Disclosed is a method of producing an object, the object being made of metal or an alloy, having a desired shape and being non-porous, the method comprising: providing some metal or alloy having a first average solute level; using the provided metal or alloy, performing a net-shape or near-net shape manufacturing process to produce an intermediate object, the intermediate object having the desired shape, being non-porous, and having a second average solute level, the second average solute level being greater than or equal to the first average solute level; and performing a solute level changing process on the intermediate object to change the solute level of at least the bulk of the intermediate object such as to provide the intermediate object with a third average solute level, thereby providing the object, the third average solute level being different to the second.
START

FORM COMPONENT

PERFORM OXYGEN REDUCTION PROCESS ON COMPONENT

END

FIG. 1
PERFORM METAL INJECTION MOULDING PROCESS TO PRODUCE A "GREEN PART"

REMOVE BINDER MATERIAL FROM GREEN PART TO PRODUCE A "BROWN PART"

SINTER BROWN PART TO FORM COMPONENT

FIG. 2
FIG. 4

KEY

- HIGH OXYGEN LEVEL
- MEDIUM OXYGEN LEVEL
- LOW OXYGEN LEVEL
PLACE SUBSTANCE IN DE-OXIDATION CHAMBER

PLACE COMPONENT IN DE-OXIDATION CHAMBER

SEAL DE-OXIDATION CHAMBER

FOR FIRST PRE-DETERMINED TIME PERIOD, HEAT DE-OXIDATION CHAMBER SUCH THAT SUBSTANCE IS VAPOURISED

LEAVE DE-OXIDATION CHAMBER TO COOL

REMOVE COMPONENT FROM DE-OXIDATION CHAMBER

CLEAN COMPONENT

FIG. 6
FIG. 7
PLACE COMPONENT IN RE-OXIDATION CHAMBER

PLACE FURTHER SUBSTANCE IN RE-OXIDATION CHAMBER

SEAL RE-OXIDATION CHAMBER

FOR SECOND PRE-DETERMINED TIME PERIOD, HEAT RE-OXIDATION CHAMBER SUCH THAT FURTHER SUBSTANCE IS VAPOURISED

LEAVE RE-OXIDATION CHAMBER TO COOL

REMOVE COMPONENT FROM RE-OXIDATION CHAMBER

CLEAN COMPONENT

FIG. 9
FIG. 10

KEY
- HIGH OXYGEN LEVEL
- MEDIUM OXYGEN LEVEL
- LOW OXYGEN LEVEL
PLACE COMPONENT IN HOMOGENISATION CHAMBER

FILL HOMOGENISATION CHAMBER WITH INERT GAS

FOR THIRD PRE-DETERMINED TIME PERIOD, HEAT THE COMPONENT WITHIN THE HOMOGENISATION CHAMBER

LEAVE COMPONENT TO COOL

FIG. 11
START

PARTIALLY FORM COMPONENT

PERFORM OXYGEN REDUCTION PROCESS ON PARTIALLY FORMED COMPONENT

PERFORM SINTERING PROCESS TO FULLY SINTER COMPONENT

END

FIG. 13
PERFORM METAL INJECTION MOULDLING PROCESS TO PRODUCE A "GREEN PART"

REMOVE BINDER MATERIAL FROM GREEN PART TO PRODUCE A "BROWN PART"

PARTIALLY SINTER BROWN PART

FIG. 14
PRODUCTION OF METAL OR ALLOY OBJECTS
CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application is the National Stage of International Application No. PCT/GB2012/000494, filed Jun. 6, 2012, which claims the benefit of and priority to GB 1205372.9, filed Mar. 1, 2012 and GB 1109914.0, filed Jun. 13, 2011, the contents of all of which are incorporated by reference as if fully set forth herein.

FIELD OF THE INVENTION

[0002] The present invention relates to the production of objects, in particular objects made of metals or alloys.

BACKGROUND

[0003] Titanium alloys are used in many market sectors, including the aerospace, medical and sports and leisure sectors.

[0004] Typically, the manufacture of titanium alloy parts, or components, is performed by machining processes or a combination of forging and machining processes. Some parts are also made using casting and powder metallurgy routes.

[0005] Typically, titanium alloys contain oxygen in solid solution. Oxygen tends to strengthen and harden the alloy. However, if there is too high an oxygen level in the alloy, toughness and ductility may be reduced.

[0006] Titanium has a relatively strong affinity for absorbing oxygen. Many processing operations performed when producing a titanium alloy component, including forging and heat treatment, tend to introduce oxygen into the alloy. However, this introduction of oxygen is usually restricted to a thin region near the surface of the component. This layer may be machined off or removed in some other way if desired.

[0007] Powder metallurgy manufacture tends to provide a number of advantages over alternative routes. For example, near-net-shape components can be produced with very little wastage. Also, relatively complex shapes that may be prohibitively expensive to machine can be made relatively easily.

[0008] However, high quality titanium alloy powder tends to be relatively expensive. The cost of the powder tends to be a very significant proportion of the total manufacturing cost.

[0009] Cheaper alloy powders are available. These tend not to be suitable for production of structural parts due to their relatively high oxygen content.

[0010] Also, due to the relatively high surface area of the titanium alloy powder, oxygen absorption tends to be a particular problem when using titanium alloy powders e.g. in the production of the powder, in the handling of the powder, and in the hot compaction or selective melting of the powder into a solid material.

[0011] Titanium alloy powders may be chemically treated at an elevated temperature to reduce oxygen content. Similar methods have been used on titanium alloy components to remove oxygen contamination on, or just below, the surface of those components.

[0012] However, for titanium powders, such elevated temperature chemical treatments tend not to be applicable for smaller powder sizes (e.g. those used in metal injection moulding) because of the strong tendency of the powder particles to sinter.

SUMMARY OF THE INVENTION

[0013] US 2005 139483 discloses a method of purifying a metal salt. This method comprises bringing the metal salt formed by melting an alkali metal salt, an alkaline earth metal salt or a mixture thereof into contact with one or more of titanium, titanium alloy, zirconium and zirconium alloy. The metal salt is melted in a vessel made of titanium or titanium alloy, or a vessel lined with titanium or titanium alloy.


[0015] GB 718211 discloses treating a titanium sheet in a calcium bath at a temperature of 1000° C.

[0016] GB 608753 discloses a process for the purification of titanium containing oxygen which comprises treating the titanium with calcium or calcium hydride at a temperature not less than 900° C. in a fluid medium.

[0017] GB 1450039 discloses a method of removing oxygen from a metal powder that has been compacted into a shape. Carbon, which is contained in the metal powder, is used to remove oxygen. However, this process has to be carried out whilst the object has open porosity so that carbon oxides (gases) may be removed from the object. The deoxidation process tends not to be useful for full density or near full density objects. Furthermore, the disclosed methods would tend not to work for titanium or titanium alloy objects.

[0018] In a first aspect, the present invention provides a method of producing an object, the object being made of metal or an alloy, the object having a desired shape, the object being a non-porous object, the method comprising providing some metal or alloy, the provided metal or alloy having a first average solute level, using the provided metal or alloy, performing a net-shape or near-net-shape manufacturing process to produce an intermediate object, wherein the intermediate object has the desired shape, the intermediate object is a non-porous object, and the intermediate object has a second average solute level, the second average solute level being greater than or equal to the first average solute level, and performing a solute level changing process on the intermediate object to change the solute level of at least the bulk the intermediate object (i.e. the greater part of the body of the intermediate object) such as to provide the intermediate object with a third average solute level, thereby providing the object, wherein the third average solute level is different to the second average solute level.

[0019] The third average solute level may be lower than the first average solute level.

[0020] The solute level reduction process may be performed such that the intermediate object substantially retains its shape and solid density.

[0021] In a further aspect, the present invention provides an object produced by performing a method according to the first aspect.

[0022] In a further aspect, the present invention provides a method of treating an object, the object being made of a metal or alloy, and the object having a first average solute level, the method comprising performing a solute level changing process on the object to add or remove a solute from the surface of the object, thereby producing an object having a second average solute level and an increased gradient of the solute level, and performing a homogenisation process on the object.
having the second average solute level to reduce the gradient of the solute level within the object having the second average solute level.

[0023] In a further aspect, the present invention provides a method of producing an object with a second average solute level, wherein the second average solute level is equal to a desired level or within a desired range, the object being made of a metal or alloy, the method comprising, from a raw material having a first average solute level, forming an object to produce a formed object, performing a solute level changing process on the formed object to add or remove a solute from a surface of the formed object, thereby changing the average solute level of the formed object to the second average solute level, and performing a homogenisation process on the object having the second average solute level to diffuse the solute within the object having the second average solute level.

[0024] The solute level changing process may be performed to reduce an average solute level within the object.

[0025] The solute level changing process may comprise performing a solute level decreasing process, and performing a solute level increasing process, wherein the solute level decreasing process and the solute level increasing process are performed on the object prior to the homogenisation process being performed, the solute level decreasing process is performed to remove the solute from the surface of the object, thereby reducing the average solute level of the object, the solute increasing process is performed to add the solute to the surface of the object, thereby increasing the average solute level of the object, and an amount that the average solute level is reduced by during the solute level decreasing process is different to an amount that the average solute level is increased by during the solute increasing process.

[0026] The metal or alloy may comprise one or more of the following metals: titanium, tantalum, vanadium, zirconium, molybdenum, niobium.

[0027] The metal or alloy may be a titanium alloy.

[0028] The alloy may be Ti—6Al—4V.

[0029] The solute may be one of the following: oxygen, carbon, hydrogen or nitrogen.

[0030] The solute may be oxygen.

[0031] The second average solute level may be between 1000 and 2500 parts per million by weight.

