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(71) Applicant: **United Technologies Corporation**
Farmington, CT 06032 (US)

(72) Inventors:
- **LENZ, Brendan M.**
  West Hartford, CT 06107 (US)
- **WOODARD, Shiela R.**
  South Windsor, CT 06074 (US)
- **BERCZIK, Douglas Michael**
  Manchester, CT 06042 (US)
- **UCASZ, Mark Thomas**
  Middletown, CT 06457 (US)
- **BAJOREK, William John**
  Cromwell, CT 06416 (US)

(74) Representative: **de Bresser, Sara Jean**
Dehns
St Bride's House
10 Salisbury Square
London EC4Y 8JD (GB)

(54) METHOD OF FORMING MO-SI-B POWDER

(57) A method of forming Mo-Si-B alloy powder includes preparing a mixture that includes Mo powder, Si_3N_4 powder and BN powder, adding a polymer binder and liquid to the mixture to form a slurry, spray drying the slurry to form a spray dried powder containing Mo powder, Si_3N_4 powder and BN powder particles, and thermally treating the spray dried powder to remove the binder, alloy the powders of the spray dried powder, and remove carbon, nitrogen and oxygen atoms, wherein thermally treating the spray dried powder forms at least some partially sintered Mo-Si-B alloy powder pieces. The partially sintered Mo-Si-B alloy powder pieces are then milled in a milling container having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material to break down the pieces, and the milled Mo-Si-B alloy powder pieces are sieved to reclaim the Mo-Si-B alloy powder.

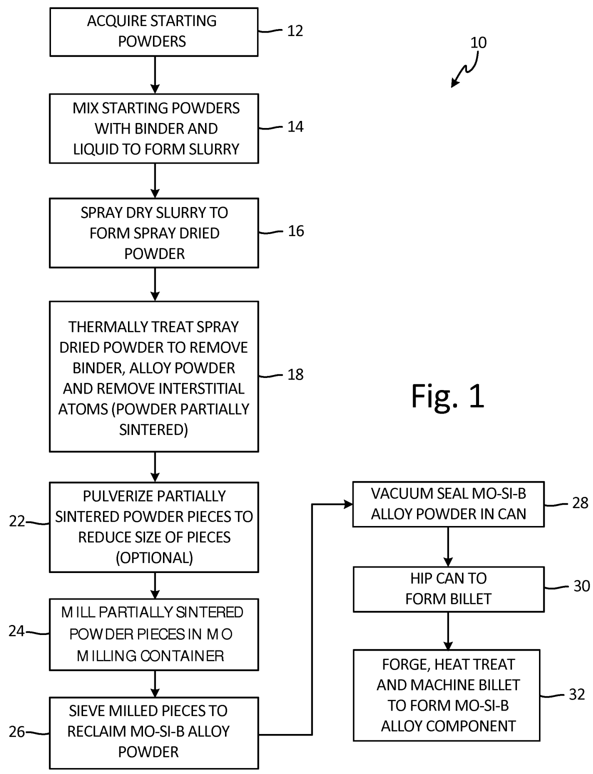

Fig. 1

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Description

BACKGROUND

[0001] Molybdenum (Mo) has excellent high temperature strength which makes it attractive for structural applications at elevated temperatures. The utility of Mo and Mo based alloys however are often limited by their poor elevated temperature oxidation resistance. In an oxidizing environment, the first oxidation product that forms is molybdenum trioxide (MoO_3). MoO_3 has a high vapor pressure and sublimes at substantial rates above 1000°F (538°C), resulting in accelerated metal loss from the alloy. Mo and Mo-based alloys are therefore largely limited to use in non-oxidizing environments at elevated temperatures without some form of externally applied oxidation protective coating.

[0002] Commonly owned U.S. Patent Nos. 5,595,616, 5,693,156 and 6,652,674 (incorporated by reference herein in their entireties) disclose high temperature oxidation resistant Mo-Si-B alloys. In these alloys the silicon (Si) and boron (B) which remain after the initial MoO_3 surface layer volatilizes, oxidize to form a protective borosilicate based oxide scale. If properly processed, these alloys can exhibit mechanical properties similar or superior to other Mo-based alloys while also maintaining good oxidation resistance at elevated temperatures of 1500°F to 2500°F (816°C to 1371°C). This combination of mechanical properties and oxidation resistance makes these materials very attractive for high temperature structural applications.

[0003] Mo-Si-B alloys formed from some powder precursors require handling in the proper environment and/or thermal treatments to achieve the desired microstructure/phase assembly and/or interstitial element levels. This thermal processing can result in partial sintering of the material, forming pieces that are more difficult to process into a final product than a fine Mo-Si-B alloy powder.

