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MENETELMÄ NIKKELIPOHJAISEEN SEOKSEEN PERUSTUVAN JAUHEEN VALMISTAMISEKSI

METHOD FOR PRODUCING A NICKEL BASE ALLOY POWDER

METHOD FOR PRODUCING A NICKEL BASE ALLOY POWDER

The invention relates to a method for producing a powder from a nickel base alloy.

WO 2015/110668 A2 discloses a method for producing iron-based powders which are atomised with the aid of a centrifuge, comprising the following method steps:

- providing an alloy having a melting point above 1040°C,
- melting the composition,
- atomising the molten composition with the aid of a centrifuge or a rotating atomisation device.

DE 10 2015 016 729 A discloses a method for producing metallic semi-finished products from an alloy with more than 50 % nickel, including the following method steps:

- an electrode is generated by VIM,
- the electrode is subjected to heat treatment in a furnace in the temperature range between 400 and 1250°C for a period of 10 to 336 hours to reduce stresses and overaging,
- the electrode is cooled in air or in the furnace depending on its dimensions, in particular depending on its diameter, to a temperature between room temperature and less than 1,250°C, in particular less than 900°C,
- the cooled electrode is then remelted by VAR at a remelting rate of 3.0 to 10 kg/minute to form a VAR block,
- the VAR block is heat-treated in a furnace in the temperature range between 400 and 1250°C for a period of 10 to 336 hours,
- the VAR block is cooled in air or in the furnace depending on its dimensions, in particular depending on its diameter, to a temperature between room temperature and less than 1,250°C, in particular less than 900°C,
- the VAR block is remelted again at a remelting rate of 3.0 to 10 kg/minute,
- the remelted VAR block is subjected to heat treatment in the temperature range between 400 and 1250°C for a period of 10 to 336 hours,
- the VAR block is then brought into the desired product shape and dimensions by hot and/or cold forming.

  EP 1 377 690 B1 has disclosed a method for producing a nickel-based super-alloy which is substantially free of positive and negative segregation, wherein the method comprises the following:

- pouring an alloy, which is a nickel-based super-alloy, into a casting mold;

- annealing and overaging of the alloy by heating the alloy to at least 1200°F (649°C) for a duration of at least 10 hours;
- electro-slag remelting of the alloy at a melting rate of at least 8 lb/min (3.63 kg/min);
- keeping the alloy in the heating furnace for 4 hours after full hardening;
- holding the alloy in the heating furnace at a first temperature of 600°C (316°C) to 1800°F (982°C) for a duration of at least 10 hours;
- increasing the furnace temperature from the first temperature to a second temperature of at least 2125°C (1163°C) in such a way as to avoid the thermal stresses in the alloy;
- holding to the second temperature for a duration of at least 10 hours;
- vacuum arc remelting of a VAR electrode of the alloy at a melting rate of 8 to 11 lbs/min (3.63 to 5 kg/min) to produce the VAR block.

It is generally known that Ni- and Ni-Co alloys are used to manufacture parts with special properties with respect to corrosion resistance and good mechanical properties such as strength, heat-resistance and fracture toughness.

The object of the invention is to provide a method for producing a powder for the additive manufacturing of components, the powder having the advantageous properties of Ni- and Ni-Co alloys and being cost-effective to produce. In addition, the powder described above can be used for producing parts using hot isostatic pressing (HIP) or conventional sintering and extrusion processes as required. It is also possible to combine methods involving additive manufacturing followed by HIP treatment. In the process, the post-processing steps described below for generative manufacturing can be used for HIP parts. What is important here is that the special requirements on particle size distribution, particle shape and pourability of the powder must be met.

This object is achieved by a method for producing a powder from a nickel base alloy, the contents (in % by weight) being defined as follows:

C	max. 0.5 %
S	max. 0.15 %, in particular max. 0.03 %
N	max. 0.25 %
Cr	14 - 35 %, in particular 17 - 28 %
Ni	remainder (> 38 %)
Mn	max. 4 %
Si	max. 1.5 %
Mo	> 0 - 22 %
Ti	≤ 4 %, in particular < 3.25 %

Nb up to 6.0 %

Cu up to 3 %, in particular up to 0.5 %

Fe ≤ 50 %

P max. 0.05 %, in particular max. 0.04 %

Al up to 3.15 %, in particular up to 2.5 %

Mg max. 0.015 %

V max. 0.6 %

Zr max. 0.12 %, in particular max. 0.1 %

W up to 4.5 %, in particular up to max. 3 %

Co up to 28 %

B ≤ 0.125 %

O > 0.00001 – 0.1 %

and production-related impurities,

wherein:

Ni + Fe + Co 56 - 80 %

Nb + Ta ≤ 6.0 %, by

- melting an alloy in a VIM furnace,
- holding the liquid melt for 5 min to 2 hours for homogenisation,
- setting a closed atomisation system with a supplied gas to a dew point from -10°C to 120°C,
- using a nozzle, blowing the melt into a gas stream at a gas flow rate of from 2 qm^3/min to 150 qm^3,
- collecting the solidified powder particles in a sealed, gas-impermeable container, wherein
- the particles have a particle size from 5 µm to 250 µm,
- the particles of the powder are spherical,
- the powder has gas inclusions from 0.0 to 4 % pore area (pores > 1 µm) as a proportion of the total area of evaluated objects,
- the powder has a bulk density of 2 up to the density of the alloy of about 8 g/cm^3,
- the powder is packed airtight under the protective gas atmosphere with argon.

Advantageously, the powder alloy should have the following composition (in % by weight):

C max. 0.25 %

S max. 0.03 %

N > 0 - 0.15 %

Cr 17 - 28 %, in particular 17 - 24 %

Ni remainder (38 - 72 %)

Mn max. 2 %, in particular max. 1 %

Si max. 1.2 %, in particular ≤ 0.5 %

Mo > 0 - 21, in particular 2.5 - 21 %

Ti > 0 - < 3.25 %

Nb > 0 - 5.5 %

Cu max 3 %, in particular up to 0.5 %

Fe > 0 - 38 %

P max. 0.04 %

Al > 0 - 0.8 %

Mg max. 0.015 %

V max. 0.4 %

Zr max. 0.1 %

W up to 0.5 %

Co ≤ 10 %

B ≤ 0.01 %

Pb max. 0.001 %, in particular max. 0.0005 %

Se max. 0.0005 %, in particular max. 0.0003 %

Bi max 0.00005 %, in particular max. 0.00003 %

O > 0.00001 – 0.1 %

Nb +Ta > 0 - 5.5 %

and production-related impurities,

wherein:

Ni + Fe + Co 57 – 77 %.

