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(54) **METHOD OF PREPARING IRON-BASED COMPONENTS BY COMPACTION WITH ELEVATED PRESSURES**

VERFAHREN ZUR HERSTELLUNG VON KOMPONENTEN AUF EISENBASIS DURCH  
VERDICHTUNG MIT ERHÖHTEN DRÜCKEN

PROCEDE DE PREPARATION DE COMPOSANTS A BASE DE FER PAR COMPACTAGE A DES  
PRESSIONS ELEVEES

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(56) References cited:  
**EP-A1- 0 554 009 GB-A- 2 315 115**  
**US-A- 3 901 661 US-A- 4 190 441**  
**US-A- 5 892 164**

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## Description

### FIELD OF THE INVENTION

**[0001]** The present invention concerns a method for the preparation of components having high density by using metal powder compositions useful within the powder metallurgical industry. The invention is defined in claims 1-10.

**[0002]** There are several advantages by using powder metallurgical methods for producing structural parts compared with conventional machining processes of full dense steel. Thus, the energy consumption is much lower and the material utilisation is much higher. Another important factor in favour of the powder metallurgical route is that components with net shape or near net shape can be produced directly after the sintering process without costly shaping processes such as turning, milling, boring or grinding. However, normally a full dense steel material has superior mechanical properties compared with PM components. This is mainly due to the occurrence of porosity in the PM components. Therefore, the strive has been to increase the density of PM components in order to reach values as close as possible to the density value of a full dense steel.

**[0003]** Among the methods used in order to reach higher density of PM components the powder forging process has the advantage that full dense components may be obtained. The process is however costly and is utilised mainly for mass production of heavier components, such as connection rods. Full dense materials can also be obtained by elevated pressures at high temperatures, such as in hot isostatic pressing, HIP, but also this method is costly.

**[0004]** By using warm compaction, a process where the compaction is performed at an elevated temperature, typically at 120 to 250 °C, the density can be increased with about 0,2 g/cm<sup>3</sup>, which results in a considerable improvement of the mechanical properties. A disadvantage is however that the warm compaction method involves additional investment and processing. Other processes, such as double pressing, double sintering, sintering at elevated temperatures etc, may further increase the density. Also these methods will add further production costs hence reducing the overall cost effectiveness.

**[0005]** In order to expand the market for powder metallurgical components and utilise the advantages with the powder metallurgical technique there is thus a need for a simple, less expensive method of achieving high density compacts with improved static and dynamic mechanical strength.

### SUMMARY OF THE INVENTION

**[0006]** It has now been found that high density components can be obtained by using high compaction pressures in combination with coarse powders. In view of the general knowledge, that conventionally used powders,

i.e. powders including fine particles, cannot be compacted to high densities without problems with e.g. damaged or deteriorated surfaces of the compacts this finding is quite unexpected. Specifically, the method according to the present invention includes the steps of providing an iron-based powder essentially free from fine particles; optionally mixing said powder with graphite and other additives; uniaxially compacting the powder in a die at high pressure and ejecting the green body, which may subsequently be sintered.

### DETAILED DESCRIPTION OF THE INVENTION

**[0007]** The term "high density" is intended to mean compacts having a density of about at least 7.3 g/cm<sup>3</sup>. Components having lower densities can of course also be produced but are believed to be of less interest.

**[0008]** The iron-based powder according to the present invention includes pure iron powder such as atomised iron powder, sponge iron powder, reduced iron powder; partially diffusion-alloyed steel powder; and completely alloyed steel powder. The partially diffusion-alloyed steel powder is preferably a steel powder alloyed partially with one or more of Cu, Ni, and Mo. The completely alloyed steel powder is preferably a steel powder alloyed with Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S and B. Also stainless steel powders are of interest.

**[0009]** As regards the particle shape it is preferred that the particles have an irregular form as is obtained by water atomisation. Also sponge iron powders having irregularly shaped particles may be of interest.