SUMMARY

[0004] A method of forming Mo-Si-B alloy powder includes preparing a mixture that includes Mo powder, Si_3N_4 powder and BN powder, adding a polymer binder and liquid to the mixture to form a slurry, spray drying the slurry to form a spray dried powder containing Mo powder, Si_3N_4 powder and BN powder particles, and thermally treating the spray dried powder to remove the binder, alloy the powders of the spray dried powder, and remove carbon, nitrogen and oxygen atoms, wherein thermally treating the spray dried powder forms at least some partially sintered Mo-Si-B alloy powder pieces. The partially sintered Mo-Si-B alloy powder pieces are then milled in a milling container having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material to break down the pieces, and the milled Mo-Si-B alloy powder pieces are sieved to reclaim the Mo-Si-B alloy powder.

[0005] A milling assembly for milling partially sintered Mo-Si-B alloy powder pieces to form Mo-Si-B alloy powder includes a milling container configured to receive the partially sintered Mo-Si-B alloy powder pieces. The milling container has contact surfaces being composed of Mo-based material or lined and/or coated with Mo-based material. In some embodiments, a milling media is provided inside or complementary to the milling container, the milling media having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material and configured to break down the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder.

BRIEF DESCRIPTION OF THE DRAWINGS

[0006]

FIG. 1 is a flow diagram showing a sequence of manufacturing steps for a Mo-Si-B alloy powder and a component formed of Mo-Si-B alloy powder.

FIG. 2 is an exploded view of a milling assembly that includes a Mo milling container (which has contact surfaces that may be composed of Mo-based material or lined and/or coated with Mo-based material) that may be used to mill partially sintered Mo-Si-B powder pieces.

DETAILED DESCRIPTION

[0007] Mo-Si-B alloys are attractive candidates for next generation high temperature structural materials in gas turbine engines. The alloys of interest include α-Mo solid solutions and intermetallic phases such as Mo_5SiB_2, Mo_5Si_3, Mo_3Si, Mo_2B and others depending on alloying elements added to increase high temperature mechanical properties, oxidation resistance and other properties. An example fabrication process in accordance with embodiments of the present disclosure for bulk components utilizes powder precursor materials. A manufacturing process 10 for forming Mo-Si-B alloy powder and a component formed of Mo-Si-B alloy powder according to an embodiment of the present disclosure is shown in FIG. 1. In an embodiment, the starting powders may include Mo powder, Si_3N_4 powder, BN powder, and other alloying elements (step 12). In the next step, the powders may be mixed with a binder, such as an organic binder, and a suitable liquid to form a slurry (step 14). Examples of suitable liquids include, without limitation, acetone, water, isopropyl alcohol, ethanol and mixtures thereof. Examples of binders, without limitation, are polymethyl methacrylate (PMMA), polyvinyl alcohol (PVA), polyethyl methacrylate (PEMA), and hydroxypropylcellulose.

[0008] The slurry may be spray dried to form spray dried powder containing Mo powder, Si_3N_4 powder, BN powder and other alloy powder (step 16). The powder may then be thermally treated in an appropriately controlled environment (step 18), with the times and temperatures of the thermal treatment being controlled so that the binder is removed, and so that the constituent powders are alloyed to form Mo-Si-B alloy powder and undesirable atoms such as carbon, nitrogen and oxygen are removed. As appreciated by those skilled in the art, various thermal treatments can be employed without departing from the scope of the present disclosure. For example, thermal treatments as discussed in the following document can be used: "Fabrication, strength and oxidation of molybdenum-silicon-boron alloys from reaction synthesis", Middlemas, Michael Robert (2009) (Doctoral dissertation), retrieved from Georgia Tech University SMARTech libary, https://smartech.gatech.edu/handle/1853/28253. During thermal treatment, it is possible for the Mo-Si-B powder to partially sinter together. These partially sintered Mo-Si-B powder pieces need to be separated back into a relatively fine powder in order to be used for certain applications, such as being formed into a billet for producing a Mo-Si-B component, for example.

[0009] In order to transform the partially sintered Mo-Si-B powder pieces into powder, it may be necessary or desirable to first pulverize large partially sintered Mo-Si-B powder pieces to form smaller pieces of partially sintered alloy powder (optional step 22). Alternatively, in some situations, the partially sintered Mo-Si-B powder pieces may be sufficiently small that this step is not needed, or later processing steps may not require the pieces to be particularly small. For pulverizing to be performed without contaminating the partially sintered material, an alumina mortar and pestle may be used, for example, possibly in an inert atmosphere in some embodiments. In an example process, the pieces may be pulverized into smaller pieces for further processing that are about 0.20 to about 0.62 inch (5.1 to 15.8 mm) in diameter, although other size ranges may be used in other embodiments. To prevent oxidation and other atmospheric contamination, the pieces may be stored in a container under an inert atmosphere in some embodiments, such as an argon atmosphere in one particular example.

