

(19) AUSTRALIAN PATENT OFFICE

(54) Title
Composition and process for warm compaction of stainless steel powders

(51)⁶ International Patent Classification(s)
B22F 1/00 (2006.01) 141/06
C10M 141/06 20060101ALI2006040
(2006.01) 8BMEP **C10M**
C10M 161/00 161/00
(2006.01) 20060101ALI2006040
C22C 33/02 (2006.01) 8BMEP **C22C**
B22F 1/00 33/02
20060101AFI2006010 20060101ALI2006040
1BHAU **C10M** 8BMEP
PCT/SE03/01001

(21) Application No: 2003239021 (22) Application Date: 2003 .06 .13

(87) WIPO No: W003/106077

(30) Priority Data

(31) Number	(32) Date	(33) Country
0201825-7	2002 .06 .14	SE

(43) Publication Date : 2003 .12 .31
(43) Publication Journal Date : 2004 .02 .26

(71) Applicant(s)
Hoganas AB

(72) Inventor(s)
Dahlberg, Mikael, Bergkvist, Anders

(74) Agent/Attorney
Phillips Ormonde & Fitzpatrick, Level 22 367 Collins Street, Melbourne, VIC, 3000

(56) Related Art
GB 779949
WO 00/16934
US 6123748

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
24 December 2003 (24.12.2003)

PCT

(10) International Publication Number
WO 03/106077 A1

- (51) International Patent Classification⁷: **B22F 1/00**,
C22C 33/02
- (21) International Application Number: PCT/SE03/01001
- (22) International Filing Date: 13 June 2003 (13.06.2003)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
0201825-7 14 June 2002 (14.06.2002) SE
- (71) Applicant (for all designated States except US): **HÖ-
GANÄS AB** [SE/SE]; S-263 83 Höganäs (SE).
- (72) Inventors; and
(75) Inventors/Applicants (for US only): **BERGKVIST,
Anders** [SE/NO]; Seilduksgata 22, N-0553 Oslo (NO).
DAHLBERG, Mikael [SE/SE]; Krappervägen 41,
S-260 41 Nyhamnsläge (SE).
- (74) Agent: **AWAPATENT AB**; Box 5117, S-200 71 Malmö
(SE).
- (81) Designated States (national): AE, AG, AL, AM, AT, AU,
AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU,
CZ, DE (utility model), DE, DK (utility model), DK, DM,
DZ, EC, EE (utility model), EE, ES, FI (utility model), FI,
GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KP,
KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK,
MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU,
SC, SD, SE, SG, SK, SL,
- (84) Designated States (regional): ARIPO patent (GH, GM,
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW),
Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),
European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE,
ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO,
SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM,
GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Declarations under Rule 4.17:**
as to applicant's entitlement to apply for and be granted
a patent (Rule 4.17(ii)) for the following designations AE,
AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH,
CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI,
GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG,
KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK,
MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU,
SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG,
UZ, VC, VN, YU, ZA, ZM, ZW, ARIPO patent (GH, GM,
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian
patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European
patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR,
GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR),
OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,
ML, MR, NE, SN, TD, TG)
of inventorship (Rule 4.17(iv)) for US only
- Published:**
with international search report
- For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.

WO 03/106077 A1

(54) Title: COMPOSITION AND PROCESS FOR WARM COMPACTION OF STAINLESS STEEL POWDERS

(57) Abstract: The invention concerns a composition for warm compaction comprising a water-atomised standard stainless steel powder including, in addition to iron and 10-30 % by weight of chromium, optional alloying elements and inevitable impurities, 0.8 % -2.0 % by weight of a warm compaction lubricant. The invention also concerns a process for the preparation of sintered bodies from this composition.

COMPOSITION AND PROCESS FOR WARM COMPACTION OF STAINLESS STEEL
POWDERS

Field of the Invention

5 The present invention concerns steel powder compositions as well as the compacted and sintered bodies obtained thereof. Specifically the invention concerns stainless steel powder compositions for warm compaction.

10 Background to the Invention

The discussion of the background to the invention herein is included to explain the context of the invention. This is not to be taken as an admission that any of the material referred to was published, known or part of the common general knowledge as at the
15 priority date of any of the claims.