[0032] The second average solute level may be between 1300 and 1800 parts per million by weight.

[0033] The second average solute level may be between 1700 and 1800 parts per million by weight.

[0034] The second average solute level may be less than 2000 parts per million by weight.

[0035] The second average solute level may be less than 1300 parts per million by weight.

[0036] The first average solute level may be between 2300 and 10000 parts per million by weight.

[0037] The first average solute level may be between 5000 and 10000 parts per million by weight.

[0038] The first average solute level may be greater than 10000 parts per million by weight.

[0039] The step of performing a homogenisation process may be performed such that the solute level within the object is substantially uniform throughout the object having the second average solute level.

[0040] The object may be made from a powder form of the alloy using a metal injection moulding process.

[0041] The step of performing the solute level changing process may comprise exposing at least a portion of the object to a fluid, wherein the fluid comprises a chemical reactant, and the chemical reactant has a different level of reactivity to the solute than the alloy.

[0042] The chemical reactant may be an alkali earth metal e.g. calcium.

[0043] The fluid may further comprise a flux material, the flux material being for facilitating the removal of a product of a reaction between the chemical reactant and the alloy from a surface of the object.

[0044] The flux material may be calcium chloride.

[0045] The fluid may be a vapour.

[0046] The step of performing the solute level changing process may comprise heating the object to between 1000°C and 1200°C.

[0047] The step of performing the homogenisation process may comprise heating the object to between 1000°C and 1300°C.

[0048] The heating of the object to between 1000°C and 1300°C may be performed for between 0.5 and 100 hours.

[0049] In a further aspect, the present invention provides apparatus for treating an object, the object being made of a metal or alloy, and the object having a first average solute level, the apparatus comprising means for performing a solute level changing process on the object to add or remove a solute from the surface of the object, thereby producing an object having a second average solute level and an increased gradient of the solute level, and means for performing a homogenisation process on the object having the second average solute level to reduce the gradient of the solute level within the object having the second average solute level.

[0050] In a further aspect, the present invention provides apparatus for producing an object with a second average solute level, wherein the second average solute level is equal to a desired level or within a desired range, the object being made of a metal or alloy, the apparatus comprising production means for, from a raw material having an first average solute level, forming an object to produce a formed object, means for performing a solute level changing process on the formed object to add or remove a solute from the surface of the formed object, thereby changing the average solute level of the object to the second average solute level, and means for performing a homogenisation process on the object having the second average solute level to diffuse the solute within the object having the second average solute level.

[0051] In a further aspect, the present invention provides a method of changing a concentration of a solute within an object, the object being made of a metal or alloy, the method comprising exposing at least a portion of the object to a vapour, wherein the vapour comprises a chemical reactant, the chemical reactant having a different level of reactivity to the solute than the metal or alloy, and a flux material, the flux material being for facilitating the removal of a product of a reaction between the chemical reactant in the vapour and the alloy from a surface of the object.

[0052] A product of a reaction between the chemical reactant in the vapour and the alloy may be dissolvable in the flux material.

[0053] The metal or alloy may comprise one or more of the following metals:

[0054] titanium, tantalum, vanadium, zirconium, molybdenum, niobium.

[0055] The metal or alloy may be a titanium alloy.

[0056] The metal or alloy may be Ti—6Al—4V.
The solute may be one of the following: oxygen, carbon, hydrogen or nitrogen.

The solute may be oxygen.

The concentration of the solute within the object may be changed to be within the range 1000 and 2300 parts per million by weight.

The concentration of the solute within the object may be changed from being within the range 5000 and 10000 parts per million by weight.

The chemical reactant may have a higher level of reactivity to the solute than the alloy.

The chemical reactant may be calcium.

The chemical reactant may have a lower level of reactivity to the solute than the alloy.

The chemical reactant may be calcium oxide.

The flux material may be calcium chloride.

The method may comprise heating the object to between 1000°C and 1200°C.

The object may be made from a powder form of the alloy using a metal injection moulding process.

The method may further comprise performing a homogenisation process on the object to reduce a gradient of a level of the solute within the at least a portion of the object.

In a further aspect, the present invention provides apparatus for changing a concentration of a solute within an object, the object being made of a metal or alloy, the apparatus comprising means for producing a vapour such that at least a portion of the object is exposed to the vapour, wherein the vapour comprises a chemical reactant, the chemical reactant having a different level of reactivity to the solute than the metal or alloy, and a flux material, the flux material being for facilitating the removal of a product of a reaction between the chemical reactant in the vapour and the alloy from a surface of the object.

In a further aspect, the present invention provides a method of producing an object with a second average solute level, wherein the second average solute level is equal to a desired solute level or within a desired range, the object being made of a metal or alloy, the method comprising from a raw material having a first average solute level, forming an object to produce a formed object, the formed object having an initial porosity, and the formed object having open porosity, performing a solute level changing process on the formed object to add or remove solute from the formed object, thereby changing the average solute level of the object to the second average solute level, and performing a consolidation process on the object having the second average solute level to reduce the porosity of the object to be equal to a desired level or within a desired range.

The solute level changing process may be performed to reduce an average solute level within the formed object.

The consolidation process may comprise sintering the object having the second average solute level.

The formed object may have a solid density within the range 60% to 92%.

The formed object may have a solid density within the range 70% to 80%.

After the step of performing the consolidation process, the object may have a solid density within the range 92% to 100%.

The method may further comprise performing a homogenisation process on the object having the second average solute level to diffuse the solute within the object having the second average solute level.

The method further comprises performing a homogenisation process on the object having the second average solute level to diffuse the solute within the object having the second average solute level.

The step of performing the homogenisation process may be performed such that the solute level throughout the object having the second average solute level is substantially uniform.

The solute level changing process may comprise performing a solute level decreasing process, and performing a solute level increasing process, wherein the solute level decreasing process and the solute level increasing process are performed on the formed object prior to the consolidation process being performed, the solute level decreasing process is performed to remove the solute from the surface of the formed object, thereby reducing the average solute level of the formed object, the solute increasing process is performed to add the solute to the surface of the formed object, thereby increasing the average solute level of the formed object, and an amount that the average solute level is reduced by during the solute level decreasing process is different to an amount that the average solute level is increased by during the solute increasing process.

The alloy may comprise one or more of the following metals: titanium, tantalum, vanadium, zirconium, molybdenum, niobium.

The alloy may be a titanium alloy.

The alloy may be Ti—6Al—4V.

The solute may be one of the following: oxygen, carbon, hydrogen or nitrogen.

The solute may be oxygen.

The second average solute level may be between 1000 and 2300 parts per million by weight.

The second average solute level may be between 1300 and 1800 parts per million by weight.

The second average solute level may be between 1700 and 1800 parts per million by weight.

The second average solute level may be less than 2000 parts per million by weight.

The second average solute level may be less than 1300 parts per million by weight.

The first average solute level may be between 2300 and 10000 parts per million by weight.

The first average solute level may be between 5000 and 10000 parts per million by weight.

The first average solute level may be greater than 10000 parts per million by weight.

The step of forming an object may comprise forming an object using a powder form of the alloy and a metal injection moulding process.

The step of performing a solute level changing process may comprise exposing at least a portion of the formed object to a fluid, wherein the fluid comprises a chemical reactant, and the chemical reactant has a different level of reactivity to the solute than the metal or alloy.

The chemical reactant may be calcium.

The fluid may further comprise a flux material, the flux material being for facilitating the removal of a product of a reaction between the chemical reactant and the metal or alloy from a surface of the formed object.

The flux material may be calcium chloride.

The fluid may be a vapour.

The step of performing a solute level changing process may comprise heating the formed object to between 1000°C and 1200°C.
In a further aspect, the present invention provides apparatus for producing an object with a second average solute level, wherein the second average solute level is equal to a desired solute level or within a desired range, the object being made of a metal or alloy, the apparatus comprising a production means for, from a raw material having a first average solute level, forming an object to produce a formed object, the formed object having an initial porosity, and the formed object having open porosity, means for performing a solute level changing process on the formed object to add or remove solute from a surface of the formed object, thereby changing the average solute level of the object to the second average solute level, and means for performing a consolidation process on the object having the second average solute level to reduce the porosity of the object to be equal to a desired level or within a desired range.

In a further aspect the present invention provides a method, the method comprising performing method steps in accordance with the first aspect of the invention, wherein the solute level changing process is performed in accordance with any of the other aspects.

In a further aspect the present invention provides a method, the method comprising performing method steps in accordance with the first aspect of the invention or the previous aspect of the invention, the method further comprising, from a raw material having a first average solute level, forming the object to produce a formed object, the formed object having an initial porosity; and the formed object having open porosity, and performing a solute level changing process and a consolidation process in accordance with any of the other aspects.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a process flow chart showing certain steps of an example of a process for producing a titanium alloy machine component in which an embodiment of an oxygen reduction process is performed;

FIG. 2 is a process flow chart showing certain steps of a process of forming the component, as performed in FIG. 1;

FIG. 3 is a schematic illustration (not to scale) of a component formed using the process shown in FIG. 2;

FIG. 4 is a schematic illustration (not to scale) of a first cross section and a second cross section of the component after it has been formed using the process shown in FIG. 2;

FIG. 5 is a process flow chart showing certain steps of an embodiment of an oxygen reduction process performed on the component during the process shown in FIG. 1;

FIG. 6 is a process flow chart showing certain steps of a de-oxygenation process performed on the component during the process shown in FIG. 5;

FIG. 7 is a schematic illustration (not to scale) of a de-oxygenation chamber used in the process of FIG. 6;

FIG. 8 is a schematic illustration (not to scale) of the first cross section and the second cross section after the de-oxygenation process has been performed on the component;

FIG. 9 is a process flow chart showing certain steps of a re-oxygenation process performed on the component during the process shown in FIG. 5;

FIG. 10 is a schematic illustration (not to scale) of the first cross section and the second cross section after the re-oxygenation process has been performed on the component;

FIG. 11 is a process flow chart showing certain steps of an oxygen homogenisation process performed on the component during the process shown in FIG. 5;

FIG. 12 is a schematic illustration (not to scale) of the first cross section and the second cross section after the oxygen homogenisation process has been performed on the component;

FIG. 13 is a process flow chart showing certain steps of a process for producing a titanium alloy component in which an oxygen reduction process is performed prior to the component being fully consolidated; and

FIG. 14 is a process flow chart showing certain steps of a process of partially consolidating the component, as during the process of FIG. 13.