[0010] To further process the reclamation of Mo-Si-B alloy powder from the partially sintered pieces, the pieces may be milled (step 24). The milling process may be performed in a number of different ways and with different types of equipment, and generally involves a milling container in which the partially sintered pieces are placed and milled, either with a milling media or by self-milling. In order to prevent contamination of the Mo-Si-B alloy powder during milling, the milling container and any milling media that is employed are either composed of Mo-based material or have contact surfaces that are lined and/or coated with Mo-based material. Examples of milling processes that may be employed include jar milling, mortar grinding, planetary ball milling, and attritor milling, among others. Jar milling (e.g., where the milling container is a jar composed of a Mo-based material or a jar having inner contact surfaces lined and/or coated with Mo-based material) is an example of a suitable milling process in which jars are rotated on automated rollers and the partially sintered Mo-Si-B powder pieces self-mill with no other milling media present. In other embodiments, milling media is included inside or complementary to the milling container to break down the partially sintered Mo-Si-B powder pieces. By forming the contact surfaces of the milling container and any milling media out of Mo-based material or lining and/or coating the contact surfaces of the milling container and any milling media with Mo-based material, levels of contamination in the Mo-Si-B powder after milling can be minimized. For example, in some tested embodiments, contamination of Mo-Si-B powder produced as described herein can be maintained below 0.08wt/% carbon and 0.06wt/% oxygen, or more particularly below 0.006wt% carbon and 0.01wt% oxygen, and even more particularly below 0.003wt% carbon and 0.004wt% oxygen, for example. The term "contact surfaces" as used herein refers to surfaces of the milling container and milling media that come into contact with the pieces being milled during a milling operation (and therefore would have the potential to introduce contamination to the pieces being milled).

[0011] During the milling process, milling may be periodically interrupted and the powder sieved to reclaim fine powder from larger pieces (step 26). Sieving with a coarse sieve, such as a 16 mesh sieve for example (although other sizes may be used), may separate milled Mo-Si-B alloy powder pieces over a threshold size (for example, particles over 0.05 inch (1.27 mm) in diameter) for further milling. A finer sieve, such as a 120 mesh sieve for example (although other sizes may be used), may then separate particles to obtain a desired (e.g., final) powder size, such as by separating particles smaller than 4.7 micro inches (0.12 microns) in diameter that form the alloy powder, in a specific non-limiting example.

[0012] Once the Mo-Si-B alloy powder has been reclaimed, it can be used in a number of applications, such as for the formation of a Mo-Si-B alloy component. In such a formation process, the Mo-Si-B alloy powder may be consolidated by sealing the powder in a can under vacuum (step 28) and then hot isostatically pressing (HIPing) the powder to form a billet (step 30). The billet may then be thermal mechanically processed, heat treated, and machined to form the Mo-Si-B alloy component (step 32).

[0013] An exploded view of an exemplary milling assembly, that includes a Mo milling container (which has contact surfaces that may be composed of Mo-based material or lined and/or coated with Mo-based material) that may be used to mill partially sintered Mo-Si-B powder pieces, is shown in FIG. 2. In the embodiment shown in FIG. 2, the milling container is a milling jar 40 composed of Mo-based material that includes cylindrical body 42, defining a cavity C therein, end flanges 44 and 46 and end caps 48 and 50. In various embodiments, body 42, end flanges 44 and 46, and end caps 48 and 50 of milling jar 40 may be composed of Mo-based material or lined and/or coated with Mo-based material so that surfaces coming into contact with partially sintered Mo-Si-B powder pieces to be milled do not introduce contamination to the pieces during the milling process. End caps 48 and 50 are attached to flanges 44 and 46 by bolts 52 and nuts 54, although other attachment or securing mechanisms can be used without departing from the scope of the present disclosure. The ends of milling jar 40 are hermetically sealed by O-rings 56, although other sealing mechanisms can be used without departing from the scope of the present disclosure. The cylindrical body 42 has an outer diameter D1 and an inner diameter D2. Milling jar 40 (cylindrical body 42 plus end flanges 44, 46 and end caps 48, 50) has a length L1, as shown in FIG. 2. In one non-limiting embodiment, the outer diameter D1 of cylindrical body 42 can be 5.8 inches (14.7 cm), and overall length L1 with the end caps on can be 8.5 inches (21.6 cm). Further, in some embodiments, the inner diameter D2 of the cylindrical body can be 5.3 inches (13.5 cm). Those of skill in the art will appreciate that the dimensions provided herein are merely for illustrative purposes, and various sizes, dimensions, shapes and/or configurations can be employed without departing from the scope of the present disclosure.

[0014] Using milling jar 40 composed of Mo-based material or lined and/or coated with Mo-based material, the partially sintered Mo-Si-B powder pieces may be self-milled with no added milling media, which helps to ensure that the Mo-Si-B powder is not contaminated during the milling process. In an exemplary embodiment, milling may be carried out under an inert atmosphere.

[0015] Another example of a milling assembly that may be used to mill partially sintered Mo-Si-B powder pieces is an apparatus employing a mortar as the milling container and a pestle as the milling media. In this example, the contact surfaces of the mortar and the pestle are composed of Mo-based material or lined and/or coated with Mo-based material. Such an apparatus may be an automatic mortar grinder or a manually operated mortar and pestle, where the pestle and mortar function complementary to one another to grind the partially sintered Mo-Si-B powder pieces between them to break down the pieces and form the Mo-Si-B alloy powder.