Since the start of the industrial use of powder metallurgical processes i.e. the pressing and sintering of metal powders, great efforts have been made in order to enhance the mechanical properties of P/M-components and to improve the tolerances of the
20 finished parts in order to expand the market and achieve the lowest total cost.

Recently much attention has been paid to warm compaction as a promising way of improving the properties of P/M components. The warm compaction process gives the opportunity to increase the
25 density level, i.e. decrease the porosity level in finished parts. The warm compaction process is applicable to most powder/material systems. Normally the warm compaction process leads to higher strength and better dimensional tolerances. A possibility of green machining, i.e. machining in the "as-pressed" state, is also
30 obtained by this process.

Warm compaction is considered to be defined as compaction of a particulate material mostly consisting of metal powder above approximately 100°C up to approximately 150°C according to the currently available powder technologies such as Densmix™,
35 Ancorbond™ or Flow-Met™.

A detailed description of the warm compaction process is described in e.g. a paper presented at PM TEC 96 World Congress, Washington, June 1996, which is hereby incorporated by reference. Specific types of lubricants

X:\E:\n\spec\w723902.doc

used for warm compaction of iron powders are disclosed in e.g. the US patents 5 154 881 (Rutz) and 5 744 433 (Storström).

Until recently it has been observed that the general
5 advantages with warm compaction have been insignificant as only minor differences in e.g. density and green strength have been demonstrated in the case of stainless steel powders. Major problems encountered when warm compacting stainless steel powders are the high ejection
10 forces and the high internal friction during compaction.

However, as disclosed in the US patent 6 365 095 (Bergkvist), it was recently found that stainless steel powders may be subjected to warm compaction with good results provided that the stainless steel powder is
15 distinguished by very low oxygen, carbon and silicon levels. The widely used standard qualities having higher levels of these elements could however not be successfully warm compacted i.e. the properties of the warm compacts were not significantly better than the
20 green density of a corresponding body compacted at ambient temperature.

It has now unexpectedly been found that also standard stainless steel powders can be compacted at elevated temperatures with good results. In comparison
25 with the stainless steel powders disclosed in the above US patent the standard stainless powders are generally characterised in a higher amount of oxygen, carbon and silicon. These powders are also easier to produce and accordingly cheaper. According to the present invention
30 it has thus, contrary to the teaching in the US patent, been found that these standard powders can be compacted to high green densities without the use of excessively high compaction pressures. The high green density is valuable when the product is subsequently sintered as it
35 is not necessary to use high sintering temperatures and accompanying high energy consumption in order to get a high sintered density which is normally necessary in

order to get good mechanical properties. Additionally high sintering temperatures induce strains in the material which in turn gives poor dimensional stability.

5 Summary of the Invention

According to the present invention, there is provided a composition for warm compaction of a water atomised stainless steel powder including iron, 10-30% by weight of chromium, at least 0.5% by weight silicon, optional alloying elements and inevitable impurities, and a high temperature lubricant, wherein the steel powder is a standard steel powder and the lubricant is present in an amount of 0.8%-2.0% by weight.

The present invention also provides a process of preparing high density, warm compacted and sintered bodies of a water atomised, standard stainless steel powder including iron and 10-30% by weight of chromium, at least 0.5% by weight silicon optional alloying elements and inevitable impurities said process including the steps of

- providing a mixture of a pre-alloyed, water-atomised, stainless steel powder having a Cr content of 10-30% by weight, a Si content of at least 0.5% by weight, optional alloying elements and inevitable impurities;
- mixing the powder with 0.8%-2.0% by weight of a high temperature lubricant
- compacting the mixture at a temperature; and
- sintering the compacted body.

In brief the process of preparing high density, warm compacted bodies of a water atomised standard stainless steel powder according to the present invention is based on the discovery that specific amounts of lubricants have to be used in the stainless steel powder composition which is subjected to the compaction at elevated temperature. Minor amounts of selected additives included in the composition contribute to the unexpected finding that standard stainless steels can be successfully compacted.