**[0010]** A critical feature of the invention is that the powder used have coarse particles i.e. the powder is essentially without fine particles. The term "essentially without fine particles" is intended to mean that less than about 5 % of the powder particles have a size below 45 µm as measured by the method described in SS-EN 24 497. So far the most interesting results have been achieved with powders essentially consisting of particles above about 106 µm and particularly above about 212 µm. The term "essentially consists" is intended to mean that at least 50 %, preferably at least 60 %, and most preferably at least 70 % of the particles have a particle size above 106 and 212 µm, respectively. The maximum particle size may be about 2 mm. The particle size distribution for iron-based powders used at PM manufacturing is normally distributed with a gaussian distribution with a average particle diameter in the region of 30 to 100 µm and about 10-30 % less than 45 µm. Iron based powders essentially free from fine particles may be obtained by removing the finer fractions of the powder or by manufacturing a powder having the desired particle size distribution.

**[0011]** The influence of particle size distribution and the influence of particle shape on the compaction properties and properties of the compacted body have been subjected to intense studies. Thus the US patent 5,594,186 reveals a method of producing PM components with a density higher than 95 % of theoretical den-

sity by utilising substantially linear, acicular metal particles having a triangular cross section. Such particles are suitably produced by a machining or milling process.

**[0012]** Powders having coarse particles are also used for the manufacture of soft magnetic components. Thus the US patent 6 309 748 discloses a ferromagnetic powder, the particles of which have a diameter size between 40 and 600  $\mu\text{m}$ . In contrast to iron based powder particles according to the present invention, these powder particles are provided with a coating.

**[0013]** In the US patent 4,190,441 a powder composition for production of sintered soft magnetic components is disclosed. In this patent the iron powder includes particles with less than 5 % exceeding 417  $\mu\text{m}$ , and less than about 20 % of the powder particles have a size less than 147  $\mu\text{m}$ . This patent teaches that, because of the very low content of particles less than 147  $\mu\text{m}$ , the mechanical properties of components manufactured from this coarse, highly pure powder are very low. Furthermore the patent teaches that if higher strength is desired, it is not possible to increase the content of particles having a size less than 147  $\mu\text{m}$  without simultaneously deteriorating the soft magnetic properties. Therefore this powder is mixed with specific amounts of ferrophosphorus. Graphite which may be used in the compositions according to the present invention is not mentioned in this patent and besides the presence of graphite would deteriorate the magnetic properties.

**[0014]** Powder mixtures including coarse particles are also disclosed in the US patent 5225459 (EP 554 009) which also concerns powder mixtures for the preparation of soft magnetic components. Nor do these powder mixtures include graphite.

**[0015]** Within the field of powder forging it is furthermore known that pre-alloyed iron-based powders with coarse particles can be used. The US patent 3 901 661 discloses such powders. This patent discloses that a lubricant may be included and specifically that the amount of lubricant should be 1 % by weight (example 1). If the powders according to the present invention were mixed with such a high amount of lubricant it would however not be possible to achieve the high densities.

**[0016]** In order to obtain compacts having satisfactory mechanical sintered properties of the sintered part according to the present invention it is necessary to add certain amounts of graphite to the powder mixture to be compacted. Thus graphite in amounts between 0.1-1, preferably 0.2 -1.0 and most preferably 0.2-0.8 % by weight of the total mixture to be compacted could be added before the compaction.

**[0017]** Other additives may be added to the iron-based powder before compaction, such as alloying elements comprising Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S, and B. These alloying elements may be added in amounts up to 10 % by weight. Further additives are machinability enhancing compounds, hard phase material and flow agents.

**[0018]** The iron-base powder may also be combined

with a lubricant before it is transferred to the die (internal lubrication). The lubricant is added to minimize friction between the metal power particles and between the particles and the die during a compaction, or pressing, step. Examples of suitable lubricants are e.g. stearates, waxes, fatty acids and derivatives thereof, oligomers, polymers and other organic substances with lubricating effect. The lubricants are preferably added in the form of particles but may also be bonded and/or coated to the particles. According to the present invention the amount of lubricant added to the iron-based powder may vary between 0.05 and 0.6 %, preferably between 0.1-0.5 % by weight of the mixture.

**[0019]** The method according to the invention may also be performed with the use of external lubrication (die wall lubrication) where the walls of the die are provided with a lubricant before the compaction is performed. A combination of external and internal lubrication may also be used.