[0016] A further example of a milling assembly that may be used to mill partially sintered Mo-Si-B powder pieces is a planetary ball mill, in which contact surfaces of a milling container and one or more balls (employed as a milling media inside the milling container) are composed of Mo-based material or lined and/or coated with Mo-based material.

[0017] Yet another example of a milling assembly that may be used to mill partially sintered Mo-Si-B powder pieces is an attritor mill, in which contact surfaces of a milling container and rotating attritor milling media inside the milling container are composed of Mo-based material or lined and/or coated with Mo-based material.

[0018] As used herein, the disclosure of contact surfaces of the milling container and/or milling media being composed of Mo-based material or lined and/or coated with Mo-based material is intended to include the use of pure (100%) molybdenum for these contact surfaces, as well as phases of molybdenum borosilicates, molybdenum silicides and other Mo-Si-B chemistries. Chemistry examples may include but are not limited to: $MoSi_2$, Mo_5SiB_2, Mo_3Si and Mo-Si-B chemistries called out in U.S. Patent Nos. 5,595,616, 5,693,156 and 6,652,674, for example.

[0019] Although several examples of milling assemblies have been disclosed, it should be understood that other milling assemblies may be used in which a milling container and milling media each have contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material, in order to minimize contamination of Mo-Si-B powder produced in the manner disclosed herein, and the present disclosure is not limited to the examples disclosed.

Discussion of Possible Embodiments

[0020] The following are non-exclusive descriptions of possible embodiments of the present invention.

[0021] A method of forming Mo-Si-B alloy powder includes: preparing a mixture that includes Mo powder, Si3N4 powder and BN powder; adding a polymer binder and liquid to the mixture to form a slurry; spray drying the slurry to form a spray dried powder containing Mo powder, Si3N4 powder and BN powder particles; thermally treating the spray dried powder to remove the binder, alloy the powders of the spray dried powder, and remove carbon, nitrogen and oxygen atoms, wherein thermally treating the spray dried powder forms at least some partially sintered Mo-Si-B alloy powder pieces; milling the partially sintered Mo-Si-B alloy powder pieces in a milling container (e.g. as herein described) having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material to break down the pieces; and sieving the milled Mo-Si-B alloy powder pieces to reclaim the Mo-Si-B alloy powder.

[0022] The method of the preceding paragraph can optionally include, additionally and/or alternatively any, one or more of the following features, configurations and/or additional components:

[0023] Milling the partially sintered Mo-Si-B alloy powder pieces may include jar milling the partially sintered Mo-Si-B alloy powder pieces in a jar composed of, or having contact surfaces composed, of Mo-based material or lined and/or coated with Mo-based material with no other milling media, to break down the pieces by self-milling in the jar.

[0024] Milling the partially sintered Mo-Si-B alloy powder pieces may include operation of a milling media having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material that breaks down the pieces in the milling container.

[0025] Pulverizing the partially sintered Mo-Si-B alloy powder pieces to reduce a size of the partially sintered Mo-Si-B alloy powder pieces that are milled in the milling container may be performed.

[0026] The partially sintered Mo-Si-B alloy powder pieces that are milled in the milling container may be about 0.20 to 0.62 in (5.1 to 15.8 mm) in diameter.

[0027] The binder may be polymethyl methacrylate (PMMA), polyvinyl alcohol (PVA), polyethyl methacrylate (PEMA), or hydroxypropylcellulose.

[0028] The liquid may be acetone, water, isopropyl alcohol (IPA), ethanol, or mixtures thereof.

[0029] Sieving the milled Mo-Si-B alloy powder pieces may include sieving with a coarse sieve to separate milled Mo-Si-B alloy powder pieces over a threshold size for further milling, and sieving with a fine sieve to collect Mo-Si-B alloy powder.

[0030] The threshold size of Mo-Si-B powder pieces separated by the coarse sieve may be 0.05 in (1.27 mm) in diameter, and particles of the Mo-Si-B alloy powder may have a diameter smaller than 4.7 micro inches (0.12 microns).

[0031] The Mo-Si-B alloy powder may formed with contamination, e.g. interstitial contamination, that is less than 0.08wt/% carbon and 0.06wt/% oxygen.

[0032] The Mo-Si-B alloy powder may be formed with contamination, e.g. interstitial contamination, that is less than 0.006wt% carbon and 0.01wt% oxygen.

[0033] The Mo-Si-B alloy powder may be formed with contamination, e.g. interstitial contamination, that is less than 0.003wt% carbon and 0.004wt% oxygen.

[0034] A method of forming a Mo-Si-B component includes producing Mo-Si-B powder by a method including any of the features, configurations and/or additional components listed above; sealing the Mo-Si-B powder under vacuum in a can; hot isostatic pressing (HIP) the can to form a billet; and forging, heat treating and machining the billet to form the Mo-Si-B alloy component.