Moreover, there is the option of providing the following powder alloy composition for concrete applications:

C > 0 - 0.1 %, in particular > 0 - 0.08 %

S max. 0.015 %

N > 0 - 0.03 %

Cr 17 – 24 %

Ni remainder (> 50 – 63 %)

Mn max. 1.0 %, in particular max. 0.5 %

Si max. 0.5 %

Mo 2.8 – 16.5 %

Ti > 0 – 1.15 %

Nb > 0 – ≤ 5.5 %

Cu max. 0.5 %

Fe > 0 - 25 %

P max. 0.015 %

Al 0.1 – 0.6 %

Mg max. 0.015 %

V max. 0.2 %

Zr max. 0.1 %

W up to 0.2 % in particular > 0 – 0.2 %

Co ≤ 2.5 %, in particular ≤ 1.0 %

B ≤ 0.01 %

Pb max. 0.001 %, in particular max. 0.0005 %

Se max. 0.0005 %, in particular max. 0.003 %

Bi max 0.0005 %, in particular max. 0.0003 %

O > 0.00001 – 0.1 %

Nb +Ta > 0 - 5.5 %

Al + Ti < 1.2 %

and production-related impurities,

wherein:

Ni + Fe + Co 59 - 72 %.

The following initial manufacturing steps are conceivable:

- a master alloy block with defined chemical analysis is produced by melting in the VIM furnace, VIM/ESU, VIM/ESU/VAR, VIM/VAR, VOD or VLF with subsequent remelting in the ESR and/or VAR if required, depending on the purity requirements of the material,
- a master alloy block is divided up into small pieces by sawing.
- the pieces of the master alloy are melted in a VIM furnace, or
- alloying elements of a defined weight are melted in a VIM according to the chemical analysis,

  or
- the combination between master alloy material, process-related scrap (including scrap from customers such as recycled powder and support structures or defective parts) and new alloying elements at a proportion of 0 to 100 %. The exact proportion is considered in each case, taking into account qualitative, economic and ecological aspects. It can be advantageous if the master alloy block is

subjected to surface treatment (e.g. brushing, grinding, pickling, separating, peeling, etc.) before cutting. Faults can be removed here which are not eliminated by further remelting and may be harmful for later applications. In addition, the use of possible master alloys leads to compliance with the highest quality requirements on the chemical purity of the powder, which can only be guaranteed by prior remelting processes,

- holding the liquid melt from 5 min to 2 hours for homogenization.

The following further processing steps can follow the alternative initial manufacturing steps:

- the closed atomisation system is set with argon gas to a dew point of from -10°C to -120°C, preferably in the range from -30°C to -100°C,
- the melt is blown through a nozzle into an argon gas stream with a gas flow rate from 2 m^3/min to 150 m^3/min,
- the solidified powder particles are collected in a sealed, gas-impermeable container,
- the particles have a particle size of 5 μm to 250 μm, with preferred ranges being between 5 and 150 μm, or 10 and 150 μm,
- the particles of the powder are spherical,
- the powder has gas inclusions constituting from 0.0 to 4 % of the pore area (pores > 1 μm) in proportion to the total area of evaluated objects, wherein preferred regions lie between 0.0 and 2 %. The number of gas inclusions in the powder enables a low residual porosity of the manufactured parts.
- the powder has a bulk density of 2 up to the density of the alloy of about 8 g/cm^3, wherein preferred ranges are values of 4 to 5 g/cm^3,
- the powder is packed airtight under an argon protective gas atmosphere.

The powder is preferably produced in a vacuum inert gas atomisation system (VIGA). In this plant, the alloy is melted in a vacuum induction melting furnace (VIM), fed into a casting funnel which leads to a gas nozzle in which the molten metal is atomised into metal particles under high pressure of 5 to 100 bar using inert gas. The melt is heated in the melting pot at 5 to 400 °C above the melting point. The metal flow rate during atomisation is 0.5 to 80 kg / min and the gas flow rate is 2 to 150 m^3/ min. The rapid cooling causes the metal particles to solidify in spherical form (spherical particles). The inert gas used for atomisation can contain 0.01 to 100 % nitrogen as required. The gas phase is then separated from the powder in a cyclone and the powder is then packaged.

Alternatively, the powder can be produced using the so-called EIGA method instead of VIGA. A prefabricated alloy block in the form of a rotating electrode is thereby melted without contact by an induction coil. The melt drips from the electrode directly into the gas stream of a gas nozzle.

The alloy block for EIGA can in turn be produced using the VIM, ESU, VAR, VOD or VLF melting methods and combinations thereof and can optionally undergo hot forming processes such as forging and rolling. It is advantageous to clean the surface of the block by means of a treatment such as grinding and/or peeling before using the EIGA method.

The inert gas used in powder production can be optionally argon or a mixture of argon with 0.01 to < 100 % nitrogen. Possible restrictions of the nitrogen content can be:

0.01 to 80 %

0.01 to 50 %

0.01 to 30 %

0.01 to 20 %

0.01 to 10 %

0.01 to 10 %

0.1 to 5 %

0.5 to 10 %

1 to 5 %

2 to 3 %

Alternatively, the inert gas can be optionally helium.

The inert gas may preferably have a purity of at least 99.996 vol%. In particular, the nitrogen content should be from 0.0 to 10 ppmv, the oxygen content from 0.0 to 4 ppmv and the H_2O content of ≤ 5 ppmv.

In particular, the inert gas may preferably have a purity of at least 99.999 vol%. In particular, the nitrogen content should be from 0.0 to 5 ppmv, the oxygen content from 0.0 to 2 ppmv and the H_2O content of ≤ 3 ppmv. The dewpoint in the plant is in the range of -10 to -120°C. It is preferably in the range of -30 to -100°C.

The pressure during powder atomisation can preferably be 10 to 80 bar.

The parts and components or layers on parts and components produced using additive manufacturing are built up with layer thicknesses of 5 to 500 µm and have a textured structure with grains of an average grain size of 2 µm to 1000 µm stretched in the direction of construction directly after production. The preferred range is between 5 µm and 500 µm.