**[0020]** The term "at high compaction pressure" is intended to mean at pressures of about at least 800 MPa. More interesting results are obtained with higher pressures such as pressures above 900, preferably above 1000, more preferably above 1100 MPa.

**[0021]** Conventional compaction at high pressures, i.e. pressures above about 800 MPa with conventionally used powders including finer particles, in admixture with low amounts of lubricants (less than 0.6 % by weight) are generally considered unsuitable due to the high forces required in order to eject the compacts from the die, the accompanying high wear of the die and the fact that the surfaces of the components tend to be less shiny or deteriorated. By using the powders according to the present invention it has unexpectedly been found that the ejection force is reduced at high pressures, about 1000 MPa, and that components having acceptable or even perfect surfaces may be obtained also when die wall lubrication is not used.

**[0022]** The compaction may be performed with standard equipment, which means that the new method may be performed without expensive investments. The compaction is performed uniaxially in a single step at ambient or elevated temperature. Alternatively the compaction may be performed with the aid of a percussion machine (Model HYP 35-4 from Hydropulsor) as described in patent publication WO 02/38315.

**[0023]** The sintering may be performed at temperatures normally used within the PM field, e.g. at standard temperature between 1080 and 1160°C or at higher temperatures above 1160°C and in conventionally used atmospheres.

**[0024]** Other treatments of the green or sintered component may as well be applied, such as machining, case hardening, surface densification or other methods used in PM technology.

**[0025]** In brief the advantages obtained by using the method according to the present invention are that high density green compacts can be cost effectively produced.

The new method also permits production of higher components which are difficult to produce by using the conventional technique. Additionally standard compaction equipment can be used for producing high density compacts having acceptable or even perfect surface finish.

**[0026]** Examples of products which suitably can be manufactured by the new method are connecting rods, gears and other structural parts subjected to high loads. By using stainless steel powders flanges are of special interest.

**[0027]** The invention is further illustrated by the following examples.

#### Example 1

**[0028]** Two different iron-based powder compositions according to the present invention were compared with a standard iron-based powder composition. All three compositions were produced with Astaloy Mo available from Höganäs AB, Sweden. 0.2 % by weight of graphite and 0.4 % by weight of a lubricant (Kenolube™) were added to the compositions. In one of the iron-based powder compositions according to the invention, particles of the Astaloy Mo with a diameter less than 45 µm were removed and in the other composition according to the invention particles of Astaloy Mo less than 212 µm were removed. The compaction was performed at ambient temperature and in standard equipment. As can be seen from figure 1-1 a clear density increase at all compaction pressures is obtained with the powder having a particle size above 212µm.

**[0029]** Figure 1-2 shows that in order to obtain components without deteriorated surfaces the most important factor is the reduction or elimination of the smallest particles, i.e. particles below 45 µm. Furthermore from this figure it can be seen that the force needed for ejection of the compacts produced by the iron based powder composition without particles less than 212µm was considerably reduced compared with the ejection force needed for compacts produced from the standard iron-based powder composition having about 20 % of the particles less than 45µm. The ejection force needed for compacts produced from the iron-based powder composition according to the invention without particles less than 45µm is also reduced in comparison with the standard powder.

**[0030]** A noticeable phenomenon is that the ejection force for compacts produced according to the present invention decreases with the increasing ejection pressure whereas the opposite is valid for the standard composition.

**[0031]** It was also observed that the compacts obtained when the standard powder was compacted at a pressure above 700 MPa had deteriorated surfaces and were accordingly not acceptable. The compacts, which were obtained when the powder essentially without particles less than 45 µm was compacted at a pressure above 700 MPa, had a less shiny surface which at least under certain circumstances is acceptable.

#### Example 2

**[0032]** Example 1 was repeated but as lubricant 0.5 % of EBS (ethylene bisstearamide) was used and the compaction was performed with the aid of a percussion machine (Model HYP 35-4 from Hydropulsor, Sweden)

**[0033]** From figure 2-1 and 2-2, respectively, it can be noticed that higher green densities and lower ejection forces were obtained with the powder composition according to the invention compared with the powder composition with the standard powder. It can also be noticed that components produced from the standard powder had deteriorated surfaces at all compaction pressures.