[0035] A milling assembly for milling partially sintered Mo-Si-B alloy powder pieces to form Mo-Si-B alloy powder (e.g. in a method as herein described) includes: a milling container configured to receive the partially sintered Mo-Si-B alloy powder pieces, the milling container having contact surfaces being composed of Mo-based material or lined and/or coated with Mo-based material.

[0036] The milling assembly of the preceding paragraph can optionally include, additionally and/or alternatively any one or more of the following features, configurations or additional components:

[0037] The milling container may include a jar having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material, configured to self-mill the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder in ajar milling operation.

[0038] The milling assembly may further include milling media inside or complementary to the milling container, the milling media having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material and configured to break down the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder.

[0039] The milling container may be a mortar and the milling media may be a pestle, the mortar and pestle having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

[0040] The mortar and pestle may be configured as an automatic mortar grinder.

[0041] The milling container and the milling media may together form a planetary ball mill having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

[0042] The milling container and the milling media may together form an attritor mill having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

[0043] Preferred embodiments of the present disclosure are as follows:

1. A method of forming Mo-Si-B alloy powder comprising:

    preparing a mixture that includes Mo powder, Si3N4 powder and BN powder;
    adding a polymer binder and liquid to the mixture to form a slurry;
    spray drying the slurry to form a spray dried powder containing Mo powder, Si3N4 powder and BN powder particles;
    thermally treating the spray dried powder to remove the binder, alloy the powders of the spray dried powder, and remove carbon, nitrogen and oxygen atoms, wherein thermally treating the spray dried powder forms at least some partially sintered Mo-Si-B alloy powder pieces;
    milling the partially sintered Mo-Si-B alloy powder pieces in a milling container having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material to break down the pieces; and
    sieving the milled Mo-Si-B alloy powder pieces to reclaim the Mo-Si-B alloy powder.

2. The method of embodiment 1, wherein milling the partially sintered Mo-Si-B alloy powder pieces comprises jar milling the partially sintered Mo-Si-B alloy powder pieces in ajar composed of Mo-based material or lined and/or coated with Mo-based material with no other milling media, to break down the pieces by self-milling in the jar.

3. The method of embodiment 1, wherein milling the partially sintered Mo-Si-B alloy powder pieces comprises operation of a milling media composed of Mo-based material or lined and/or coated with Mo-based material that breaks down the pieces in the milling container.

4. The method of embodiment 1, further comprising:

pulverizing the partially sintered Mo-Si-B alloy powder pieces to reduce a size of the partially sintered Mo-Si-B alloy powder pieces that are milled in the milling container.

5. The method of embodiment 4, wherein the partially sintered Mo-Si-B alloy powder pieces that are milled in the milling container are about 0.20 to 0.62 in (5.1 to 15.8 mm) in diameter.

6. The method of embodiment 1, wherein the binder comprises polymethyl methacrylate (PMMA), polyvinyl alcohol (PVA), polyethyl methacrylate (PEMA), or hydroxypropylcellulose.

7. The method of embodiment 1 wherein the liquid comprises acetone, water, isopropyl alcohol (IPA), ethanol, or mixtures thereof.

8. The method of embodiment 1, wherein sieving the milled Mo-Si-B alloy powder pieces comprises sieving with a coarse sieve to separate milled Mo-Si-B alloy powder pieces over a threshold size for further milling, and sieving with a fine sieve to collect Mo-Si-B alloy powder.

9. The method of embodiment 8, wherein the threshold size of Mo-Si-B powder pieces separated by the coarse sieve is 0.05 in (1.27 mm) in diameter, and particles of the Mo-Si-B alloy powder have a diameter smaller than 4.7 micro inches (0.12 microns).

10. The method of embodiment 1, wherein the Mo-Si-B alloy powder is formed with contamination that is less than 0.08wt/% carbon and 0.06wt/% oxygen.

11. The method of embodiment 10, wherein the Mo-Si-B alloy powder is formed with contamination that is less than 0.006wt% carbon and 0.01wt% oxygen.

12. The method of embodiment 11, wherein the Mo-Si-B alloy powder is formed with interstitial contamination that is less than 0.003wt% carbon and 0.004wt% oxygen.

13. A method of forming a Mo-Si-B alloy component comprising:

producing Mo-Si-B alloy powder by the method of embodiment 1;
sealing the Mo-Si-B alloy powder under vacuum in a can;
hot isostatic pressing (HIP) the can to form a billet; and
forging, heat treating and machining the billet to form the Mo-Si-B alloy component.

14. A milling assembly for milling partially sintered Mo-Si-B alloy powder pieces to form Mo-Si-B alloy powder, the milling assembly comprising:

a milling container configured to receive the partially sintered Mo-Si-B alloy powder pieces, the milling container having contact surfaces being composed of Mo-based material or lined and/or coated with Mo-based material.

15. The milling assembly of embodiment 14, wherein the milling container comprises a jar having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material, configured to self-mill the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder in a jar milling operation.

16. The milling assembly of embodiment 14, further comprising:

milling media inside or complementary to the milling container, the milling media having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material and configured to break down the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder.