The parts and components or layers on parts and components produced by additive manufacturing can optionally be subjected to solution annealing in the temperature range from 700°C to 1250°C for 0.1 min to 70 h, possibly under protective gas, such as argon or hydrogen, followed by cooling in air, in the moving annealing atmosphere or in a water bath. The surface can then optionally be cleaned or processed by pickling, blasting, grinding, turning, peeling or milling. Such processing can take place either partially or completely even before annealing.

The parts and components or layers on parts and components produced using additive manufacturing can optionally be subjected to homogenization, stress relief, solution and/or precipitation hardening annealing. The heat treatments may be carried out under protective gas, such as argon or hydrogen, followed by cooling in the furnace, if necessary, under protective gas, in air, in the moving annealing atmosphere or in a water bath.

The parts and components or layers on parts and components produced using additive manufacturing have an average grain size of 2 µm to 2000 µm after annealing. The preferred range is between 20 µm and 500 µm.

The parts and components or layers on parts and components generated from the powder produced according to the invention using additive manufacturing should preferably be used in areas in which the raw material is also used as a wrought or cast alloy with related analysis.

Additive manufacturing also covers terms such as generative manufacturing, rapid technology, rapid tooling, rapid prototyping and the like.

A general distinction is made here:

3D printing with powders,

Selective laser sintering and

Selective laser melting

Laser deposition welding

Selective electron beam welding.

The abbreviations used here are defined as follows:

VIM Vacuum Induction Melting

VIGA Vacuum Inert Gas atomisation system

VAR Vacuum Arc remelting

VOD Vaccum Oxygen Decaburization

VLF Vacuum Ladle Furnace

EIGA Electrode Induction Melting Gas Atomisation

Advantageous further developments of the method according to the invention can be found in the dependent claims.

The spread range for the particle size of the powder is between 5 and 250 µm, with preferred ranges being between 5 and 150 µm, or 10 and 150 µm.

The powder has gas inclusions making up from 0.0 to 4 % of the pore area (pores > 1 µm) in proportion to the total area of evaluated objects, wherein preferred ranges lie from

0.0 bis 2 %

0.0 to 0.5 %

0.0 to 0.2 %

0.0 to 0.1 %

0.0 to 0.05 %

The powder has a bulk density of 2 to the density of the alloy of approx. 8 g/cm^3, wherein preferred ranges can lie within the following values:

4-5 g/cm^3

2-8 g/cm^3

2-7 g/cm^3

3-6 g/cm^3

The number of gas inclusions in the powder enables a low residual porosity of the manufactured parts.

Compared to the prior art, the centrifuge method is not used, as a result of which the system's operating time is optimized. The subsequent refining processes optimize the quality of the powder for additive manufacturing.

It can be advantageous if the block is subjected to surface treatment (e.g. brushing, grinding, pickling, separating, peeling, etc.) before cutting. Faults can be removed here which cannot be eliminated by further remelting and may be harmful for later applications.

The method according to the invention can be applied to every Ni- or Ni-Co based alloy.

An alloy composition is presented below which can be produced as a powder using the method parameters according to the invention. All information is given in % by weight:

C	max. 0.5 %
S	max. 0.150 %, in particular max. 0.03 %
Cr	17 - 32 %, in particular 17 - 26 %
Ni	45 - 72 %, in particular 45 - 71 %
Mn	max. 1 %
Si	max. 1 %
Mo	> 0 - 10 %

Ti max.3.25 %, in particular max. 2.7 %

Nb max. 5.5 %

Cu max. 5 %, in particular max. 0.5 %

Fe max. 25 %

Al max. 3.15 %, in particular max. 2.5 %

V max. 0.6 %

Zr max. 0.12 %, in particular max. 0.1 %

Co max. 28 %

O 0.00001 - 0.1 %

and production-related impurities.

Moreover, the following elements can be given as follows (in % by weight):

Nb+Ta max. 6 %

B max 0.02 %, in particular max. 0.006 %

Se max. 0.0005 %

Bi max. 0.00005 %

Pb max. 0.002 %

P max. 0.03 %, in particular max. 0.02 %

Advantageously, the following elements can be set as follows (in % by weight):

C 0.015 - 0.5 %; in particular 0.015 - 0.2 %

S max. 0.1 %, in particular max. 0.02 %

Cr 17 - 25 %

Ni 45 - 58 %

Mn max. 0.6 %

Si max. 0.4 %

Mo 0 - 6.1 %

Ti 0.1 - 2.7 %

Al max. 1.7 %

Co max. 13 %

To improve the mechanical properties, the elements boron and carbon can also be set as follows:

C 0.015 – 0.5 %

B 0.006 – 0.125 %

In the process, it is also advantageous for the sum C + B to lie between 0.1875 and 0.530 %, between 0.156 and 0.625 %, in particular in the range of 0.16 and 0.6 %, especially in the range of 0.1875 and 0.530 %, and for the proportion C/B to lie between 12 and 18.

An example of a powder made from a nickel alloy based on Alloy 718 is presented below (data in % by weight):

C	max. 0.08 %
S	max. 0.015 %
Cr	17 - 21 %
Ni	50 - 55 %
Mn	max. 0.35 %
Si	max. 0.35 %
Mo	2.8 - 3.3 %
Ti	0.65 - 1.15 %
Nb	4.75 - 5.5 %
Cu	max. 0.3 %
Fe	6 - 25 %
P	max. 0.015 %
Al	0.2 - 0.8 %
Co	max. 1 %
B	max. 0.006 %
Ta	max. 0.05 %
O	0.00001 - 0.1 %
Pb	max. 0.001 %, in particular max. 0.0005 %
Se	max. 0.0005 %, in particular max. 0.0003 %
Bi	max 0.00005 %, in particular max. 0.00003 %

Alternatively, this alloy can also have higher Ni contents (in % by weight).