DETAILED DESCRIPTION

FIG. 1 is a process flow chart showing certain steps of an example of a process for producing a titanium alloy machine component (hereinafter referred to as “the component”) in which an embodiment of an oxygen reduction process is performed.

At step s2, the component is formed.

An example process by which the component is formed is described in more detail later below with reference to FIG. 2.

At step s4, an oxygen reduction process is performed on the component.

In this example, the oxygen reduction process is performed to reduce a level of oxygen (i.e. an oxygen concentration) within the component.

An embodiment of an oxygen reduction process is described in more detail later below with reference to FIG. 5.

Thus, a process for producing a titanium alloy component is provided.

FIG. 2 is a process flow chart showing certain steps of a process of forming the component, as performed at step s2 of the process for producing the component (described above with reference to FIG. 1).

At step s6, a metal injection moulding process is performed to produce a so-called “green part”.

In this example, a conventional metal injection moulding process is performed. A relatively finely-powdered alloy is mixed with binder material to produce a so-called “feedstock”. This feedstock is shaped using an injection mould process to produce the green part.

In this example, the alloy is titanium with 6% aluminium and 4% vanadium (also known as Ti—6Al—4V, or 6-4-5%, ASTM B348 Grade 5). Also, in this example, the alloy contains an amount of oxygen in solid solution. In this example, the alloy comprises greater than 0.18% oxygen, i.e. greater than 1800 parts per million (ppm) by weight.

At step s7, after the green part is cooled and demoulded, a portion of the binder material is removed from the green part to produce a so-called “brown part”.

In this example, a conventional process for removing binder material from the green part is used, e.g., by using a solvent, a thermal evaporation, and/or a catalytic process, etc.

In this example, the brown part produced by the metal injection moulding and binder removal processes has a solid density of approximately 60%. In other words, the brown part is relatively porous.
Also, in this example the brown part has substantially uniform porosity throughout the part. The surface and internal structure of the brown part have substantially equal porosity.

At step s8, a sintering process is performed on the brown part to form the component. In this example, a conventional sintering process is used.

In this example, the brown part is sintered at a temperature in the range 1900°C to 1300°C. This sintering process tends to agglomerate the metal particles in the brown part, thereby increasing the solid density of the part.

In this example, the component formed by sintering the brown part has a solid density within the range 92% to 100%.

Also, in this example, at this stage (i.e. after the sintering process) the component has an oxygen concentration of greater than 1800 ppm. Furthermore, this oxygen concentration is substantially uniform throughout the component.

Thus, a process by which the component is formed is provided.

FIG. 3 is a schematic illustration (not to scale) of the component 2 formed using the forming process described in more detail above with reference to FIG. 2.

In this example, the component 2 is a single, solid piece of titanium alloy.

The terminology “solid” is used herein to refer to a material that has a density by volume (i.e., solid density) of between 92% and 100%.

For convenience and ease of understanding, the shape of the component 2 can be described with reference to two cylinders (hereinafter referred to as the “first cylinder 4” and the “second cylinder 6”) that are configured as described in more detail later below. However, it should be understood that in this embodiment the component 2 is a single solid piece.

In this example, the first cylinder 4 has a diameter of approximately 20 mm and a length of approximately 100 mm.

In this example, the second cylinder 6 has a diameter of approximately 9 mm and a length of approximately 100 mm.

In this example, the first cylinder 4 and the second cylinder 6 are positioned such that one end of the first cylinder 4 is in contact with one end of the second cylinder 6. Furthermore, in this example the cylinders 4, 6 are positioned such that they are coaxial, i.e., the cylinders 4, 6 share a common axis. Thus, the length of the component 2 is 200 mm.

A cross-section of the first cylinder 4, hereinafter referred to as the “first cross section” is indicated in FIG. 3 by a dotted line and the reference numeral 8.

Also, a cross-section of the second cylinder 6, hereinafter referred to as the “second cross section” is indicated in FIG. 3 by a dotted line and the reference numeral 10.

FIG. 4 is a schematic illustration (not to scale) of the first cross section 8 and the second cross section 10 after the forming of the component 2 (i.e. after performing step s2 of FIG. 1).

In FIG. 4, regions in the cross-sections 8, 10 that have a relatively high concentration of oxygen (i.e., a relatively high oxygen level) are shaded in black (i.e., shown as solid filled regions). The terminology “relatively high concentration of oxygen” is used herein to refer to locations having an oxygen concentration of greater than 1800 ppm. In this example, after the forming of the component 2 (i.e., after step s2), the component 2 has an oxygen concentration of greater than 1800 ppm. Furthermore, this oxygen concentration is substantially uniform throughout the component. In other words, the component 2 comprises a substantially uniform and relatively high level of oxygen. Thus, in FIG. 4 the first and second cross sections 8, 10 are shaded black.

For completeness, although no such reasons are indicated in FIG. 4, regions in the cross-sections 8, 10 that have a medium concentration of oxygen (i.e., a medium oxygen level) would be indicated by cross-hatching. The terminology “medium oxygen level” is used herein to refer to regions having an oxygen concentration within the range 1300 ppm to 1800 ppm. Also, regions in the cross-sections 8, 10 that have a relatively low concentration of oxygen (i.e., a relatively low oxygen level) would not be shaded (i.e., shown as blank regions). The terminology “relatively low concentration of oxygen” is used herein to refer to regions having an oxygen concentration of lower than 1300 ppm.

Referring now back to FIG. 1, after the forming of the component 2 (i.e., step s2), the oxygen reduction process (step s4) is performed on the component 2.

FIG. 5 is a process flow chart showing certain steps of an embodiment of the oxygen reduction process performed on the component formed at step s2.

At step s10, a de-oxidation process is performed on the component 2.

This de-oxidation process is performed to remove oxygen from the component 2.

The de-oxidation process used in this embodiment is described in more detail later below with reference to FIGS. 6 to 8.

At step s12, a re-oxidation process is performed on the component 2.

This re-oxidation process is performed to add oxygen back into the component 2.

The re-oxidation process used in this embodiment is described in more detail later below with reference to FIGS. 9 and 10.

At step s14, a process of homogenising the oxygen concentration level throughout the component 2 is performed. This is a process by which the uniformity of an oxygen concentration level throughout the component 2 is increased. A desired degree of uniformity for the oxygen level within the component 2 may depend on, for example, the service requirements for the component 2. This process is hereinafter referred to as the “oxygen homogenisation process”.

The oxygen homogenisation process used in this embodiment is described in more detail later below with reference to FIGS. 11 and 12.

Thus, an oxygen reduction process is provided.

Referring back to step s10 of the oxygen reduction process, FIG. 6 is a process flow chart showing certain steps of the de-oxidation process performed on the component 2 at step s10.

At step s16, the component 2 is placed in a chamber in which de-oxidation of the component 2 is to take place. For convenience, this chamber is hereinafter referred to as the “de-oxidation chamber”.

The de-oxidation chamber, and the positioning of the component 2 in the de-oxidation chamber, is described in more detail later below with reference to FIG. 7.
At step s17, a substance that will be used to remove oxygen from the component 2 is placed into the de-oxidation chamber. The substance and its placement with the de-oxidation chamber will be described in more detail later below with reference to FIG. 7.

At step s18, the de-oxidation chamber is sealed, as described in more detail later below such that the de-oxidation chamber wholly contains both the component 2 and the substance.

What will now be described is the de-oxidation chamber, and the configuration of the component 2 and the substance within the de-oxidation chamber. The remaining steps (steps s19 to s22) of the process of FIG. 6 will be described in more detail after this information.

FIG. 7 is a schematic illustration (not to scale) of the de-oxidation chamber 12.

In this embodiment, the de-oxidation chamber 12 comprises a container 13, and a stand 14. The stand 14 is positioned wholly within the container 13.

Also described above, the component 2 is placed wholly within the de-oxidation chamber 12. In this embodiment, the substance 16 inside the container 13 is sealed with the component 2 and the substance 16 inside. Upon heating the de-oxidation chamber 12 (as described in more detail later below with reference to step s19 of the FIG. 6), the substance 16 melts and a portion of the substance 16 is vaporised. The vaporised substance, hereinafter referred to as "the vapour" is depicted in FIG. 7 as wavy lines and indicated by the reference numeral 18. The vapour 18 contacts the component 2.

Referring now back to FIG. 6, at step s19, the de-oxidation chamber 12 is heated to melt and then vaporise the substance 16 (thereby producing the vapour 18).

In this embodiment, the de-oxidation chamber 12 is heated to between 772°C. (the melting temperature of the flux) and approximately 1660°C. (the melting temperature of the titanium alloy, i.e. the component 2). Preferably, the de-oxidation chamber 12 is heated to between 1000 and 1200°C. This temperature advantageously tends to provide reasonably rapid de-oxidation, while at the same time reactions with the de-oxidation chamber fixtures (e.g. the stand 14), and other problems associated with very high temperature, are avoided or alleviated.

