a plurality of materials, the plurality of materials including at least a first material and a second material, the first material comprised of cobalt (Co), chromium (Cr), ruthenium (Ru), nickel (Ni), or iron (Fe), the second material comprised of a boride, the second material not more than 50 microns in size.
30. The sputter target of claim 29, wherein the plurality of materials includes a third material, the third material comprised of platinum (Pt) or tantalum (Ta).
31. The sputter target of claim 29, wherein the second material is less than 10 microns in size.
32. The sputter target of claim 29, wherein the second material is less than 2 microns in size.
33. The sputter target of claim 29, wherein the plurality of materials further includes a third material comprised of a transition element, a refractory element, a cobalt-transition element alloyed material, or a cobalt-refractory element alloyed material.
34. The sputter target of claim 30, wherein the sputter target comprises grains of the plurality of materials.
35. The sputter target of claim 30, wherein the second material is comprised of boron (B).