2003239021 04 Aug 2006

3a

Detailed Description of the Invention

Type of Powder

- 5 Preferably the powders subjected to warm compaction are pre-alloyed, water atomised powders which include, by percent of weight, 10-30% of chromium. These powders are stainless steel powders of standard type and include at least 0.5% by weight of silicon. Normally the silicon content is between 0.7 and 1.0% by weight of the steel powder. The stainless steel powder may also
- 10 include other elements such as, molybdenum, nickel, manganese, niobium, titanium, vanadium. The amounts of these elements may be 0-5% of molybdenum, 0-22% of nickel, 0-1.5% of manganese, 0-2% of niobium, 0-2% of titanium, 0-2% of vanadium, and at most 1% of inevitable impurities and most preferably 10-20% of chromium, 0-3% of molybdenum, 0.1-0.4% of manganese, 0-0.05% of niobium, 0-0.5% of
- 15 titanium, 0-0.5% of vanadium and essentially no nickel or alternatively 5-15% of nickel, the balance being iron and unavoidable impurities (normally less than 1% by weight). Furthermore, the average particle size of the steel

powder should preferably be above about 30 μm and a suitable interval is between 30 and 70 μm .

Examples of stainless steel powders which are suitably used according to the present invention are 316 L, 409 Nb, 409 L, 410 L, 434 L. The standard steel powders used according to the present invention generally include more than 0.5% by weight of Si and normally the Si content is 0.7-1.0% by weight. This feature distinguishes standard stainless powders from the stainless powders used for the warm compaction according to the US patent 6 365 095 (Bergkvist) mentioned above.

Amount of lubricant

The amount of lubricant in the composition to be compacted is an important factor for the possibility to get a satisfactory result. It has thus been found that the total amount of lubricant should be above 0.8% by weight, preferably at least 1.0% by weight and most preferably at least 1.2% by weight of the total powder composition. As increasing amounts of lubricant decrease the final green density due to the fact that the lubricants normally have much lower density than the metal powder, lubricant amounts above 2.0% by weight are less important. In practice it is believed that the upper limit should be less than 1.8% by weight. A minor amount, such as at least 0.05 and at most 0.4% by weight of the lubricant should preferably be a compound having high oxygen affinity, which promotes the sintering activity.

Type of lubricant

The lubricant may be of any type as long as it is compatible with the warm compaction process. Examples of such lubricants are disclosed in e.g. the US patents 5 154 881 (Rutz) and 5 744 433 (Storström), which are referred to above and which are hereby incorporated by reference. The lubricants may also be e.g. metal stearates, such as lithium stearate, zinc stearate;

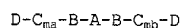
04 Aug 2006

2003239021

5

paraffins; waxes ; natural and synthetic fat derivatives and polyamides. Preliminary results have also shown that lubricants conventionally used for cold compaction, such as EBS, may be used for warm compaction of the standard steel powders according to the present invention although the flow properties of such powder compositions are inferior.

So far however the most promising results have been obtained by using a type of lubricants disclosed in the copending patent application SE02/00762 PCT. These type of lubricants include an amide component which can be represented by the following formula



wherein

D is -H, COR, CNHR, wherein R is a straight or branched aliphatic or aromatic group including 2-21 C atoms

C is the group -NH (CH₂)_n CO-

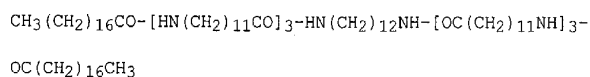
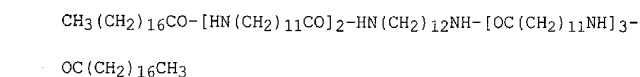
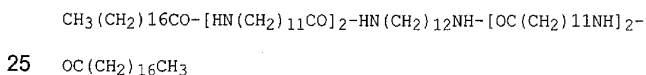
B is amino or carbonyl