The heating of the de-oxidation chamber 12 such that the substance 16 vapourises causes the vapour 18 to contact the component 2. As the calcium in the substance 16 (and therefore the vapour 18) has a greater affinity for oxygen than the titanium of the component 2, oxygen atoms tend to be removed from the surface of the component 2 by the vapour 18. Thus, an oxygen concentration at the surface of the component 2 tends to be lower than an oxygen concentration towards the base of the component 2. This concentration gradient tends to produce a net flow of oxygen from the interior of the component 2 to the surface of the component 2 (by solid state diffusion).

As the calcium in the vapour 18 reacts with the oxygen in the component 2, calcium oxide is formed. This calcium oxide tends to be deposited on the surface of the component 2. The flux in the vapour 18 (i.e. the vaporised calcium chloride) advantageously tends to dissolve, direct away or in some other way remove calcium oxide from the surface of the component 2. For example, during de-oxidation the component may become coated with a liquid film of calcium chloride/calcium solution which tends to perform a similar function to a liquid flux in embodiments in which the component is immersed in liquid flux and reactant (e.g. that of dissolving the calcium oxide reaction product and supplying fresh calcium to the surface). Calcium can contact the component in vapour form and dissolve into the flux. However, as calcium oxide is relatively not volatile, it tends to remain in the liquid film. This liquid film may run off the component and returns to the liquid pool below. It has been found in experiments that the de-oxidation rate is higher in a calcium chloride/calcium vapour than in calcium vapour alone.

In other words, the flux in the vapour 18 (i.e. the vaporised calcium chloride) advantageously tending to dissolve, direct away or in some other way remove calcium oxide from the surface of the component 2 tends to facilitate access to the component 2 by the vapour 18, and tends to maintain the concentrations of reactants and reaction products that provide that the reaction to continues at a desired rate. Thus, use of the flux material advantageously tends to facilitate the removal of oxygen from the component 2.

In this embodiment, the de-oxidation chamber 12 is heated for a first pre-determined time-period. In other words, the temperature of the de-oxidation chamber 12 is maintained at, e.g. between 1000 and 1200°C, for a first pre-determined
time-period. In this embodiment, the first pre-determined time-period is between 0.5 to 100 hours. For example, an average oxygen level in a 9.3 mm diameter Ti-6% bar may be reduced from 2000 ppm to 600 ppm with a 20 mol % solution of calcium in calcium chloride at 100°C for 72 hrs.

[0183] In this embodiment, the first pre-determined time-period is predetermined to achieve a desired oxygen level within the component 2. Advantageously, it tends to be possible to determine/change this first pre-determined time period and/or the amount of substance 16 used and/or concentration of chemical de-oxidant in the substance 16 to achieve a desired oxygen level within the component 2. These may be determined depending on certain parameters, for example an initial oxygen content within the component 2, a thickness of the component 2, and the temperature that the de-oxidation chamber 12 is heated to.

[0184] In this embodiment, the first pre-determined time period and/or amount of chemical de-oxidant is such that, after treatment of the component 2 for this time period, the component 2 has an average oxygen level of less than 1300 ppm of oxygen by weight (i.e. a low oxygen level, in the terminology of this description).

[0185] In this embodiment, during step s19 (i.e. during the heating of the de-oxidation chamber 12), the de-oxidation chamber 12 is either evacuated or back filled with a partial pressure of argon. A partial pressure of argon tends to avoid undesirable internal pressurisation of the container at the processing temperature or, in the case of evacuation, external pressurisation of the chamber by atmospheric pressure. In this embodiment, the partial pressures of the reactants and the argon are such that, at the processing temperature, the total internal pressure of the container 13 is approximately one atmosphere.

[0186] In this embodiment, the container 13 and the stand 14 are made from a material that does not alloy with or react adversely with titanium or any of the de-oxidation chemicals at the processing temperature. In this embodiment, titanium is used for the container 13 and the stand 14. However, in other embodiments refractory metals such as molybdenum and/or high temperature steels may be used.

[0187] At step s20, after the first predetermined time period has expired, the de-oxidation chamber 12 is left to cool.

[0188] At step s21, the container 13 of the de-oxidation chamber 12 of the component 2 is removed.

[0189] At step s22, the component 2 is cleaned. In this embodiment, water is used to remove the flux from the component 2, and a dilute acid (e.g. dilute hydrochloric acid) is used to remove calcium oxide residues.

[0190] Thus, a de-oxidation process is provided.

[0191] FIG. 8 is a schematic illustration (not to scale) of the first cross section 8 and the second cross section 10 after the de-oxidation process has been performed as described above with reference to FIG. 6 (i.e. after performing steps s10 of FIG. 5).

[0192] In FIG. 8, as in FIG. 4, regions in the cross-sections 8, 10 that have a relatively high concentration of oxygen (i.e. an oxygen concentration of greater than 1800 ppm) are shaded in black, regions that have a medium concentration of oxygen (i.e. within the range 1300 ppm to 1800 ppm) are indicated by cross-hatching, and regions that have a relatively low concentration of oxygen (i.e. an oxygen concentration of lower than 1300 ppm) are not shaded.

[0193] In this embodiment, after the de-oxidation process the component 2 has, at its first cross section 8, a relatively small central internal portion (hereinafter referred to as the “inner core” and indicated in FIG. 8 by the reference numeral 20) that has an oxygen concentration of greater than 1800 ppm.

[0194] Also, the component 2 has, at its first cross section 8, a region surrounding the inner core 20 (hereinafter referred to as the “outer core” and indicated in FIG. 8 by the reference numeral 22) that has an oxygen concentration of between 1300 ppm and 1800 ppm.

[0195] Also, the component 2 has, at its first cross section 8, a region next to the component surface (hereinafter referred to as the “edge” and indicated in FIG. 8 by the reference numeral 23) that has an oxygen concentration of between 1300 ppm and 1800 ppm.

[0196] In FIG. 8, for reasons of clarity and ease of understanding the three regions of the first cross-section 8 (i.e. the inner core 20, the outer core 22, and the edge 23) are shown as having distinct and discrete boundaries. However, in reality, in this embodiment after the de-oxidation process, the oxygen level across the first cross-section 8 of the component 2 will vary continuously—from relatively low at the edge/surface of the component 2, to relatively high at the component centre.

[0197] In this embodiment, after the de-oxidation process the component 2 has, through its second cross section 10, an oxygen concentration of less than 1300 ppm. Furthermore, in this embodiment this oxygen concentration is substantially uniform throughout the parts of the component 2 corresponding to the second cross section 10. Thus, in FIG. 8 the second cross section 10 is not shaded.

[0198] In this embodiment, after the de-oxidation process the component 2 has an average oxygen level of less than 1300 ppm of oxygen by weight (i.e. a low oxygen level). In other words, after the de-oxidation process if all the oxygen within the component was spread substantially uniformly throughout the component 2, the oxygen level of the component 2 would be low (i.e. less than 1300 ppm).

[0199] Thus, in this embodiment, because during the de-oxidation process oxygen is removed substantially uniformly from the surface of the component 2, regions of the component 2 that have different thickness (i.e. the first cylinder 4 and the second cylinder 6) tend to have different oxygen concentrations after the de-oxidation process. In particular, relatively thin sections (the second cylinder 6) tend to have lower oxygen levels than relatively thick sections (the first cylinder 4).

[0200] Thus, in this embodiment, the de-oxidation process is performed on the component 2 to reduce the average oxygen level within the component 2. Also, the de-oxidation component is performed to increase the gradient of the oxygen level across the component 2. This is in contrast to conventional de-oxidation processes (such as those performed to reduce the alpha case within a component) which tend to be performed to decrease a gradient of the oxygen level within an object being treated.

[0201] In this embodiment, an optional additional step is provided in which a re-oxidation of the component 2 is performed (i.e. step s12 of FIG. 5 is performed). This optional re-oxidation process tends to increase the oxygen level of a thinner portion of a component proportionally more than the oxygen level of a thicker portion is increased. This is performed in this embodiment to increase the average oxygen level of the thinner section of the component 2 to be substantially equal to that of the thicker section.
Referring now back to FIG. 5, the re-oxidation process performed in this embodiment at step s12 will now be described.

FIG. 9 is a process flow chart showing certain steps of the re-oxidation process performed on the component 2 at step s12.

At step s24, the component 2 is placed in a chamber in which re-oxidation of the component 2 is to take place. For convenience, this chamber is hereinafter referred to as the "re-oxidation chamber."

In this embodiment, the re-oxidation chamber is a chamber that is substantially the same as the de-oxidation chamber 12 described above with reference to FIG. 7 (i.e. the re-oxidation chamber comprises a metal container and a stand configured in the same way as those components of the de-oxidation chamber 12).

In this embodiment, the component 2 is placed upon the stand of the re-oxidation chamber such that the component 2 is wholly within the walls of the container of the re-oxidation chamber.

At step s25, a further substance that will be used to introduce oxygen into the component 2 is placed into the re-oxidation chamber.

In this embodiment, the further substance is placed into the bottom of the container of the re-oxidation chamber such that the component 2 is submerged beneath the further substance.

In this embodiment, the further substance comprises an oxidising chemical and a flux. In this embodiment, the oxidising chemical and the flux are placed in the container in solid form, e.g. in the form of powder or pellets.

In this embodiment, the oxidising chemical is calcium oxide.

In this embodiment, the flux used in the re-oxidation process is the same as the flux used in the de-oxidation process, i.e. calcium chloride. The flux advantageously aids the transport of the oxidising chemical to the surface of the component 2.