17. The milling assembly of embodiment 16, wherein the milling container comprises a mortar and the milling media comprises a pestle, the mortar and pestle having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

18. The milling assembly of embodiment 17, wherein the mortar and pestle are configured as an automatic mortar grinder.

19. The milling assembly of embodiment 16, wherein the milling container and the milling media together form a planetary ball mill having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

20. The milling assembly of embodiment 16, wherein the milling container and the milling media together form an attritor mill having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

[0044] While the invention has been described with reference to an exemplary embodiment(s), it will be understood by those skilled in the art that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the invention. In addition, many modifications may be made to adapt a particular situation or material to the teachings of the invention without departing from the essential scope thereof. Therefore, it is intended that the invention not be limited to the particular embodiment(s) disclosed, but that the invention will include all embodiments falling within the scope of the appended claims.

Claims

1. A method of forming Mo-Si-B alloy powder comprising:

   preparing a mixture that includes Mo powder, Si_3N_4 powder and BN powder;
   adding a polymer binder and liquid to the mixture to form a slurry;
   spray drying the slurry to form a spray dried powder containing Mo powder, Si_3N_4 powder and BN powder particles;
   thermally treating the spray dried powder to remove the binder, alloy the powders of the spray dried powder, and remove carbon, nitrogen and oxygen atoms, wherein thermally treating the spray dried powder forms at least some partially sintered Mo-Si-B alloy powder pieces;
   milling the partially sintered Mo-Si-B alloy powder pieces in a milling container having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material to break down the pieces; and
   sieving the milled Mo-Si-B alloy powder pieces to reclaim the Mo-Si-B alloy powder.

2. The method of claim 1, wherein milling the partially sintered Mo-Si-B alloy powder pieces comprises jar milling the partially sintered Mo-Si-B alloy powder pieces in a jar composed of Mo-based material or lined and/or coated with Mo-based material with no other milling media, to break down the pieces by self-milling in the jar.

3. The method of any preceding claim, wherein milling the partially sintered Mo-Si-B alloy powder pieces comprises operation of a milling media composed of Mo-based material or lined and/or coated with Mo-based material that breaks down the pieces in the milling container.

4. The method of any preceding claim, further comprising:

   pulverizing the partially sintered Mo-Si-B alloy powder pieces to reduce a size of the partially sintered Mo-Si-B alloy powder pieces that are milled in the milling container, and/or
   wherein the partially sintered Mo-Si-B alloy powder pieces that are milled in the milling container are about 0.20 to 0.62 in (5.1 to 15.8 mm) in diameter.

5. The method of any preceding claim, wherein the binder comprises polymethyl methacrylate (PMMA), polyvinyl alcohol (PVA), polyethyl methacrylate (PEMA), or hydroxypropylcellulose.

6. The method of any preceding claim wherein the liquid comprises acetone, water, isopropyl alcohol (IPA), ethanol, or mixtures thereof.

7. The method of any preceding claim, wherein sieving the milled Mo-Si-B alloy powder pieces comprises sieving with a coarse sieve to separate milled Mo-Si-B alloy powder pieces over a threshold size for further milling, and sieving with a fine sieve to collect Mo-Si-B alloy powder, preferably
   wherein the threshold size of Mo-Si-B powder pieces separated by the coarse sieve is 0.05 in (1.27 mm) in diameter, and particles of the Mo-Si-B alloy powder have a diameter smaller than 4.7 micro inches (0.12 microns).

8. The method of any preceding claim, wherein the Mo-Si-B alloy powder is formed with contamination that is less than 0.08wt/% carbon and 0.06wt/% oxygen, preferably
   wherein the Mo-Si-B alloy powder is formed with contamination that is less than 0.006wt% carbon and 0.01wt% oxygen.

9. The method of any preceding claim, wherein the Mo-Si-B alloy powder is formed with interstitial contamination that is less than 0.003wt% carbon and 0.004wt% oxygen.

10. A method of forming a Mo-Si-B alloy component comprising:

    producing Mo-Si-B alloy powder by the method of any preceding claim;
    sealing the Mo-Si-B alloy powder under vacuum in a can;
    hot isostatic pressing (HIP) the can to form a billet; and
    forging, heat treating and machining the billet to form the Mo-Si-B alloy component.

11. A milling assembly for milling partially sintered Mo-Si-B alloy powder pieces to form Mo-Si-B alloy powder, the milling assembly comprising:

    a milling container configured to receive the partially sintered Mo-Si-B alloy powder pieces, the milling container having contact surfaces being composed of Mo-based material or lined and/or coated with Mo-based material.

12. The milling assembly of claim 11, wherein the milling container comprises a jar having contact surfaces composed of Mo-based material or lined and/or coated with Mo-based material, configured to self-mill the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder in ajar milling operation.

13. The milling assembly of claim 11 or claim 12, further comprising:

milling media inside or complementary to the milling container, the milling media having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material and configured to break down the partially sintered Mo-Si-B alloy powder pieces to form the Mo-Si-B alloy powder.