C	max. 0.1 %
S	max. 0.03 %, in particular max. 0.02 %
Cr	17 - 32 %, in particular 17 - 30 %
Ni	58 - 79, in particular 58 - 72 %
Nb	max. 4.1 %, in particular max. 0.6 %
Fe	max. 18 %
C	max. 0.1 %
S	max. 0.02 %
Mn	max. 1 %
Si	max. 1 %
Mo	> 0 - 10 %
Ti	max 3.25 %, in particular max. 2.7 %

Cu	max. 0.5 %
Al	max. 3.15 %
V	max. 0.6 %
Zr	max. 0.1 %
Co	max. 15 %, in particular max. 7 %
O	0.00001 - 0.1 %

and optionally, if necessary (in % by weight):

B	max 0.008 %, in particular max. 0.006 %
Se	max. 0.0005 %
Bi	max. 0.00005 %
Pb	max. 0.002 %
P	max. 0.03 %, in particular max. 0.02 %

Further limitations are conceivable as follows (in % by weight):

C	0.01 - 0.04 %
Mn	max. 0.5 %
Si	max. 0.5 %
Cu	max. 0.2 %

and optionally, if necessary (in % by weight):

Mo	8 - 10 %

Optionally, the oxygen content of the produced alloy can lie within 0.00001 – 0.1 %, 0.0001 – 0.1 %, 0.001 – 0.1 %, 0.001 – 0.002 % or 0.0015 – 0.002 %.

Optionally, the oxygen content can also be set as follows:

- 0.00001 – 0.1
- 0.00002 – 0.1
- 0.00005 – 0.1
- 0.00008 – 0.1
- 0.0001 – 0.1
- 0.0002 – 0.1
- 0.0005 – 0.1
- 0.0008 – 0.1
- 0.001 – 0.1
- 0.002 – 0.1
- 0.005 – 0.1
- 0.008 – 0.1
- 0.010 – 0.1

- 0.00001 – 0.10
- 0.00001 – 0.08
- 0.00001 – 0.05
- 0.00001 – 0.03
- 0.00001 – 0.02

The nitrogen content must be less than or equal to 0.100 % in order to ensure the producibility and utility of the alloy. If the nitrogen content is too high, nitrides are formed which have a negative effect on the properties of the alloy. Too low of a nitrogen content increases the costs. The nitrogen content is therefore ≥ 0.00001 %. The following restrictions of the nitrogen content are conceivable:

- 0.00001 – 0.1
- 0.00002 – 0.1
- 0.00005 – 0.1
- 0.00008 – 0.1
- 0.0001 – 0.1
- 0.0002 – 0.1
- 0.0005 – 0.1
- 0.0008 – 0.1
- 0.001 – 0.1
- 0.002 – 0.1
- 0.005 – 0.1
- 0.008 – 0.1
- 0.010 – 0.1
- 0.00001 – 0.10
- 0.00001 – 0.08
- 0.00001 – 0.05
- 0.00001 – 0.03
- 0.00001 – 0.02

The particle sizes of nitrides as well as carbides and/or carbonitrides are very small (approx. < 8 µm), both in the powder and in manufactured components (3D-printed samples). In some cases, the aforementioned particles may not be present or may only become visible after heat treatment. Small particle sizes of precipitates containing N have a positive effect on high-temperature properties and resistance to alternating loads (low-cycle fatigue - LCF), since precipitates containing N act as crack initiation sites in conventionally produced alloys.

The argon content must be less than or equal to 0.08 % in order to ensure the producibility and utility of the alloy. Argon cannot be dissolved in the γ-matrix, which is why it can have a negative effect on the mechanical properties of the part, as argon inclusions can act as a crack initiation point. Too low of an argon content increases the costs. The argon content is therefore ≥ 0.0000001 %(≥ 1 ppb). The following restrictions on the argon content are conceivable, wherein the argon contents from the powder production as well as from parts production are included:

- 0.0000001 – 0.05
- 0.0000002 – 0.05
- 0.0000001 – 0.005
- 0.0000001 – 0.002
- 0.0000001 – 0.001

The method according to the invention should preferably be used for the following alloys:

- Alloy 601
- Alloy 602 CA and its variant MCA
- Alloy 2120 MoN
- Alloy B2
- Alloy C 276
- Crofer 22 APU
- Crofer 22 H

Table 1 shows analysis ranges of the aforementioned alloys as examples.

Table 1

	Alloy 601	Alloy 602 CA / MCA	FM 602	Alloy 617 (B/OCC)	FM 617	Alloy 625	FM 625	Alloy 690	Alloy 699XA
C	0.03-0.1	0.15-0.25	0.15-0.25	0.05-0.08	0.05-0.15	-0.03	-0.1	-0.05	0.005-0.12
S	-0.015	-0.01		-0.008		-0.01		-0.015	-0.01
N									-0.05
Cr	21-25	24-26	24-26	21-23	20-24	21-23	20-23	27-31	26-30
Ni	58-63	59-66	59-66	45-58	50-61	58-71	58-71	58-66	62-72
Mn	-1	-0.5	-0.5	-0.5	-1	-0.5	-0.5	-0.5	-0.5
Si	-0.5	-0.5	-0.5	-0.3	-1	-0.4	-0.5	-0.5	-0.5
Mo				8-10	8-10	8-10	8-10		
Ti	-0.5	0.1-0.2	0.1-0.2	0.25-0.5	-0.6	-0.4	-0.4		-0.6
Nb				-0.6			3-4.1		-0.5
Cu	-0.5	-0.1	-0.1		-0.5		-0.5	-0.5	-0.5
Fe	-18	8-11	8-11	-1.5	-3	-5	-5	7-11	-2.5
P	-0.02	-0.02		-0.012	-0.03	-0.01			-0.02
Al	1-1.7	1.8-2.4	1.8-2.4	0.8-1.3	0.8-1.5	-0.4	-0.4		2-3
Mg									
Ca									
V				-0.6					
Zr		0.01-0.1	0.01-0.1						-0.1
W					-0.5				
Co			-1	11-13	10-15	-1			
B	-0.006			0.001-0.005					-0.008
Ni + Co									
Nb + Ta			3.2-3.8			3.2-3.8	3.2-3.8		
Additional elements									

- Alloy 617 and its variants 617 B and 617 OCC
- Alloy 625
- Alloy 690
- Alloy 699XA
- Alloy 718 and its variants
- Alloy 780

- Alloy 788
- Alloy 80A
- Alloy 81
- Alloy X-750
- Alloy C-263
- Alloy K-500
- Waspaloy
- FM 625
- FM 617 and
- FM 602
- Alloy 31
- Alloy 31 Plus
- Alloy 25
- Alloy 28
- Alloy 33
- Alloy 59
- Alloy 188
- Alloy 310 L
- Alloy 330
- Alloy 333
- Alloy 400
- Alloy 600 + 600 L
- Ally 800 + 800 H, HP, L
- Alloy 825 + 825 CTP
- Alloy 925
- Alloy 926