A is alkylen having 4-16 C atoms optionally including up to 4 O atoms

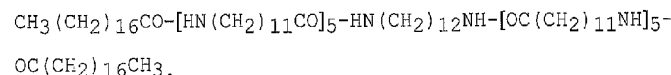
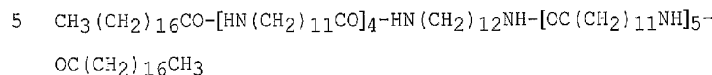
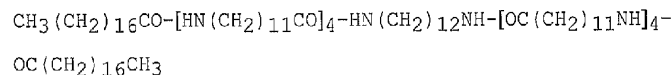
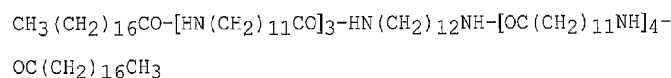
ma and mb which may be the same or different is an integer 1-10

n is an integer 5-11.

Examples of preferred such amides are:



6



As previously mentioned the lubricant should
 10 preferably also include a compound having high affinity
 for oxygen. Examples of such high affinity compounds are
 alkali metal stearates. Other examples are stearates of
 alkaline earth metals. The presently most preferred
 compound being lithium stearate.

15

Selected additives

According to a preferred embodiment of the invention
 minor amounts of selected additives may be included in
 the composition before the powder composition is
 20 subjected to warm compaction. These additives include
 fatty acids and flow enhancing agents.

The fatty acid may be selected from the group
 consisting of stearic acid and oleic acid. The amounts of
 the fatty acid in the composition according to the
 25 invention may vary between 0.005 and 0.5, preferably
 between 0.010 and 0.16 and most preferably between 0.015
 and 0.10% of the lubricant composition. The fatty acid
 has an beneficial effect on the apparent density.

The flow agent may be a material of the type
 30 described in the US patent 5 782 954 (Luk). This material
 is comprised of nanoparticles of various metals and their
 oxides such as silicon oxide. Typically, the metal and
 metal oxide powders have average particle sizes below
 about 500 nanometers. The silicon oxide flow agents are

preferably blended with the iron-based powders in an amount of from about 0.005 to about 2 percent by weight of the resultant powder composition. The preferred silicon oxide flow agents are powders or particles of
5 silicon dioxide having an average particle size below about 40 nanometers. An example of a suitable flow agent is Aerosil.

Warm compaction

10 The stainless steel powder including the lubricant and optional additives is subsequently compacted at an elevated temperature. The warm compaction may be performed with a preheated powder, a preheated die or both. The powder could e.g. be preheated to a temperature
15 above 60°C preferably above 90°C. A suitable interval for the warm compaction is between 100°C and 200°C, and preferably the compaction could be performed at a temperature less than about 150°C. The compaction is performed in standard compaction equipment with
20 compaction pressures preferably between about 400 and 2000 MPa, preferably between about 500 and 1000 MPa.

The powder mixes used for the warm compaction can be prepared mainly in two ways. An alternative is to prepare the powder mix by carefully blending the steel powder,
25 the lubricant(s) in the form of solid particles and a flow agent to a homogenous mix. An other alternative is to make the lubricants stick (adhere) to the stainless steel powder particles. This can be done by heating a mixture including the steel powder and the lubricant(s)
30 to a temperature above the melting point of the lubricant(s), mixing the heated mixture and cooling the obtained mixture before the flow agent is added. It can also be done by dissolving the lubricant(s) in a solvent, mixing the obtained solution with the steel powder,
35 evaporating the solvent in order to obtain a dry mixture to which the flow agent is subsequently added.

Sintering

- The obtained green bodies are then sintered in the same way as the standard materials, i.e. at temperatures between 1100°C and 1400° C, the most pronounced advantages being obtained when the sintering is performed between 1250 and 1325°C. A lower sintering temperature may be used in order to reach a given sintered density by using warm compaction instead of compaction at ambient temperature. Furthermore the sintering is preferably carried out in standard non oxidative atmosphere for periods between 15 and 90, preferably between 20 and 60 minutes. The high densities according to the invention are obtained without the need of recompacting, resintering and/or sintering in vacuum or reduced atmosphere.
- The invention is illustrated by the following non limiting examples.

Examples

Example 1

- This experiment was carried out with a standard materials 434 LHC, 409 Nb, 316 LHD och 410 LHC which are all available from Höganäs, Belgium and have the compositions indicated in table 1.