#### **Claims**

1. Process for the preparation of high density green compacts comprising the following steps:

providing an atomised iron-based powder, optionally comprising Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S and B, wherein less than 5% of the iron-based powder particles have a size below 45 µm;

mixing said powder with 0.1-1.0% by weight graphite and a lubricant in an amount between 0.05 and 0.6% by weight, optionally machinability enhancing agents, hard phase materials and flow agents;

uniaxially compacting the powder in a die at a compaction pressure of at least 800 MPa; and ejecting the green body from the die.

2. Process according to claim 1, wherein the compaction is performed in a single step.
3. Process according to claim 1 or 2, wherein at least 50%, preferably at least 60% and most preferably at least 70% of the iron-based powder consists of particles having a particle size above 106 µm.
4. Process according to any one of the claims 1-3, wherein at least 50%, preferably at least 60% and most preferably at least 70% of the iron-based powder consists of particles having a particle size above 212 µm.
5. Process according to claim 4, wherein the maximum particle size is 2 mm.
6. Process according to any one of claims 1-5, wherein the compaction is performed in a lubricated die.
7. Process according to any one of claims 1-6, wherein the compaction is performed at a pressure of at least 900 MPa, more preferably at least 1000 MPa and most preferably above 1100 MPa.

8. Process according to any one of claims 1-7, wherein the compaction is performed at ambient temperature.
9. Process according to any one of claims 1-8, wherein the compaction is performed at elevated temperature.
10. Process according to any one of claims 1-9 for preparing sintered products, said process further including a single sintering step at a temperature above 1100°C.

#### Patentansprüche

1. Verfahren zur Herstellung von Grünlingen hoher Dichte, welches die folgenden Schritte umfasst:

Bereitstellen eines zerstäubten Pulvers auf Eisenbasis, welches gegebenenfalls Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S und B umfasst, wobei weniger als 5 % der Teilchen des Pulvers auf Eisenbasis eine Größe von weniger als 45 µm aufweisen;  
 Vermischen des Pulvers mit 0,1 bis 1,0 Gewichts-% Graphit und einem Schmiermittel in einer Menge von 0,05 bis 0,6 Gewichts-%, gegebenenfalls Mitteln zur Verbesserung der Verarbeitbarkeit, Hartstoffphasen-Materialien und Flussmitteln;  
 Uniaxiales Verdichten des Pulvers in einer Pressform mit einem Verdichtungsdruck von mindestens 800 MPa und  
 Auswerfen des Grünlings aus der Pressform.

2. Verfahren nach Anspruch 1, wobei die Verdichtung in einem einzigen Schritt durchgeführt wird.
3. Verfahren nach Anspruch 1 oder 2, wobei mindestens 50 %, vorzugsweise mindestens 60 %, insbesondere mindestens 70 %, des Pulvers auf Eisenbasis aus Teilchen bestehen, die eine Teilchengröße von mehr als 106 µm aufweisen.
4. Verfahren nach einem der Ansprüche 1 bis 3, wobei mindestens 50 %, vorzugsweise mindestens 60 %, insbesondere mindestens 70 %, des Pulvers auf Eisenbasis aus Teilchen bestehen, die eine Teilchengröße von mehr als 212 µm aufweisen.
5. Verfahren nach Anspruch 4, wobei die maximale Teilchengröße 2 mm beträgt.
6. Verfahren nach einem der Ansprüche 1 bis 5, wobei die Verdichtung in einer geschmierten Pressform durchgeführt wird.

7. Verfahren nach einem der Ansprüche 1 bis 6, wobei die Verdichtung mit einem Druck von mindestens 900 MPa, vorzugsweise mindestens 1.000 MPa, insbesondere mehr als 1.100 MPa, durchgeführt wird.
8. Verfahren nach einem der Ansprüche 1 bis 7, wobei die Verdichtung bei Umgebungstemperatur durchgeführt wird.
9. Verfahren nach einem der Ansprüche 1 bis 8, wobei die Verdichtung bei erhöhter Temperatur durchgeführt wird.
10. Verfahren nach einem der Ansprüche 1 bis 9 zum Herstellen von gesinterten Produkten, wobei das Verfahren ferner einen einzigen Sinterschritt bei einer Temperatur von mehr als 1.100 °C umfasst.