In this embodiment, the proportion of the oxidising chemical in the further substance is in the range 1 to 20 mol% and preferably within the range 15 to 20 mol%. This tends to provide that the re-oxidation process is particularly effective.

In this embodiment, the re-oxidation of the component uses the same chemical reaction as the de-oxidation process. In this embodiment, a preferably 15 to 20 mol% solution of calcium oxide in calcium chloride is used. By increasing the concentration of calcium oxide and reducing the concentration of calcium, the equilibrium point of the reaction is changed and oxidation takes place rather than de-oxidation (in accordance with Le Chatelier’s Principle).

In other embodiments, the oxidation of the component 2 could be carried out in a different way, for example using gaseous oxygen. However, this tends to be difficult to control. In other embodiments, a different oxidising chemical is used to re-oxidise the component 2, e.g. a chemical element that has less of an affinity for oxygen than titanium (i.e. the component 2). In other embodiments, water vapour is used to re-oxidise the component 2. In such cases the titanium tends to react with the water vapour and both the oxygen and the hydrogen diffuse into the metal. The hydrogen may be removed later, e.g. by vacuum degassing.

At step s26, the re-oxidation chamber is sealed such that the container wholly contains both the component 2 and the further substance.

At step s19, the re-oxidation chamber is heated to melt the further substance, thereby producing liquidised further substance.

In this embodiment, the re-oxidation chamber is heated to between 772°C (the melting temperature of the flux) and approximately 1600°C (the melting temperature of the titanium alloy, i.e. the component 2). Preferably, the re-oxidation chamber is heated to between 1000 and 1200°C. This temperature advantageously tends to provide that the oxidation proceeds at a suitable rate and reactions with the re-oxidisation chamber fixtures (e.g. the stand of the re-oxidation chamber), and other problems associated with very high temperature, are avoided or alleviated.

The heating of the re-oxidation chamber causes the further substance to melt and contact the component 2. Oxygen atoms tend to be removed from the further substance (thereby forming calcium), and introduced into the surface of the component 2. Thus, an oxygen concentration at the surface of the component 2 tends to be increased by the re-oxidation process. A net flow of oxygen from the surface of the component 2 towards the interior of the component 2 tends to be produced.

As the calcium oxide in the further substance reacts with the component 2, calcium is formed. This calcium tends to be deposited on the surface of the component 2. The flux in the further substance (i.e. the vapourised calcium chloride) advantageously tends to dissolve, or remove, calcium from the surface of the component 2. This tends to facilitate access to the component 2 by the calcium oxide and tends to maintain the concentrations of reactants and reaction products that provide that the reaction continues at a desired rate. Thus, use of the flux material advantageously tends to facilitate the introduction of oxygen to the component 2. In this embodiment, the re-oxidation chamber is heated for a second pre-determined time-period. In other words, the temperature of the re-oxidation chamber is maintained at, e.g. between 1000 and 1200°C, for a second pre-determined time-period. In this embodiment, the second pre-determined time-period is between 0.5 to 100 hours. For example, the oxygen level of a 9.3 mm diameter Ti-6/4 bar may be increased from approximately 2000 ppm to 3500 ppm by immersion in a solution of 20 mol% calcium oxide in calcium chloride for 72 hrs at 1000°C. In this embodiment, the second pre-determined time-period is pre-determined in order to achieve a further desired oxygen level within the component 2. Advantageously, it tends to be possible to determine/change this second pre-determined time period and/or the amount of further substance used and/or concentration of oxidising chemical in the further substance to achieve the further desired oxygen level within the component 2. These may be determined depending on certain parameters, for example an initial oxygen content within the component 2 (i.e. oxygen content in the component 2 after the de-oxidation process), a thickness of the component 2 and the temperature that the re-oxidation chamber is heated to.

In this embodiment, the second pre-determined time period and/or amount of oxidising chemical is such that, after treatment of the component 2 for this time period, the component 2 has an average oxygen level between 1300 ppm and 1800 ppm of oxygen by weight (i.e. a medium oxygen level, in the terminology of this description).

In this embodiment, during step s27 (i.e. during the heating of the re-oxidation chamber), the re-oxidation chamber is either evacuated or back filled with a partial pressure of
argon. A partial pressure of argon tends to avoid undesirable internal pressurisation of the container of the re-oxidation chamber at the processing temperature or, in the case of evacuation, external pressurisation of the chamber by atmospheric pressure. In this embodiment, the partial pressures of the reactants and the argon are such that, at the processing temperature, the total internal pressure of the container of the re-oxidation chamber is approximately one atmosphere.

At step s28, after the second predetermined time period has expired, the re-oxidation chamber is left to cool. At step s29, the container of the re-oxidation chamber is opened and the component 2 removed.

At step s22, the component 2 is cleaned. In this embodiment, water is used to remove the flux from the component 2, and a dilute acid (e.g. dilute hydrochloric acid) is used to remove calcium and calcium oxide residues.

Thus, a re-oxidation process is provided.

Fig. 10 is a schematic illustration (not to scale) of the first cross section 8 and the second cross section 10 after the re-oxidation process has been performed as described above with reference to Fig. 9 (i.e. after performing step s12 of Fig. 5).

In Fig. 10, as in Figs. 4 and 8, regions in the cross-sections 8, 10 that have a relatively high concentration of oxygen (i.e. an oxygen concentration of greater than 1800 ppm) are shaded in black, regions that have a medium concentration of oxygen (i.e. within 1300 ppm to 1800 ppm) are indicated by cross-hatching, and regions that have a relatively low concentration of oxygen (i.e. an oxygen concentration of lower than 1300 ppm) are not shaded.

This embodiment, after the re-oxidation process the inner core 20 of the first cross section 8 has an oxygen concentration of greater than 1800 ppm. Also, the outer core 22 of the first cross section 8 has an oxygen concentration between 1300 ppm and 1800 ppm.

Also, in this embodiment, oxygen has been introduced to the surface of the component 2 at the first cross section 8. Thus, after the re-oxidation process the region referred to as the edge 23 in Fig. 8 has had oxygen introduced into it (from the surface of the component).

Thus in this embodiment, after re-oxidation the edge 23 comprises a first region (indicated in Fig. 10 by the reference numeral 24) next to the surface of the component 2 that has an oxygen concentration of between 1300 ppm and 1800 ppm, and a second region (indicated in Fig. 10 by the reference numeral 25) between the first region 24 and the outer core 22 that has an oxygen concentration lower than 1300 ppm.

In this embodiment, oxygen has been introduced to the surface of the component 2 at the second cross section 10.

Thus, after the re-oxidation process, the second cross section 10 comprises an “inner region” (indicated in Fig. 10 by the reference numeral 26) that has an oxygen concentration lower than 1300 ppm, and an “outer region” (indicated in Fig. 10 by the reference numeral 27) that has an oxygen concentration of between 1300 ppm and 1800 ppm. The outer region 27 is next to the surface of the component 2 at the second cross-section 10, and the inner region 26 is located towards the centre of the second cross section 10, away from the surface of the component 2 (i.e. inside the outer region 27).

In this embodiment, after the re-oxidation process the component 2 has an average oxygen level of between 1300 ppm and 1800 ppm of oxygen by weight (i.e. a medium oxygen level). In other words, after the re-oxidation process if all the oxygen within the component 2 was spread substantially uniformly throughout the component 2, the oxygen level of the component 2 would be medium (i.e. less between 1300 ppm and 1800 ppm).

In Fig. 10, as in Fig. 8, for reasons of clarity and ease of understanding the different regions of the cross-sections 8, 10 are shown as having distinct and discrete boundaries. However, in reality, in this embodiment after the re-oxidation process, the oxygen level across each of the cross-sections 8, 10 will vary continuously.

Thus, in this embodiment, because during the re-oxidation process oxygen is introduced substantially uniformly to the surface of the component 2, regions of the component 2 that have different thickness (i.e. the first cylinder 4 and the second cylinder 6) tend to have oxygen introduced into them at different average rates per unit volume. Thus, in this embodiment after the re-oxidation process the first and second cylinder 4, 6 have substantially equal average oxygen concentrations (i.e. medium oxygen concentrations). However, at this stage, oxygen within the cylinders 4, 6 (i.e. within the component 2) is not spread uniformly throughout the component 2.

Thus, in this embodiment, the re-oxidation process is performed on the component 2 to increase the average oxygen level within the component 2. Also, the re-oxidation component is performed to increase the gradient of the oxygen level across (at least a part of) the component 2.

Referring back to Fig. 5, after the re-oxidation process (step s12) is performed, the oxygen homogenisation process of step s14 is performed. This process is performed to make the oxygen concentration level substantially uniform throughout the whole component 2.

The oxygen homogenisation process performed in this embodiment at step s14 will now be described.

Fig. 11 is a process flow chart showing certain steps of the oxygen homogenisation process performed on the component 2 at step s14.

At step s32, the component 2 is placed in a chamber in which homogenisation of the oxygen level throughout the component 2 is to take place. For convenience, this chamber is hereinafter referred to as the “homogenisation chamber”.

In this embodiment, the homogenisation chamber is a conventional vacuum furnace. Preferably, the homogenisation chamber comprises refractory metal heater elements and is capable of maintaining a vacuum of better than 10^-5 mbar.

At step s34, the homogenisation chamber is filled with an inert gas (e.g. argon) such that an inert gas atmosphere is maintained within the chamber during the homogenisation process. In other embodiments, a vacuum may be formed in the homogenisation chamber instead of the chamber being filled with an inert gas.