14. The milling assembly of any one of claims 11-13, wherein the milling container comprises a mortar and the milling media comprises a pestle, the mortar and pestle having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material, preferably
wherein the mortar and pestle are configured as an automatic mortar grinder.

15. The milling assembly of any one of claims 11-14, wherein the milling container and the milling media together form a planetary ball mill having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material, and/or wherein the milling container and the milling media together form an attritor mill having contact surfaces that are composed of Mo-based material or lined and/or coated with Mo-based material.

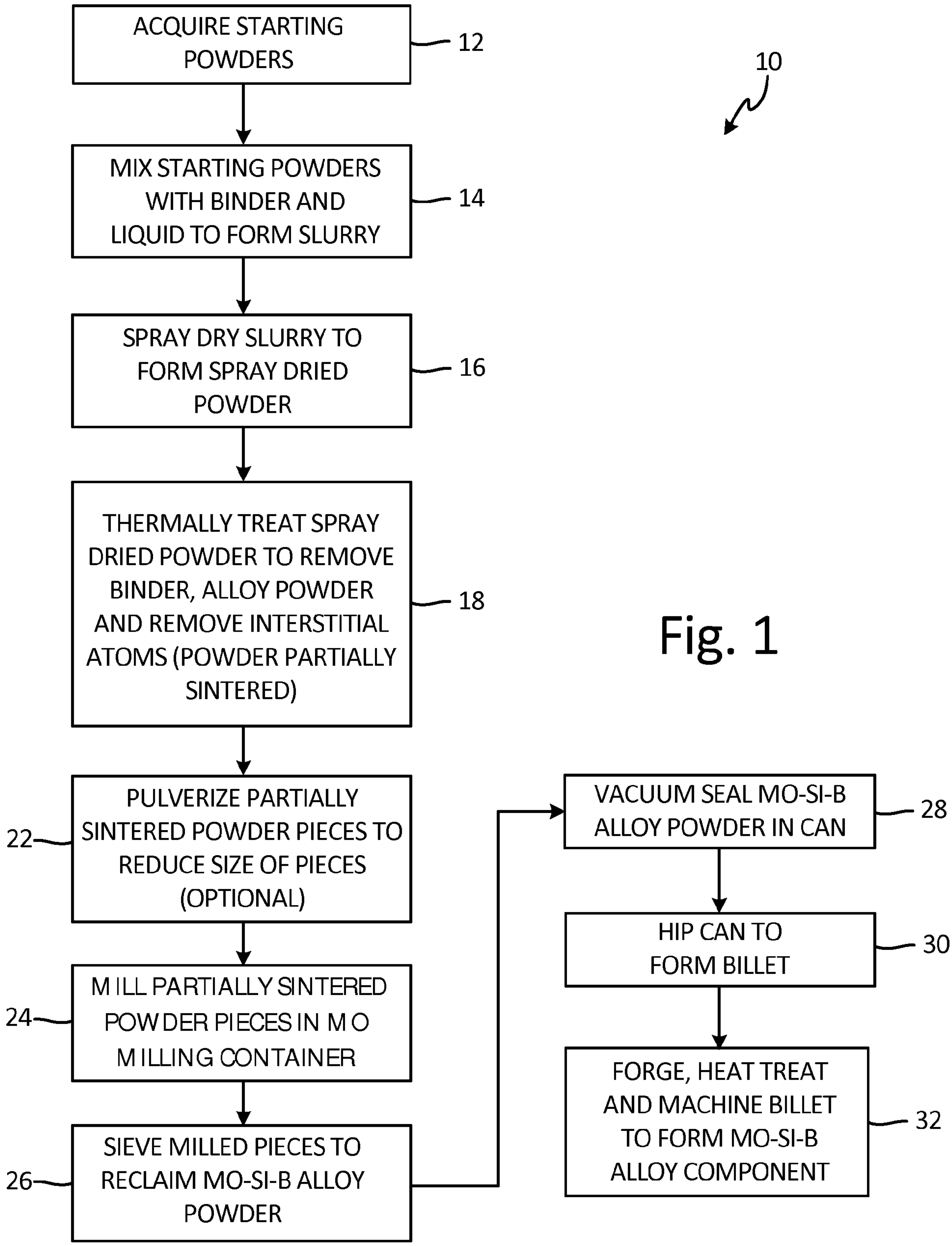
ACQUIRE STARTING POWDERS
12
10
MIX STARTING POWDERS WITH BINDER AND LIQUID TO FORM SLURRY
14
SPRAY DRY SLURRY TO FORM SPRAY DRIED POWDER
16
THERMALLY TREAT SPRAY DRIED POWDER TO REMOVE BINDER, ALLOY POWDER AND REMOVE INTERSTITIAL ATOMS (POWDER PARTIALLY SINTERED)
18
22
PULVERIZE PARTIALLY SINTERED POWDER PIECES TO REDUCE SIZE OF PIECES (OPTIONAL)
24
MILL PARTIALLY SINTERED POWDER PIECES IN MO MILLING CONTAINER
26
SIEVE MILLED PIECES TO RECLAIM MO-SI-B ALLOY POWDER
VACUUM SEAL MO-SI-B ALLOY POWDER IN CAN
28
HIP CAN TO FORM BILLET
30
FORGE, HEAT TREAT AND MACHINE BILLET TO FORM MO-SI-B ALLOY COMPONENT
32