	Alloy 718	Alloy 718 CTP	FM 718	Alloy 780	Alloy 788	Waspaloy	Alloy C-263	Alloy 80A	Alloy 81
C	-0.08	-0.045	-0.08	-0.1	0.04-0.1	0.02-0.1	0.04-0.08	0.04-0.1	-0.08
S	-0.015	-0.01		-0.015	-0.01	-0.03	-0.007	-0.015	-0.02
N				-0.03					
Cr	17-21	17-21	17-21	16-20	18-21	18-21	19-21	18-21	28-32
Ni	50-55	50-55	50-55	26-62	51-69	49.6-62.5	50-55	65-79	59-66
Mn	-0.35	-0.35	-0.3	-0.5	-1	-1	-0.6	-1	-0.7
Si	-0.35	-0.35	-0.3	0.3	-0.5	-0.75	-0.4	-1	-0.7
Mo	2.8-3.3	2.8-3.3	2.8-3.3	2-4		3.5-5	5.6-6.1		-0.5
Ti	0.65-1.15	0.8-1.15	0.7-1.1	0.1-1	1.8-2.7	2.75-3.25	1.9-2.4	1.8-2.7	1.5-2.1
Nb	4.75-5.5	Nb+Ta	4.8-5.5						
Cu	-0.3	-0.23	-0.3	-0.5	-0.2	-0.5	-0.2	-0.2	-0.25
Fe	6-25	12-24	-24	-10	8-15	-2	-0.7	-1.5	-1.5
P	-0.015	-0.01	-0.015	-0.03	-0.02	-0.03	-0.015		
Al	0.2-0.8	0.4-0.6	0.2-0.8	1-3	1-1.8	1.2-1.6	0.3-0.6	1-1.8	-1.2
Mg				-0.01					-1
Ca				-0.01					
V									
Zr				-0.05		0.02-0.12			0.01-0.1
W									
Co	-1	-1		15-28	3-7	12-15	19-21		01. Mrz
B	-0.006	-0.006	-0.006	-0.02	-0.006	0.003-0.01	-0.005		-0.006
Ni + Co									
Nb+Ta		4.87-5.2		4-6					
Additional elements									

C	Alloy K-500	Alloy X-750	Alloy 31 Plus®	Alloy 28	Alloy 31	Alloy 33	Alloy 59	Alloy 188
S	-0.18	-0.08	≤ 0.01	≤ 0.015	≤ 0.015	≤ 0.015	≤ 0.01	0.05 to 0.15
N	-0.01	-0.01	≤ 0.01	≤ 0.01	≤ 0.01	≤ 0.01	≤ 0.01	≤ 0.015
Cr			0.1 to 0.25	0.04 to 0.07	0.15 to 0.25	0.35 to 0.6		
Ni		14-17	26.0 to 27.0	26 to 28	26 to 28	31 to 35	22 to 24	20 to 24
Mn	63-70	70-77.5	33.5 to 35.0	30 to 32	30 to 32	30 to 33	57 to 63	20 to 24
Si	-1.5	-1	1.0 to 4.0	≤ 2	≤ 2	≤ 2	≤ 0.5	≤ 1.25
Mo	-0.5	-0.5	≤ 0.10	≤ 0.7	≤ 0.3	≤ 0.5	≤ 0.1	0.2 to 0.4
Ti			6.0 to 7.0	3 to 4	6 to 7	0.5 to 2	15 to 16.5	
Nb	0.35-0.85	2.25-2.75						
Cu		0.7-1.2						
Fe	27-33	-0.5	0.5 to 1.5	1 to 1.4	1 to 1.4	0.3 to 1.2	≤ 0.5	
P	0.5-2	5-9	22.5 to 33.5	32 to 40	29 to 37	26 to 38	≤ 1.5	≤ 3
Al	-0.02		≤ 0.02	≤ 0.02	≤ 0.02	≤ 0.02	≤ 0.015	≤ 0.015
Mg	2.3-3.15	0.4-1	≤ 0.3				0.1 to 0.4	≤ 0.2
Ca								
V								
Zr								
W								
Co								13 to 16
B		-1					≤ 0.3	
Ni + Co								≤ 0.01
Nb + Ta								
Additional elements		0.7-1.2						

C	Alloy 310 L	Alloy 330	Alloy 333	Alloy 400	Alloy 600	Alloy 600 L	Alloy 800	Alloy 800 H	Alloy 800 HP
S	≤ 0.02	≤ 0.15	0.03 to 0.06	≤ 0.15	0.05 to 0.1	≤ 0.025	≤ 0.1	0.06 to 0.10	0.06 to 0.1
N	≤ 0.005	≤ 0.015		≤ 0.02	≤ 0.015	≤ 0.01	≤ 0.01	≤ 0.01	≤ 0.01
Cr									
Ni	24 to 25	15 to 17	24 to 26		14 to 17	14 to 17	19.0 to 21.5	19 to 21	19 to 22
Mn	20 to 21	33 to 37	44 to 47	≥ 63	≥ 72	≥ 72	30.0 to 32.0	30 to 32	30 to 32
Si	≤ 1	≤ 2	1.2 to 2	≤ 2	≤ 1	≤ 1	0.5 to 1.0	0.5 to 1	0.5 to 1
Mo	≤ 0.15	1 to 2	0.8 to 1.2	≤ 0.5	≤ 0.5	≤ 0.5	0.2 to 0.6	0.2 to 0.6	0.2 to 0.6
Ti	≤ 0.1		2.5 to 3.5						
Nb			0.1 to 0.2		≤ 0.3	≤ 0.3	0.20 to 0.5	0.2 to 0.6	0.3 to 0.6
Cu									
Fe			≤ 0.5	28 to 34	≤ 0.5	≤ 0.5	≤ 0.5	≤ 0.5	≤ 0.5
P		42 to 51	13 to 22	1 to 2.5	6 to 10	6 to 10	41 to 47	43 to 50	43 to 50
Al	≤ 0.02	≤ 0.045			≤ 0.02	≤ 0.015	≤ 0.015	≤ 0.015	≤ 0.015
Mg				≤ 0.5	≤ 0.3	≤ 0.3	0.20 to 0.40	0.2 to 0.6	0.3 to 0.6
Ca									
V									
Zr									
W									
Co			2.5 to 3.5						
B			2.5 to 3.5						
Ni + Co						≤ 0.006			
Nb + Ta									
Additional elements									
							Al + Ti max. 1.0%	Al + Ti max 0.7%	Al + Ti 0.85% - 1.2%