At step s36, the component 2 is heated (within the homogenisation chamber) to a relatively high temperature. Preferably, the component 2 is heated to between 1000 and 1300°C. This temperature range advantageously tends to provide that problems associated with very high temperature, are avoided or alleviated. Furthermore, this temperature range tends to be high enough to provide relatively fast processing time, and also be low enough to avoid damaging of the component 2, e.g. due to distortion under its own weight, incipient melting, excessive grain growth or other deleterious metallurgical reactions.
In general, the oxygen homogenisation process can be carried out at a higher temperature than the de-oxidation (or re-oxidation) process because no reacting chemicals are used. Suitable high temperature furnaces exist.

In this embodiment, the component 2 is heated for a third pre-determined time-period. In this embodiment, the third pre-determined time-period is between 0.5 to 100 hours. Preferably, the third pre-determined time-period is less than 12 hours. This tends to ensure that the homogenisation process is relatively cost-effective.

Advantageously, it tends to be possible to determine/change this third pre-determined time-period depending on certain parameters, for example an initial oxygen content within the component 2 (i.e. oxygen content in the component 2 after the re-oxidation process), the specified oxygen range for the different parts of the component, a thickness of the component 2, and the homogenisation temperature.

In this embodiment, the third pre-determined time period is sufficiently long to provide that oxygen homogenisation within the component 2 occurs, i.e. oxygen gradients within the component 2 disappear or are reduced. Thus, a component 2 having substantially uniform properties, or properties that vary in the different regions by acceptable amounts, tends to be advantageously produced. Furthermore, for a porous powder product, such as that produced by MIM, the heating process performed during oxygen homogenisation tends to sinter the component further. This can be allowed for in the MIM process by using a shorter initial sinter time.

At step s38, after the third predetermined time period has expired, the homogenisation chamber is left to cool.

Thus, an oxygen homogenisation process is provided.

FIG. 12 is a schematic illustration (not to scale) of the first cross section 8 and the second cross section 10 after the oxygen homogenisation process (i.e. after performing step s14 of FIG. 5).

In FIG. 12, as in FIGS. 4, 8 and 10, regions in the cross-sections 8, 10 that have a relatively high concentration of oxygen (i.e. greater than 1800 ppm) are shaded in black, regions that have a medium concentration of oxygen (i.e. within the range 1300 ppm to 1800 ppm) are indicated by cross-hatching, and regions that have a relatively low concentration of oxygen (i.e. lower than 1300 ppm) are not shaded.

In this embodiment, after the oxygen homogenisation process the component 2 has a medium oxygen concentration. Furthermore, this oxygen concentration is substantially uniform throughout the component. Thus, in FIG. 12 the first and second cross sections 8, 10 are cross-hatched.

Thus, a process for producing a titanium alloy component that has a substantially uniform oxygen level at a concentration of 1300 ppm to 1800 ppm of oxygen by weight is provided.

An advantage provided by the above described processes is that a component is produced using a powder metallurgy manufacturing technique. This tends to provide that a near-net-shape component is produced with very little wastage. Furthermore, it tends to be relatively easy to make relatively complex shapes that may be prohibitively expensive to machine.

A further advantage provided by the above described processes is that it tends to be possible to use relatively cheap alloy powders (that are typically not suitable for production of structural parts due to their high oxygen content) to produce structural components (e.g. components). Low quality and cost (i.e. high oxygen concentration) alloys may be used to form a component, which is then treated (as described above) to reduce and homogenise oxygen, so as to produce a relatively higher quality (i.e. lower oxygen concentration) component. Conventionally, higher quality (i.e. lower oxygen concentration) components are produced using relatively high quality alloys. These tend to be of relatively high cost. For example, high quality titanium alloy powder may cost in the region of £150-£250 per kilogram, whereas lower quality titanium alloy powder may cost £20-£100 per kilogram. The cost of an alloy powder, for example, tends to be a very significant proportion of the total manufacturing cost of a component produced using it. The above described processes tend to provide a significant cost saving compared to conventional methods.

The above described processes tend to provide that a desired oxygen content within a component can be achieved at a lower cost.

The above described processes advantageously address a problem of removing bulk oxygen from components e.g. titanium alloy components. Furthermore, the above described process can be performed at a commercial scale. As described above, the removal of bulk oxygen from components may be performed by first removing oxygen from the surface of the component (i.e. reducing the oxygen level of the portion of the component proximate to the surface of the component), and then homogenising the oxygen level throughout the component. This reduces the oxygen level throughout the bulk (i.e. the greater part of the body) of the component. In other words, the oxygen level is not just lowered at or proximate to the surface of the component. Instead, the oxygen level tends to be reduced throughout (the bulk of) the component. Elevated temperature chemical treatments are conventionally applied to some titanium powders to reduce oxygen content of those powders. Such elevated temperature chemical treatments are typically not appropriate for smaller powder sizes (e.g. that are preferable for metal injection moulding) because of the strong tendency of the powder particles to sinter. The above described processes are advantageously applicable to components made from powders of any size. This is because treatment is performed after the formation of the component (i.e. after the powders have been sintered).

A further advantage provided by the above described processes is that it tends to be possible to achieve a desired oxygen concentration in a component. This can be achieved using the above described de-oxidation and re-oxidation processes. At certain concentrations, oxygen strengthens and hardens titanium alloys. It tends to be an important alloying addition in many alloys. However, if an oxygen concentration within an alloy is too high, the oxygen tends to embrittle the alloy and reduce toughness and ductility.

Many conventional processing operations, including forging and heat treatment, introduce oxygen into an alloy. This introduced oxygen is usually restricted to a thin region near the outer surface of a component, called ‘alpha case’ in Ti—6Al—4V alloy. This thin layer may be machined off or removed chemically. However, machining tends to be expensive and chemical treatment, called ‘chemical milling’ tends to be both expensive and subject to increasingly restrictive environmental and safety legislation. The above described processes tend to provide a lower cost and more environmentally acceptable way of removing alpha case. A
further advantage provided by the above described processes is that any of the de-oxidation, re-oxidation, and oxygen homogenisation processes may be performed on a large number of components simultaneously. Thus, a cost of performing any or all of these operations (per component) may be significantly reduced.

[0260] At any stage in the above described process for producing a titanium alloy component it tends to be possible to estimate or determine an oxygen level in a component. Conventional techniques may be used to estimate or determine an average oxygen level in a component. For example, this can be performed by performing a process of “Oxygen Determination by Gas Fusion” (i.e. or other hardness testing process) e.g. using a TC400-Series Leono(TM) machine on a test component that is substantially identical to the component being produced. Also, by testing selected pieces of the component conventional techniques may be used to estimate or determine an oxygen level gradient through a component.

[0261] In the above embodiments the oxygen reduction process is implemented on solid material after step s2 has been completed and the component has a density in the range 92-100% (i.e. the component is “fully consolidated”).

[0262] What will now be described is a further embodiment in which the oxygen reduction process is performed prior to the component being fully consolidated.

[0263] FIG. 13 is a process flow chart showing certain steps of a process for producing a titanium alloy component in which an oxygen reduction process is performed prior to the component being fully consolidated.

[0264] At step s42, the component is formed and partially consolidated. The terminology “partially consolidated” is used herein to refer to the component having a solid density of less than 92% and preferably in the range 70-80%. In other embodiments, the terminology “fully consolidated” and/or “partially consolidated” may refer to different ranges of solid density.

[0265] The process by which the component is formed and partially consolidated will now be described in more detail with reference to FIG. 14. The remaining steps (steps s44 and s46) of the process of FIG. 13 will be described after the description of FIG. 14.

[0266] FIG. 14 is a process flow chart showing certain steps of a process of partially consolidating the component, as performed at step s42 of FIG. 13.

[0267] At step s48, a metal injection moulding process is performed to produce a so-called “green part”.

[0268] In this further embodiment, the metal injection moulding process is performed as described above with reference to step s6 of FIG. 2.

[0269] At step s50, after the green part is cooled and demoulded, a portion of the binder material is removed from the green part to produce a so-called “brown part”.

[0270] In this further embodiment, the process of producing the brown part from the green part is performed as described above with reference to step s7 of FIG. 2.

[0271] In this further embodiment, the brown part produced has a solid density of approximately 50-60%. In other words, the brown part is relatively porous. Furthermore, the brown part has substantially uniform porosity throughout the part and has what is known as ‘open porosity’, that is porosity that is both internally interconnected and is open to the surface.

[0272] At step s52, the brown part produced at step s50 is “partially sintered”.

[0273] In this further embodiment, a conventional sintering process is used. The brown part is sintered at a temperature in the range 1000°C to 1300°C. The brown part is sintered until it has a solid density of less than 92% and preferably within the range 70-80%. In other embodiments, the partially consolidated component has a solid density within a different range, e.g. 60-92%.

[0274] Thus, a process by which the component is formed and partially consolidated is provided.

[0275] Referring now back to FIG. 13, at step s44, an oxygen reduction process is performed on the partially consolidated (i.e. having a solid density less than 92% and preferably in the range 70-80%) component.

[0276] In this further embodiment, the oxygen reduction process is performed to reduce a level of oxygen (i.e. an oxygen concentration) within the component, as described above with reference to FIG. 5. This process tends to reduce the oxygen concentration in the partially consolidated component from a high level to a desired medium level.

[0277] An advantage provided by performing the oxygen reduction process on a partially consolidated component (i.e. a component that is still relatively porous) is that during the de-oxidation and/or re-oxidation process steps, the fluid (i.e. the vapourised substance or the liquidised further substance) used to perform de-oxidation/re-oxidation is able to penetrate deeper into the component than the case with a fully consolidated component (i.e. the fluid passes more easily between the grains of the partially consolidated component than it would with a fully consolidated component). Thus, de-oxidation/re-oxidation of a relatively porous component tends to be relatively efficient. This tends to reduce the time and cost of the de-oxidation and/or re-oxidation processes.