#### Revendications

1. Procédé de préparation de comprimés crus à haute densité comprenant les étapes suivantes :  
 fournir une poudre pulvérisée à base de fer, comprenant facultativement Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S et B, dans laquelle moins de 5 % des particules de poudre à base de fer ont une taille inférieure à 45 µm ;  
 mélanger ladite poudre avec 0,1 à 1,0 % en masse de graphite et d'un lubrifiant dans une quantité comprise entre 0,05 et 0,6 % en masse, facultativement des agents favorisant l'usinabilité, des matériaux à phase dure et des agents d'écoulement ;  
 compacter de façon uniaxiale la poudre dans une filière à une pression de compactage d'au moins 800 MPa ; et  
 éjecter l'ébauche crue de la filière.
2. Procédé selon la revendication 1, dans lequel le compactage est effectué au cours d'une seule étape.
3. Procédé selon la revendication 1 ou 2, dans lequel au moins 50 %, de préférence au moins 60 % et le plus préférentiellement au moins 70 % de la poudre à base de fer sont constitués de particules ayant une taille des particules supérieure à 106 µm.
4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel au moins 50 %, de préférence au moins 60 % et le plus préférentiellement au moins 70 % de la poudre à base de fer sont constitués de particules ayant une taille des particules supérieure à 212 µm.
5. Procédé selon la revendication 4, dans lequel la taille

maximale des particules est de 2 mm.

6. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel le compactage est effectué dans une filière lubrifiée. 5
7. Procédé selon l'une quelconque des revendications 1 à 6, dans lequel le compactage est effectué à une pression d'au moins 900 MPa, plus préférablement d'au moins 1 000 MPa et le plus préférablement d'au moins 1 100 MPa. 10
8. Procédé selon l'une quelconque des revendications 1 à 7, dans lequel le compactage est effectué à température ambiante. 15
9. Procédé selon l'une quelconque des revendications 1 à 8, dans lequel le compactage est effectué à température élevée. 20
10. Procédé selon l'une quelconque des revendications 1 à 9, pour préparer des produits frittés, ledit procédé incluant également une seule étape de frittage à une température supérieure à 1 100 °C. 25