Fig. 1

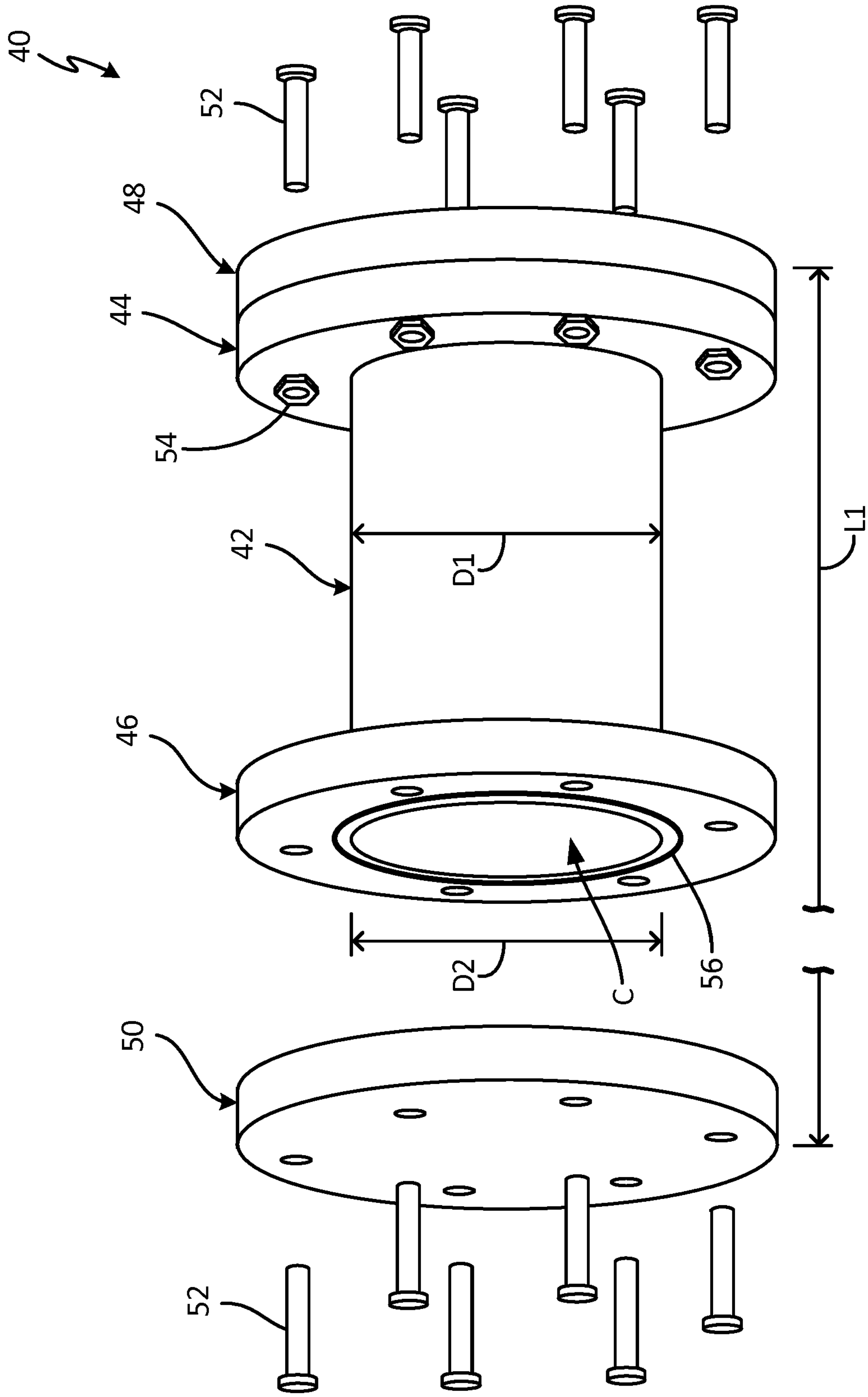

Fig. 2

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EUROPEAN SEARCH REPORT

Application Number
EP 17 17 5229

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X Y	US 2009/011266 A1 (COCHRAN JOE K [US] ET AL) 8 January 2009 (2009-01-08) * paragraphs [0025], [0035] - [0039] * -----	1,2,4,6, 7,10 3	TECHNICAL FIELDS SEARCHED (IPC)
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The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
The Hague	25 October 2017	Forestier, Gilles

CATEGORY OF CITED DOCUMENTS

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EUROPEAN SEARCH REPORT

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Place of search	Date of completion of the search	Examiner
The Hague	25 October 2017	Forestier, Gilles

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page 2 of 2

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