Table 1

	%Cr	%Ni	%Mo	%Si	%Mn	%Nb	%C	%O	%Fe
434 L	16.9	0.1	1.0	0.76	0.16	0	0.016	0.22	Bal
409 Nb	11.3	0.1	0	1.0	0.1	0.5	0.01	0.15	Bal
316 L	16.9	12.8	2.3	0.8	0.1	0	0.02	0.36	Bal
410 L	11.8	0.2	0	0.8	0.1	0	<0.01	0.24	Bal

- Compaction was made on samples of 50 g of these stainless steel powders at 600 and 800 MPa. The warm compaction was performed with a powder temperature and a die temperature of 110°C. The amounts of lubricants are disclosed in the following table 2, wherein CC (cold compaction which is the conventional type of compaction)

indicates that the compaction was performed at room temperature (ambient temperature) and WC indicates warm compaction.

5 Table 2

Sample	Powder	Amount of lubricant	Lubricant composition	Type of compaction
434 _{ca}	434 L	0.6*	a	CC
434 _{wb}	434 L	0.6*	b	WC
409 _{cc}	409 Nb	1.2	c	CC
409 _{wd}	409 Nb	1.2	d	WC
316 _{wd}	316 L	1.2	d	WC
410 _{wd}	410 L	1.2	d	WC
410 _{wb}	410 L	1.1	b	WC
410 _{wc}	410 L	1.1	c	WC
410 _{cc}	410 L	1.1	c	CC

*not within the scope of the invention

The following lubricants and lubricant compositions were used in the different samples:

- 10 a Ethylene bisstearamide (EBS)
 b Advawax
 c EBS +0.3% Li stearate
 d 1.0% amide oligomer (according to the patent
 publication WO 02083345) + 0.2% Li stearate,
 15 0.05% stearic acid, 0.1% Aerosil

The different compositions were prepared as follows:

- Compositions including EBS and EBS + Li stearate,
 respectively, were admixed before the compaction
 20 operation. The compositions including Advawax were
 prepared according to the method disclosed in the US
 patent 5 429 792 and the compositions including the amide
 oligomer were prepared according to the method disclosed
 in the patent publication WO 02083346.

10

The following Table 3 discloses the green densities obtained when the samples were compacted at 600 MPa and 800 MPa, respectively.

5 Table 3

Sample	Green density (g/cm ³) at 600 MPa	Green density (g/cm ³) at 800 MPa
434 _{ca}	6.38	6.62
434 _{wb}	6.43*	6.67*
409 _{cc}	6.45	6.68
409 _{wd}	6.68	6.96
316 _{wd}	6.73	7.02
410 _{wd}	6.83	7.00
410 _{wb}	6.78	7.00
410 _{wc}	6.76**	6.99**
410 _{cc}	6.61	6.82

* problems during compaction, no sintering possible.

** somewhat reduced flow

10 The green parts were sintered at 1160°C in hydrogen atmosphere for 45 min, after which the sintered density was measured (Table 4).

Table 4

Sample	Sintered density (g/cm ³) at 600 MPa	Sintered density (g/cm ³) at 800 MPa
409 _{cc}	6.52	6.77
409 _{wd}	6.74	7.01
316 _{wd}	6.90	7.19
410 _{wd}	6.88	7.05

15

The results disclosed in table 5 were obtained when the sintering was performed at 1250°C.

11

Table 5

Sample	Sintered density (g/cm ³) at 600 MPa	Sintered density (g/cm ³) at 800 MPa
409 _{cc}	7.09	7.21
409 _{wd}	7.22	7.38
316 _{wd}	7.09	7.33
410 _{wd}	7.22	7.34
410 _{wb}	7.15	7.31

The following table 6 discloses the tensile properties after sintering at 1250°C.

5

Table 6

	Ultimate tensile strength MPa	Ultimate tensile strength MPa	Elongation (%)	Elongation (%)
Sample	600 MPa	800 MPa	600 MPa	800 MPa
409 _{cc}	358	374	17.0	15.9
409 _{wd}	372	408	16.6	18.0
316 _{wd}	418	465	26.1	30.0
410 _{wb}	361	384	16.5	15.9

The following table 7 discloses the impact energy after sintering at 1250°C.