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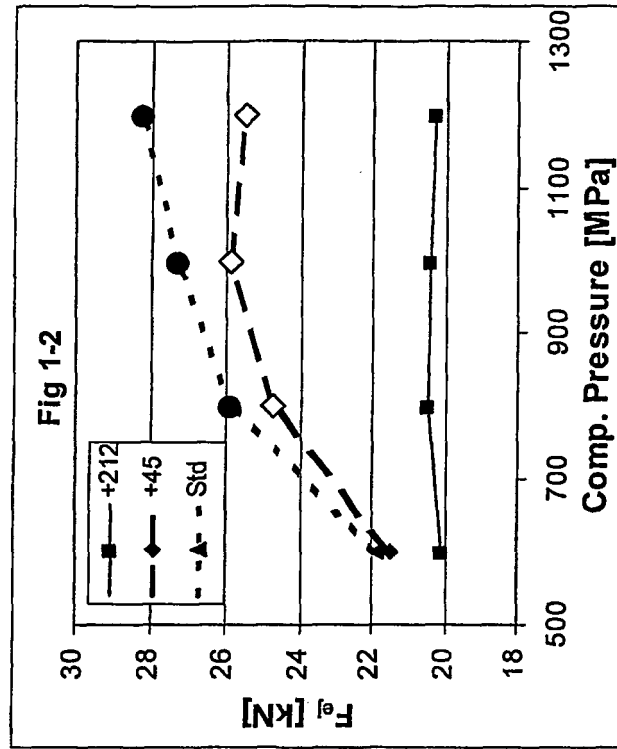
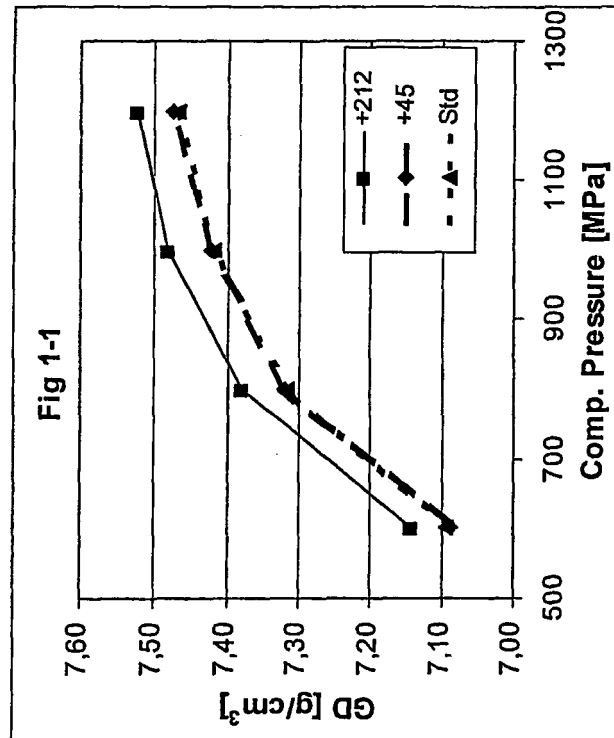
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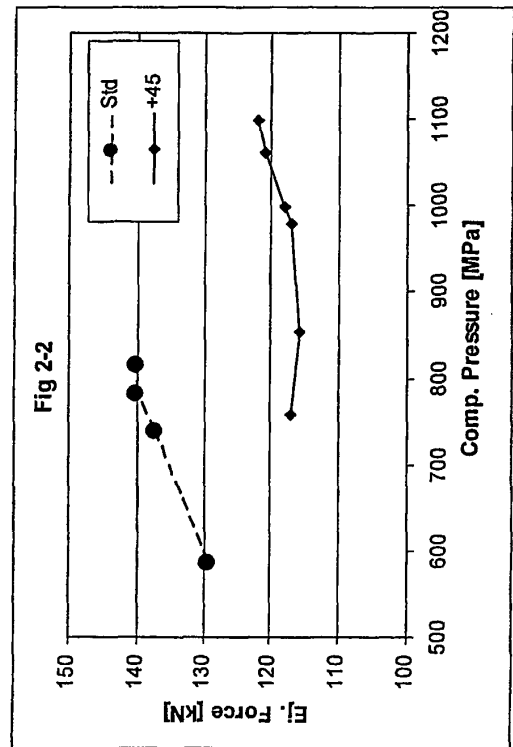
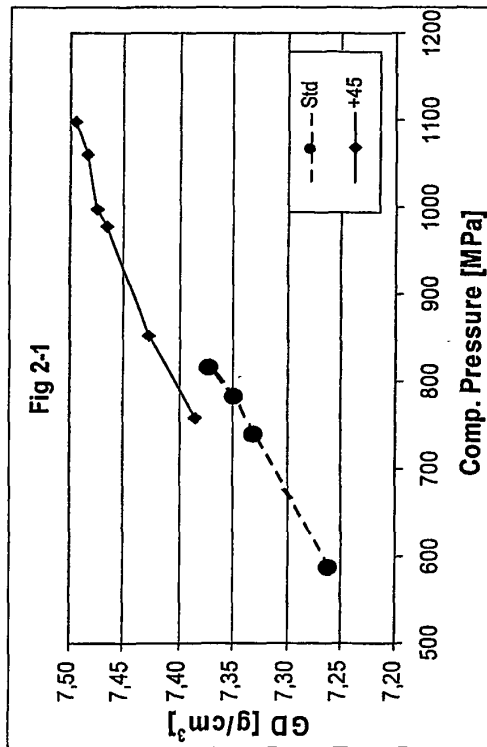
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# Example 1



- ◇ = Less shiny surface
- = Deteriorated surface

# Example 2





 Deteriorated surface



**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- US 5594186 A **[0011]**
- US 6309748 B **[0012]**
- US 4190441 A **[0013]**
- US 5225459 A **[0014]**
- EP 554009 A **[0014]**
- US 3901661 A **[0015]**
- WO 0238315 A **[0022]**