[0278] A further advantage of performing the oxygen reduction process on a partially consolidated component is that, oxygen concentration tends to be more uniform throughout the component. Thus, less oxygen homogenisation of the component may be used to achieve a desired level of oxygen uniformity within the component.

[0279] A further advantage, provided by performing the oxygen reduction process on a partially consolidated component is that it tends to be possible to perform the oxygen homogenisation process on the partially consolidated component at a high enough temperature to fully sinter/consolidate (i.e. provide that its solid density is within the range 92% to 100%) the component. Thus, the number of process steps to produce the component, and therefore the cost of producing a component, tend to be reduced.

[0280] At step s46, if desired, a sintering process is performed (in a vacuum or an inert atmosphere) to fully sinter/consolidate the component (i.e. provide that its solid density is within the range 92% to 100%). In other embodiments a different type of consolidation process may be performed to fully sinter/consolidate the component. For example, in other embodiments a hot isostatic pressing (HIP) process is performed.

[0281] Thus, a process for producing a titanium alloy component in which an oxygen reduction process is performed on a partially consolidated component is provided.

[0282] In the above embodiments, the component is formed (at step s2) as described above with reference to FIG. 2. In particular, the component is formed using a process comprising a metal injection moulding process. However, in other embodiments a component is formed using a different net-shape or near-net shape manufacturing process. The termi-
technology "near-net shape manufacturing process" is used herein to refer to processes in which the initial production of the item is (substantially) the same as, or very close (i.e. within allowed tolerances) to, the final (net) shape. This tends to reduce the need for surface finishing of the object. For example, in other embodiments a component/object/item is produced using one or more of the following near-net shape manufacturing processes: casting, permanent mould casting, powder metallurgy, linear friction welding, metal injection moulding, rapid prototyping, spray forming, and superplastic forming. Such processes may comprise using other powder metallurgy processes. Such processes may include, for example, hot isostatic pressing (HIP), cold isostatic pressing (CIP), and 3D powder melt methods using scanning laser or electron beams. Such process may be used to form a fully or partially consolidated metal or alloy component/object. Such processes may use feedstock produced by a conventional ingot route, or they may use solid feedstock materials, such as a billet, plate, or bar made from lower cost, higher oxygen alloy powder via a powder metallurgy route. The metal/alloy powders used to produce the object, may, for example, be blended elemental powders. For example, an object that is made of Ti—6Al—4V can be produced from a blended elemental powder made by blending powders of titanium, aluminium and vanadium. Blended elemental powders tend to alloy and homogenise during a sintering process. An object that is made of Ti—6Al—4V can also be produced from a blended elemental powder made by blending titanium powder with an Al—V master alloy powder.

[0283] In the above embodiments, the component is formed from an alloy comprising titanium with 6% aluminium and 4% vanadium (also known as Ti—6Al—4V, or 6-4-9, ASTM B348 Grade 5). Also, in the above embodiments the alloy contains an amount of oxygen in solid solution. However, in other embodiments, the component is formed from a different material. For example, in other embodiments, the component is formed from a pure (i.e. unalloyed) metal, or a different type of alloy to that used in the above embodiments. Preferably, the component is formed from a metal or alloy in which there is significant oxygen solid solubility and diffusivity at a convenient processing temperature and for which there exists a suitable de-oxidation metal (in vapour or liquid form, or in solution) where the negative free energy of formation of its oxide is greater than the negative free energy of formation of the oxide of the metal/alloy being de-oxidised. For example, the component may be formed from any of the following metals or an alloy of one or more following metals: titanium, tantalum, vanadium, zirconium, molybdenum, niobium.

[0284] In other embodiments, the object/component is made of the alloy Ti—3Al—2.5V. Objects made of this alloy may be required (e.g. when used for certain applications) to have a relatively low maximum oxygen level (e.g. less than or equal to 1200 ppm). Typically, a metal injection moulding (MIM) process may add about 1000 ppm of oxygen to the alloy. Thus, conventionally it tends not to be possible, or be very difficult, to produce an object, using a MIM process, made of Ti—3Al—2.5V, and with a relatively low maximum oxygen level. However, such objects may be made using the above described processes. For example, such objects may be produced by: (i) providing some Ti—3Al—2.5V powder; (ii) producing an object from the powder using a MIM process; (iii) performing a de-oxidation process (e.g. as described above with reference to FIG. 6); and (iv) (optionally) performing a homogenisation process (e.g. as described above with reference to FIG. 11). The composition range for Ti—3Al—2.5V tends to be quite large and so it tends to be possible to produce an object that conforms to the standard. The oxygen levels in the object advantageously tend to homogenise during sintering. The Ti—6Al—4V and CP material/powders and other suitable elemental or master alloy powders from which the Ti—3Al—2.5V blended powder might be formed tend to be readily available and relatively cheap, whereas Ti—3Al—2.5V tends to be more difficult to attain and more expensive.

[0285] In the above embodiments, the component is a component (e.g. a component suitable for use in or by a piece of machinery). However, in other embodiments, the component is a different type of entity, object, or item made from a metal or alloy, for example an ingot, billet, plate, sheet, wire or bar or some other intermediate product form.

[0286] In the above embodiments, the shape and size of the component is as described in more detail above with reference to FIG. 3. The shape of the component is that of two coaxial cylinders placed end to end. The first cylinder has a diameter of approximately 20 mm and a length of approximately 100 mm, and the second cylinder has a diameter of approximately 9 mm and a length of approximately 100 mm. However, in other embodiments the size and shape of the component is different, i.e. a component may be of any appropriate size and shape.

[0287] In the above embodiments, the step of forming the component (step s2) is used to form a single component. Also, in the above embodiments the oxygen reduction step (step s4) is performed on a single component at a time. However, in other embodiments, the step of forming a component is used to form a different number of substantially identical or different components simultaneously. Also, in other embodiments the oxygen reduction step (step s4) is performed on a different number of substantially identical or different components simultaneously. Implementing the forming process or oxygen reduction process for multiple components simultaneously advantageously tends to reduce the cost of the process per component.

[0288] In the above embodiments, the sintering of the component is performed at the above specified temperatures, and for the above specified time-periods. However, in other embodiments sintering of a component is performed at a different appropriate temperature and/or for a different appropriate time period.

[0289] In the above embodiments, an oxygen concentration of greater than 1800 ppm is described as a high level. Also, an oxygen concentration of between 1300 ppm and 1800 ppm is described as a medium (desired) level. Also, an oxygen concentration of less than 1300 ppm is described as a low level. However, in other embodiments the high, medium and/or low oxygen concentration levels are different. Also, in other embodiments, the de-oxidation process is used to decrease an average oxygen concentration in a component from any level
to any other level. Also, in other embodiments, the re-oxidation process is used to increase an average oxygen concentration in a component from any level to any other level.

[0290] For example, in other embodiments a desired (average) oxygen concentration is between 1000 ppm and 2300 ppm. In other embodiments, a desired oxygen concentration is between 1300 ppm and 1800 ppm. In other embodiments a desired oxygen concentration is between 1700 ppm and 1800 ppm. In other embodiments, for example when Ti—Al—Ge or Yb—Ge—Al—Cu—Zn (or other similar intermetallic or eutectic materials) are formed, a desired oxygen concentration is less than or equal to 1500 ppm, or less than or equal to 1200 ppm.

[0291] Also for example, in other embodiments, a high (average) solute concentration level, i.e., an initial solute concentration level of an untreated component, or an initial solute concentration level of a raw material from which a component is formed (e.g., by using MIM), is between 2300 ppm and 10000 ppm. In other embodiments, a high solute concentration level is between 5000 ppm and 10000 ppm (for example 8000 ppm). In other embodiments, a high solute concentration level is greater than 10000 ppm. Titanium alloy powder with high oxygen concentrations (e.g., 8000 ppm) tend to be advantageously cheap and objects produced from such powders can be deoxidised using the above described processes to form objects having much lower oxygen concentrations.

[0292] In other embodiments, a desired solute concentration level is a proportion of the (high) initial solute concentration level. For example, the desired solute concentration level may be a half, or a quarter, or a fifth of the (high) initial solute concentration level.

[0293] In the above embodiments, the de-oxidation chamber, the re-oxidation chamber, and the homogenisation chamber are each configured as described above (the de-oxidation chamber is described above with reference to FIG. 7). However, in other embodiments one or more of these chambers are configured differently (e.g., made from different appropriate materials, or have different or additional fixtures) so as to still provide the above described functionality.

[0294] In the above embodiments, the de-oxidation process (performed at step s10 as described above) is implemented by filling a bottom portion of the container of the de-oxidation chamber with the substance (such that the component is wholly above the level of the substance), sealing the container and heating the container so that the substance is vapourised. This vapourised substance de-oxidises the component. However, in other embodiments the container of the de-oxidation chamber is filled with the substance such that component is either partially or wholly submerged in the substance. The container may then be heated so that the substance is liquidised or vapourised. This liquidised/vapourised further substance tends to oxidise the component. In cases in which the component is not submerged in the further substance to any extent, the container may then be heated so that the substance is vapourised. This vapourised further substance tends to oxidise the component.

[0296] An advantage provided in embodiments in which the component is not submerged in the liquidised substance/further substance to any extent (i.e., the component is de-oxidised or re-oxidised by a vapour) is that, after treatment, the component is only lightly coated in a waste product (e.g., salt). This tends to facilitate washing/cleaning of the component. Also, removal of the component from the treatment vessel tends to be facilitated. This tends to be in contrast to embodiments in which the component is either wholly or partially immersed in liquidised substance/further substance. In such embodiments, after treatment the component tends to be at least partially encased in solid waste material (which may be removed somehow, e.g., usually by hot water washing). This problem of encasement of the component can be addressed by providing a method of separating the components from the liquid before the waste solidifies.