C	Alloy 800 L	Alloy 825	Alloy 825 CTP	Alloy 925	Alloy 926	Alloy 2120 MoN	Alloy B-2
S	≤ 0.025	≤ 0.025	≤ 0.015	≤ 0.03	≤ 0.020	≤ 0.01	≤ 0.01
N	≤ 0.01	≤ 0.015	≤ 0.005	≤ 0.03	≤ 0.010	≤ 0.01	≤ 0.01
Cr			≤ 0.03		0.15 to 0.25	0.02 to 0.15	
Ni	20 to 22	19.5 to 23.5	21 to 23	19.5 to 22.5	20.0 to 21.0	20.0 to 23.0	0.4 to 1
Mn	32 to 34	38 to 46	39 to 43	42 to 46	24.00 to 26.00	56	64 to 72
Si	0.5 to 1	≤ 1	0.5 to 0.9	≤ 1	≤ 1.00	≤ 0.50	≤ 1
Mo	≤ 0.7	≤ 0.5	0.2 to 0.5	≤ 0.5	≤ 0.50	≤ 0.1	≤ 0.08
Ti		2.5 to 3.5	4.5 to 6.5	2.5 to 3.5	6.0 to 7.0	18.5 to 21.0	26 to 30
Nb	0.35 to 0.6	0.6 to 1.2		1.9 to 2.4			
Cu			≤ 0.15	≤ 0.5			
Fe	≤ 0.5	1.5 to 3	1.6 to 2.3	1.5 to 3	0.5 to 1.5	≤ 0.5	≤ 0.5
P	41 to 47	20 to 38	20 to 38	≥ 22		≤ 1.5	1.6 to 2
Al	≤ 0.015	≤ 0.02		≤ 0.03	≤ 0.030	≤ 0.015	≤ 0.02
Mg	0.15 to 0.4	≤ 0.2	0.06 to 0.25	0.1 to 0.5		≤ 0.4	
Ca			0.006 to 0.015				
V							
Zr							
W							
Co						≤ 0.3	
B		≤ 1	≤ 0.5			≤ 0.3	≤ 1
Ni + Co			0.002 to 0.004				
Nb + Ta							
Additional elements							
	Al + Ti 1,0%						

C		Alloy C-263	Alloy C-276	Crofer 22 APU	Crofer 22 H	NeutroShield
S		0.04 to 0.08	≤ 0.01	≤ 0.03	≤ 0.03	≤ 0.08
N		≤ 0.007	≤ 0.01	≤ 0.020	≤ 0.006	≤ 0.030
Cr					≤ 0.04	≤ 0.10
Ni		19.0 to 21.0	15 to 16.5	20.0 to 24.0	20 to 24	18.00 to 20.00
Mn		50 to 55	51 to 63		≤ 0.5	12.00 to 15.00
Si		≤ 0.6	≤ 1	0.30 to 0.80	≤ 0.8	≤ 2.00
Mo		≤ 0.4	≤ 0.08	≤ 0.50	0.1 to 0.6	
Ti		5.6 to 6.1	15 to 17			
Nb		1.90 to 2.40		0.03 to 0.20	0.02 to 0.2	
Cu					0.2 to 1	
Fe		≤ 0.2		≤ 0.50	≤ 0.5	
P		≤ 0.7	4 to 7	≥ 73	70 to 79	R
Al		≤ 0.015	≤ 0.02	≤ 0.050	≤ 0.05	≤ 0.045
Mg		0.30 to 0.60		≤ 0.50	≤ 0.1	
Ca						
V						
Zr			≤ 0.3			
W						
Co			3 to 4.5		1 to 3	
B		19.0 to 21.0	≤ 2.5			≤ 0.2
Ni + Co		≤ 0.005				0.20 to 2.25
Nb + Ta						
Additional elements						
		Ag: max. 0.0005; Zr: max. 0.02; Al+Ti: 2.40 - 2.80; Ni: residual				Si 0.75%(max)
		Pb, Ag, Zr: determination only on request				

Powders with a particle size of 5 µm to 250 µm are achieved.

Too small a particle size below 5 µm worsens the flow behavior and should therefore be avoided; too large a particle size above 250 µm worsens the behavior in additive manufacturing.

Too low a bulk density of 2 g/cm^2 worsens the behavior in additive manufacturing. The maximum possible bulk density of approx. 8 g/cm^3 is given by the density of the alloy.

The oxygen content must be less than or equal to 0.100 % in order to ensure the producibility and utility of the alloy. Too low an oxygen content increases the costs. The oxygen content is therefore ≥ 0.0001 %. The powders produced according to this method can be used in additive manufacturing to build components that have the properties of the underlying alloys.

In addition, the powder described above can be used for producing parts using hot isostatic pressing (HIP) or conventional sintering and extrusion processes. It is also possible to combine methods involving additive manufacturing followed by HIP treatment. In the process, the post-processing steps described below for generative manufacturing can be used for HIP parts.

The powder produced according to this method as well as components made from this powder (3D-printed samples) are free of nitrides as well as carbides and / or carbonitriding. If nitrides as well as carbides are in fact present, they have a particle size of < 100nm in diameter, in particular < 50nm.

After heat treatment of components made from this powder (3D-printed samples) for homogenisation, diffusion annealing above 900°C, especially above 1000°C, ideally above 1100°C, for more than 1 hour, nitrides as well as carbides and/or carbonitrides can occur in the manufactured components (3D-printed samples). These have a particle size of < 8 µm or < 5 µm in diameter, ideally < 1 µm, in particular < 500 nm. The method according to the invention is explained below by way of example:

The following melts were produced using the method according to the invention and made into powder (in % by weight):

P10057	P10153	P10154	P10155
0.033	0.002	0.002	0.003
0.001	0.002	0.002	0.002
0.007	0.004	0.004	0.003
18.56	22.46	22.48	22.72
53.44	60.91	60.71	60.5
0.03	0.2	0.19	0.18
0.06	0.01	0.01	0.01
2.98	15.43	15.42	15.48
0.95	0.01	0.01	0.01
5.39	0.01	0.01	0.01
0.03	0.01	0.01	0.01
17.82	0.52	0.66	0.67
0.007	0.002	0.002	0.002
0.46	0.24	0.31	0.22
0.001	0.001	0.001	0.001
0.001	0.001	0.001	0.001
0.02	0.16	0.15	0.15
0.01	-	-	-
0.02	0.01	0.01	0.01
0.08	0.01	0.01	0.01
0.004	0.001	0.001	0.001