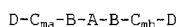
10

Table 7

	Impact energy (J)	Impact energy (J)
Sample	600 MPa	800 MPa
409 _{cc}	135	161
409 _{wd}	190	264
316 _{wd}	125	172
410 _{wb}	169	191

Claims

1. A composition for warm compaction of a water atomised stainless steel powder including iron, 10-30% by weight of chromium, at least 0.5% by weight silicon, optional alloying elements and inevitable impurities, and a high temperature lubricant, wherein the steel powder is a standard steel powder and the lubricant is present in an amount of 0.8%-2.0% by weight.
2. Composition according to claim 1 wherein the steel powder includes 0.7-1.0% by weight of silicon.
3. Composition according to any one of the preceding claims wherein the steel powder includes one or more elements selected from the group consisting of molybdenum, nickel, manganese, niobium, titanium, vanadium and at most 1.0% by weight of inevitable impurities.
4. Composition according to any one of the preceding claims wherein the lubricant is a warm compaction lubricant.
5. Composition according to any one of the claims 1-4 wherein the lubricant is combined with up to 0.4% by weight of a high oxygen affinity compound.
6. Composition according to claim 5 wherein the lubricant includes between about 0.05 and 0.3% by weight of a high oxygen affinity compound.
7. Composition according to claim 5 or 6 wherein the high oxygen affinity compound is lithium stearate.
8. Composition according to any one of the preceding claims, wherein the lubricant in addition to the optional high oxygen affinity compound essentially consists of an amide oligomer lubricant having the formula



wherein

D is -H, COR, CNHR, wherein R is a straight or branched aliphatic or aromatic group including 2-21 C atoms

5 C is the group -NH (CH₂)_n CO-

B is amino or carbonyl

A is alkylene having 4-16 C atoms optionally including up to 4 O atoms

ma and mb which may be the same or different is an integer 1-10

10 n is an integer 5-11.

9. Composition according to any one of the preceding claims also including an amount of an additive selected from the group consisting of fatty acid and flow agent.

15

10. Composition according to claim 9, wherein the fatty acid is selected from the group consisting of stearic acid and oleic acid.

11. Composition according to claim 10, wherein the amount of fatty acid is between 0.005 and 0.5% by weight of the composition.

20

12. Composition according to claim 9 including as the flow agent silicon oxide in an amount between 0.005 and 2% by weight of the composition.

25

13. Composition for warm compaction according to any one of the claims 1-3, 5-7 and 9-11 including a water-atomised, standard stainless steel powder including, in addition to iron, 10-30% of chromium, wherein the lubricant is a wax, such as ethylene bisstearamide.

30

14. A process of preparing high density, warm compacted and sintered bodies of a water atomised, standard stainless steel powder including iron, 10-30% by weight of chromium, at least 0.5% by weight silicon, optional alloying elements and inevitable impurities said process including the steps of

35

- 5 - providing a mixture of a pre-alloyed, water-atomised, stainless steel powder having a Cr content of 10-30% by weight, a Si content of at least 0.5% by weight, optional alloying elements and inevitable impurities;
- mixing the powder with 0.8%-2.0% by weight of a high temperature lubricant
- compacting the mixture at a temperature; and
- sintering the compacted body.
- 10 15. Process according to claim 14, wherein the warm compaction is performed at a temperature of at least 60°C.
- 16. Process according to claim 15, wherein the warm compaction is performed at a temperature of at least 90°C.
- 17. Process according to any one of the claims 14-16 wherein the sintering is performed in a non oxidative atmosphere without previous sintering in reduced atmosphere.
- 20 18. Process according to any one of the claims 14-17 wherein the sintering is performed at a temperature between 1100°C and 1400°C.
- 19. Process according to any one of the claims 14-17 wherein the sintering is performed at a temperature between 1250°C and 1325°C.
- 25 20. A composition according to claim 1, substantially as herein described with reference to any one of the Examples.
- 30 21. A process according to claim 14, substantially as herein described with reference to any one of the Examples.