[0297] A further advantage provided in embodiments in which the component is not submerged in the liquidised substance/further substance to any extent (i.e., the component is de-oxidised or re-oxidised by a vapour) is that less substance/further substance tends to be used. This tends to provide cost savings.

[0298] In the above embodiments, the substance comprises a chemical de-oxidant and a flux. However, in other embodiments the substance comprises a different appropriate set of constituents. For example, in other embodiments, no flux is used. Also, in other embodiments different relative proportions of chemical de-oxidant to flux are used to those used in the above embodiments.

[0299] In the above embodiments, the further substance comprises an oxidising chemical and a flux. However, in other embodiments the further substance comprises a different appropriate set of constituents. For example, in other embodiments, no flux is used. Also, in other embodiments different relative proportions of oxidising chemical to flux are used to those used in the above embodiments.

[0300] In the above embodiments, the chemical de-oxidant in the substance is calcium oxide. However, in other embodiments, one or more different chemical de-oxidants are used instead of or in addition to calcium. For example, sodium or a different alkali earth metal may be used.

[0301] In the above embodiments, the oxidising chemical in the further substance is calcium oxide. However, in other embodiments, one or more different oxidising chemicals are used instead of or in addition to calcium oxide. For example, sodium oxide may be used.

[0302] In the above embodiments, the flux used in the substance and the further substance is calcium chloride. However, in other embodiments, one or more different flux materials are used instead of or in addition to calcium chloride in either, or both, of the de-oxidation and re-oxidation process. For example, sodium chloride may be used. A sodium chloride/calcium chloride eutectic mixture has a lower melting temperature than calcium chloride. This may be desirable in some processes.

[0303] In the above embodiments, the substance and further substance solid powder/pellets which are then liquidised/vapourised. However, in other embodiments, the flux and or
chemical reactant of the substance and/or further substance may have a different state. For example, a flux in vapour form may be used.

[0304] In the above embodiments, the de-oxidation process comprises heating the de-oxidation chamber to preferably 1000°C to 1200°C for 0.5 to 100 hours. Performing the de-oxidation process at a relatively high temperature, e.g. 1100°C, advantageously tends to provide that the oxygen with the produced part is distributed in that part more evenly (i.e. the oxygen concentration throughout the produced part is more homogenised). This may eliminate or reduce the need to perform further homogenisation for some objects. However, in other embodiments during the de-oxidation process, the de-oxidation chamber is heated to a different appropriate temperature. Also, in other embodiments the de-oxidation process is performed for a different appropriate time-period.

[0305] In the above embodiments, the re-oxidation process comprises heating the re-oxidation chamber to preferably 1000°C to 1200°C for 0.5 to 100 hours. However, in other embodiments during the re-oxidation process, the re-oxidation chamber is heated to a different appropriate temperature. Also, in other embodiments the re-oxidation process is performed for a different appropriate time-period.

[0306] In the above embodiments, the oxygen homogenisation process comprises heating the de-oxidation chamber to preferably 1000°C to 1300°C for 0.5 to 100 hours. However, in other embodiments during the oxygen homogenisation process, the homogenisation chamber is heated to a different appropriate temperature. Also, in other embodiments the homogenisation process is performed for a different appropriate time-period, e.g. between 50 and 100 hours, or between 70 and 100 hours.

[0307] In the above embodiments, the component is cleaned using water to remove the flux, and a dilute acid to remove other residues. However, in other embodiments the component is cleaned using a different appropriate process. Also, in other embodiments the component is not cleaned.

[0308] In the above embodiments, during de-oxidation (or re-oxidation) of the component oxygen is removed (or added) substantially uniformly from the surface of the component. However, in other embodiments during these processes oxygen is not removed or added substantially uniformly. For example, in other embodiments a portion of the component may be masked from the vapour (or made not to be in contact with the vapour) during a de-oxidation and/or re-oxidation process.

[0309] In the above embodiments, the de-oxidation and re-oxidation process are stopped by stopping heating the relevant chamber after the relevant time-period. However, in other embodiments de-oxidation and/or re-oxidation of the component may be stopped by a different method. For example, in other embodiments a desired average oxygen content may be achieved by supplying only enough de-oxidation/re-oxidation chemical for a certain proportion of the oxygen to be removed/introduced before the reactants are exhausted or the chemical reactions come to equilibrium.

[0310] In the above embodiments, the above described process is implemented to remove (or add) bulk oxygen from a component part. However, in other embodiments the process may be implemented to remove or add a different interstitial solute from the component. For example, in other embodiments the process is implemented to remove carbon, hydrogen or nitrogen from the component.

[0311] It should be noted that certain of the process steps depicted in the above described flowcharts may be omitted (for example, the re-oxidation process of step s12 or the homogenisation step s14) or such process steps may be performed in differing order to that presented above and shown in the Figures. Furthermore, although all the process steps have, for convenience and ease of understanding, been depicted as discrete temporally-sequential steps, nevertheless some of the process steps may in fact be performed simultaneously or at least overlapping to some extent temporarily.

[0312] In the above embodiments, a separate homogenisation process is performed (e.g. at step s14) to homogenise the oxygen concentration level throughout the component/object. However, in other embodiments, such a process is not performed. For example, the homogenisation process may not be performed because variation in oxygen content in the produced object can be tolerated. Also for example, the homogenisation process may not be performed because the de-oxidation and/or re-oxidation processes have sufficiently homogenised the oxygen in the produced object.

1. A method of producing an object, the object being made of titanium or titanium alloy, the object having a desired shape, the object being a non-porous object, the method comprising:

   providing some titanium or titanium alloy, the provided titanium or titanium alloy having a first average oxygen level;
   using the provided titanium or titanium alloy, performing a net-shape or near-net shape manufacturing process to produce an intermediate object,
   the intermediate object having the desired shape,
   the intermediate object being a non-porous object,
   the intermediate object having a second average oxygen level, the second average oxygen level being greater than or equal to the first average oxygen level;
   performing a deoxidation process to remove an amount of oxygen from a surface of the intermediate object such as to provide the intermediate object with a third average oxygen level, the third average oxygen level being less than the second average oxygen level; and
   thereafter, performing a homogenisation process on the intermediate object to increase the uniformity of the oxygen level within the intermediate object, thereby providing the object.

2. (canceled)

3. A method according to claim 1, wherein the deoxidation and homogenisation processes are performed such that the intermediate object substantially retains its shape and solid density.

4. (canceled)

5. A method according to claim 1, wherein the object is made of Ti—6Al—4V.

6-8. (canceled)

9. A method according to claim 1, wherein the deoxidation process comprises exposing at least part of the intermediate object to a fluid, wherein the fluid comprises a chemical reactant, and the chemical reactant has a different level of reactivity to oxygen than the titanium or titanium alloy.

10. (canceled)

11. A method according to claim 9, wherein the chemical reactant is calcium.

12. A method according to claim 9, wherein the fluid further comprises a flux material, the flux material being for
facilitating the removal of a product of a reaction between the chemical reactant and the titanium or titanium alloy from a surface of the object.

13. A method according to claim 12, wherein the flux material is calcium chloride.

14. (canceled)

15. A method according to claims 9, wherein the fluid is a vapour.

16. A method according to claim 1, wherein the produced object and the intermediate object each have a solid density within the range 92% to 100%.

17. A method according to claim 1, wherein the amount of titanium or titanium alloy is provided in powder form.

18-19. (canceled)

20. A method according to claim 1, wherein the net-shape or near-net shape manufacturing process is selected from the group consisting of casting, permanent mould casting, powder metallurgy, linear friction welding, metal injection moulding, rapid prototyping, spray forming, and superplastic forming.

21. A method according to claim 20, wherein the net-shape or near-net shape manufacturing process is metal injection moulding.

22. A method according to claim 1, wherein the deoxidation process comprises:

- removing an amount of oxygen from the intermediate object, thereby reducing the average oxygen level of the intermediate object; and
- adding an amount of oxygen to the intermediate object, thereby increasing the average oxygen level of the intermediate object; wherein an amount that the average oxygen level of the intermediate object is reduced by is greater than an amount that the average oxygen level of the intermediate object is increased by.

23. A method according to claim 22, wherein the step of adding the amount of oxygen is performed after the step of removing the amount of oxygen.

24. A method according to claim 1, wherein the first average oxygen level is a level within a range selected from the group of ranges consisting of: between 2300 and 10000 parts per million by weight, between 5000 and 10000 parts per million by weight, and greater than 10000 parts per million by weight.

25. A method according to claim 1, wherein the third average oxygen level is a level within a range selected from the group of ranges consisting of: between 1000 and 2300 parts per million by weight, between 1300 and 1800 parts per million by weight, between 1700 and 1800 parts per million by weight, less than 2000 parts per million by weight, less than 1300 parts per million by weight, and less than or equal to 1000 parts per million by weight.

26. A method according to claim 1, wherein the step of performing the deoxidation process comprises heating the intermediate object to between 1000° C. and 1200° C.

27. (canceled)

28. A method according to claim 1, wherein the homogenisation process is performed such that the oxygen level in the object is substantially uniform throughout the object.

29. A method according to claim 1, wherein the step of performing the homogenisation process comprises heating the object to between 1000° C. and 1300° C. for between 0.5 and 100 hours.

30. An object produced by performing a method according to claim 1.

31-32. (canceled)

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