P10211	P10212	P10213	P10214	P10215
0.002	0.002	0.003	0.002	0.002
0.003	0.003	0.003	0.003	0.003
0.003	0.006	0.005	0.002	0.003
22.5	22.54	22.43	22.46	22.49
60.82	60.4	61.01	60.89	60.59
0.19	0.18	0.19	0.15	0.17
0.02	0.02	0.01	0.02	0.02
15.39	15.77	15.39	15.39	15.68
0.01	0.01	0.01	0.01	0.01
0.01	0.01	0.01	0.01	0.01
0.01	0.01	0.01	0.01	0.01
0.53	0.52	0.44	0.52	0.51
0.002	0.002	0.002	0.002	0.002
0.28	0.26	0.26	0.29	0.27
0.001	0.001	0.001	0.001	0.001
0.001	0.001	0.001	0.001	0.001
0.14	0.15	0.14	0.14	0.15
-	-	-	-	-
0.01	0.01	0.01	0.01	0.01
0.07	0.09	0.06	0.08	0.06
0.001	0.001	0.001	0.001	0.001

Batch	P10030	P10036	P10038	P10039	P10046	P10049
C	0.022	0.027	0.025	0.026	0.028	0.023
S	0.0005	0.0005	0.0006	0.0005	0.0007	0.0006
N	0.007	0.008	0.006	0.008	0.005	0.008
Cr	18.33	18.38	17.94	18.35	17.94	18.95
Ni	53.07	53.24	52.33	53.24	53.32	52.2
Mn	0.04	0.03	0.18	0.1	0.03	0.11
Si	0.06	0.05	0.18	0.08	0.04	0.16
Mo	2.98	3.03	2.96	3.01	3.05	3.13
Ti	0.93	0.93	1.28	0.98	0.9	0.92
Nb	5.28	5.37	5.18	5.09	5.41	5.61
Cu	0.03	0.02	0.14	0.05	0.02	0.03
Fe	18.52	18.18	17.66	18.01	18.55	17.63
P	0.009	0.008	0.039	0.013	0.007	0.011
Al	0.55	0.58	0.8	0.6	0.52	0.53
Mg	0.001	0.001	0.011	0.005	0.001	0.002
Ca	0.001	0.001	0.002	0.004	0.001	0.004
V	0.02	0.02	0.18	0.06	0.02	0.13
Zr	0.01	0.01	0.05	0.05	0.01	0.03
W	0.03	0.03	0.19	0.09	0.06	0.06
Co	0.1	0.08	0.84	0.22	0.07	0.44
B	0.004	0.004	0.007	0.004	0.004	0.01

Batch	P10156	P10157	P10207	P10208	P10209	P10210
C	0.003	0.002	0.002	0.002	0.002	0.002
S	0.002	0.002	0.003	0.003	0.003	0.003
N	0.004	0.005	0.003	0.005	0.004	0.004
Cr	22.78	22.66	22.8	22.65	22.95	22.66
Ni	60.27	60.19	60.4	60.34	60.27	60.36
Mn	0.19	0.18	0.22	0.22	0.2	0.18
Si	0.01	0.01	0.04	0.01	0.03	0.02
Mo	15.71	16.02	15.5	15.64	15.47	15.68
Ti	0.01	0.01	0.01	0.01	0.01	0.01
Nb	0.01	0.01	0.01	0.01	0.01	0.01
Cu	0.01	0.01	0.01	0.01	0.01	0.01
Fe	0.61	0.45	0.61	0.61	0.53	0.53
P	0.002	0.002	0.002	0.002	0.002	0.002
Al	0.22	0.28	0.22	0.24	0.27	0.26
Mg	0.001	0.001	0.001	0.001	0.001	0.001
Ca	0.001	0.001	0.001	0.001	0.001	0.001
V	0.14	0.14	0.14	0.14	0.13	0.14
Zr	-	-	-	-	-	-
W	0.01	0.01	0.01	0.01	0.01	0.01
Co	0.01	0.01	0.01	0.08	0.09	0.1
B	0.001	0.001	0.001	0.001	0.001	0.001

Table 2 shows the chemical compositions of the powders produced by the method according to the invention:

Both the powder as well as manufactured components (3D-printed samples) were free of nitrides and carbides.

Following an annealing at a temperature of 1100°C for 1 hour, followed by a quenching in water, particles such as nitrides and carbides and/or carbonitrides were present in the manufactured components (3D-printed samples). These particles had a particle size of less than < 1 µm in diameter.

MENETELMÄ NIKKELIPOHJAISEEN SEOKSEEN PERUSTUVAN JAUHEEN VALMISTAMISEKSI

Patenttivaatimus

1. Menetelmä jauheen valmistamiseksi nikkelipohjaisesta seoksesta, jolle pitoisuudet (paino-%) määritellään seuraavasti:

C	maks. 0,5 %	
S	maks. 0,15 %,	erityisesti maks. 0,03 %
N	maks. 0,25 %	
Cr	14 – 35 %, erityisesti 17 – 28 %	
Ni	jäännös (> 38 %)	
Mn	maks. 4 %	
Si	maks. 1,5 %	
Mo	> 0 – 22 %	
Ti	≤ 4 %, erityisesti ≤ 3,25 %	
Nb	6,0 % saakka	
Cu	3 % saakka, erityisesti 0,5 % saakka	
Fe	≤ 50 %	
P	maks. 0,05 %, erityisesti maks. 0,04 %	
Al	3,15 % saakka, erityisesti 2,5 % saakka	
Mg	maks. 0,015 %	
V	maks. 0,6 %	
Zr	maks. 0,12 %, erityisesti maks. 0,1 %	
W	4,5 % saakka, erityisesti maks. 3 % saakka	
Co	28 % saakka	
B	≤ 0,125 %	
O	> 0,00001 – 0,1 %	

ja valmistukseen liittyvät epäpuhtaudet,

jolloin pätee:

Ni + Fe + Co 56 – 80 %

Nb + Ta ≤ 6,0 %, kun

- seos sulatetaan VIM-uunissa,

- nestemäistä sulatetta pidetään 5 minuutista 2 tuntiin homogenisointia varten,
- suljettu sumutusjärjestelmä syötetyllä kaasulla asetetaan kastepisteeseen -10 °C–120 °C,
- sulate puhalletaan suuttimen kautta kaasuvirtaukseen, jonka kaasun virtausnopeus on 2 nm^3/min–150 nm^3,
- jähmettyneet jauhehiukkaset kerätään kaasutiiviiseen suljettuun säiliöön, jolloin
- hiukkasten hiukkaskoko on 5 µm–250 µm,
- jauheen hiukkaset ovat pallomaisia,
- jauheessa on kaasusulkeumia 0,0–4 % huokospinta-alasta (huokoset > 1 µm) suhteessa arvioitujen objektien kokonaispinta-alaan,
- jauheen irtotiheys on 2 – lejeeringin tiheys n. 8 g/cm^3,
- jauhe pakataan ilmatiiviisti argon-suojakaasuilmaseokseen.

2. Patenttivaatimuksen 1 mukainen menetelmä, jossa on seuraava koostumus (paino-%):

C	maks. 0,25 %
S	maks. 0,03 %
N	> 0 – 0,15 %
Cr	17 – 28 %, erityisesti 17 – 24 %
Ni	jäännös (38 – 72 %)
Mn	maks. 2 %, erityisesti maks. 1 %
Si	maks. 1,2 %, erityisesti ≤ 0,5 %
Mo	> 0 – 21 %, erityisesti 2,5 – 21 %
Ti	> 0 – < 3,25 %
Nb	> 0 – 5,5 %
Cu	maks. 3 %, erityisesti 0,5 % saakka
Fe	> 0 – 38 %
P	maks. 0,04 %
Al	> 0 – 0,8 %
Mg	maks. 0,015 %
V	maks. 0,4 %
Zr	maks. 0,1 %

W 0,5 % saakka

Co ≤ 10 %

B ≤ 0,01 %

Pb maks. 0,001 %, erityisesti maks. 0,0005 %

Se maks. 0,0005 %, erityisesti maks. 0,0003 %

Bi maks. 0,00005 %, erityisesti maks. 0,00003 %

O > 0,00001 – 0,1 %

Nb + Ta > 0 – 5,5 %

ja valmistukseen liittyvät epäpuhtaudet,

jolloin pätee:

Ni + Fe + Co 57 – 77 %.

3. Patenttivaatimuksen 1 tai 2 mukainen menetelmä, jossa on seuraava koostumus (paino-%):

C > 0 – 0,1 %, erityisesti > 0 – 0,08 %

S maks. 0,015 %

N > 0 – 0,03 %

Cr 17 – 24 %

Ni jäännös (> 50 – 63 %)

Mn maks. 1,0 %, erityisesti maks. 0,5 %

Si maks. 0,5 %

Mo 2,8 – 16,5 %

Ti > 0 – 1,15 %

Nb > 0 – ≤ 5,5 %

Cu maks. 0,5 %

Fe > 0 – 25 %

P maks. 0,015 %

Al 0,1 – 0,6 %

Mg maks. 0,015 %

V maks. 0,2 %

Zr maks. 0,1 %

W 0,2 % saakka, erityisesti > 0 – 0,2 %

Co ≤ 2,5 %, erityisesti ≤ 1,0 %

B ≤ 0,01 %

Pb maks. 0,001 %, erityisesti maks. 0,0005 %

Se maks. 0,0005 %, erityisesti maks. 0,003 %

Bi maks. 0,0005 %, erityisesti maks. 0,0003 %

O > 0,00001 – 0,1 %

Nb + Ta > 0 – 5,5 %

Al + Ti < 1,2 %

ja valmistukseen liittyvät epäpuhtaudet,

jolloin pätee:

Ni + Fe + Co 59 – 72 %.

4. Jonkin patenttivaatimuksen 1–3 mukainen menetelmä, jolle metalliseosta käytetään aluksi pääseoksena määritellyllä kemiallisella analyysillä sulattamalla VIM-uunissa, VIM/ESU, VIM/ESU/VAR, VIM/VAR VOD tai VLF, uudelleensulatus ESU:ssa ja/tai VAR:ssa tarvittaessa,

- perusseoslohko erotetaan pieniksi paloiksi sahaamalla,
- perusseoksen palat sulatetaan VIM-uunissa.

5. Jonkin patenttivaatimuksen 1–4 mukainen menetelmä, joka on tunnettu siitä, että syötettävänä kaasuna käytetään inerttiä kaasua.

6. Jonkin patenttivaatimuksen 1–5 mukainen menetelmä, joka on tunnettu siitä, että syötettävänä kaasuna käytetään argonia.

7. Jonkin patenttivaatimuksen 1–6 mukainen menetelmä, tunnettu siitä, että kaasuvirtaus, johon sumutetaan, koostuu argonista.

8. Jonkin patenttivaatimuksen 1–7 mukainen menetelmä, tunnettu siitä, että kaasuvirtaus, johon sumutetaan, koostuu typestä.

9. Jonkin patenttivaatimuksen 1–8 mukainen menetelmä, tunnettu siitä, että kaasuvirtaus, johon sumutetaan, koostuu typen ja argonin seoksesta.

10. Jonkin patenttivaatimuksen 1–9 mukainen menetelmä, joka on tunnettu siitä, että sulate sumutetaan nopeudella 0,5–80 kg/min.

11. Jonkin patenttivaatimuksen 1–10 mukaisella menetelmällä valmistetun jauheen käyttö komponenttien tai rakenneosien lisäainevalmistukseen.

12. Jonkin patenttivaatimuksen 1–10 mukaisella menetelmällä valmistetun jauheen käyttö kerrosten lisäainevalmistukseen komponenttien tai rakenneosien päälle.

13. Jonkin patenttivaatimuksen 1–10 mukaisella menetelmällä valmistetun jauheen käyttö turbiinien komponenttien valmistukseen.

14. Jonkin patenttivaatimuksen 1–10 mukaisella menetelmällä valmistetun jauheen käyttö komponenttien valmistukseen öljy- ja kaasu- sekä kemian prosessiteollisuutta varten.

15. Jonkin patenttivaatimuksen 1–10 mukaisella menetelmällä valmistetun jauheen käyttö venttiilien tai laippojen valmistukseen.