



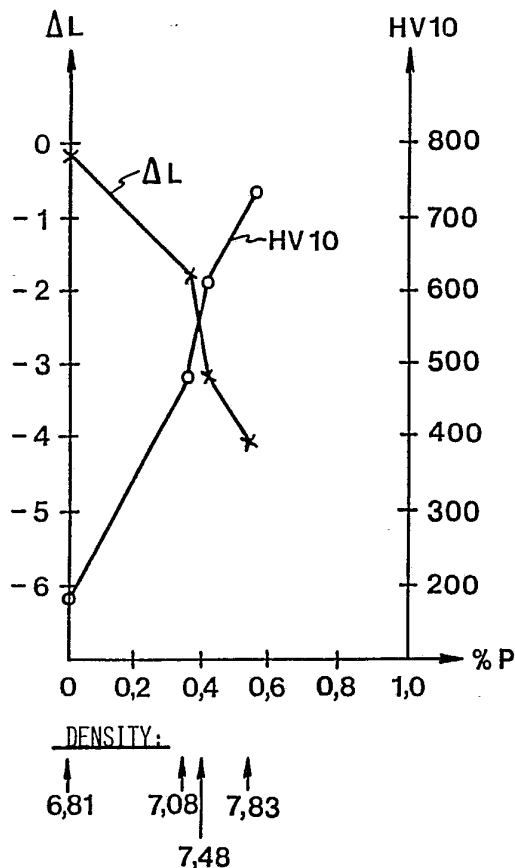
INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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| <p>(51) International Patent Classification <sup>5</sup> :<br/><b>C22C 33/02</b></p>   | <p><b>A1</b></p> | <p>(11) International Publication Number: <b>WO 91/18123</b><br/>(43) International Publication Date: 28 November 1991 (28.11.91)</p>   |
| <p>(21) International Application Number: PCT/SE91/00331<br/>(22) International Filing Date: 10 May 1991 (10.05.91)<br/>(30) Priority data:<br/>9001723-7 14 May 1990 (14.05.90) SE<br/>(71) Applicant (for all designated States except US): HÖGANÄS AB [SE/SE]; S-263 83 Höganäs (SE).<br/>(72) Inventors; and<br/>(75) Inventors/Applicants (for US only) : ENGDAHL, Per [SE/SE]; Långarödsvägen 52B, S-263 34 Höganäs (SE). SPASIC, Dragan [SE/SE]; Traktörsgatan 30, S-252 46 Helsingborg (SE). JOHANSSON, Ralf [SE/SE]; Järnvägs-gatan 27, S-261 32 Landskrona (SE).</p> |                  | <p>(74) Agent: AWAPATENT AB; Box 5117, S-200 71 Malmö (SE).<br/>(81) Designated States: AT (European patent), BE (European patent), BR, CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), NL (European patent), SE (European patent), US.<br/><br/>Published<br/><i>With international search report.</i></p> |

(54) Title: IRON-BASED POWDER, COMPONENT MADE THEREOF, AND METHOD OF MAKING THE COMPONENT

(57) Abstract

An iron-based powder for making wear-resisting and heat-resisting components by compacting and sintering contains, in addition to Fe, 3-15 % by weight of Mo and/or 3-20 % by weight of W, the total amount of Mo + W being in the range of 3-20 % by weight; 0.2-1.0 % by weight of P; 0.5-1.5 % by weight of C, and less than 3.0 % by weight of other substances. A component is powder-metallurgically made of this iron-based powder by compacting the powder into the desired shape and sintering the compact at a temperature below about 1150 °C.



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IRON-BASED POWDER, COMPONENT MADE THEREOF, AND METHOD OF  
MAKING THE COMPONENT

5           The present invention relates to an iron-based powder for making wear-resisting and heat-resisting components by compacting and sintering.

          The invention also relates to a component which is powder-metallurgically made of the inventive powder. Finally, the invention also relates to a method of powder-metallurgically making such a component.

          A well-known material in wear-resisting and heat-resisting components is the so-called high-speed steel. This is characterised by relatively high contents of alloying materials which above all are carbide-forming elements, i.e. provide wear resistance but also increase the hardenability and high-temperature strength of the component. Normal alloying materials in high-speed steel are Cr, Mo, W and V, but also Co and a number of other substances can be used.

          In order to achieve the purpose of high-speed steel, i.e. a powder-metallurgical material which is as hard, wear-resisting and heat-resisting as possible, a liquid phase sintering is performed after the powder has been compacted into the desired shape, whereby the component attains a high density.

          The high-speed steel powder itself is usually made by water atomisation. The carbon content is selected so that a subsequent soft annealing results in a powder in which the carbon is mainly bound in the form of carbides. To give the powder a desired compressibility, a low content of dissolved carbon is kept in the matrix.

          By today's technique, a high density of the sintered component is attained in that the sintering is carried out at 1250-1300°C and the content of C is kept in a narrow range. Generally use is made of vacuum sintering, but sintering in reducing atmosphere with a low dew point is

also applied. The sintering is carried out at these temperatures in order to provide sufficient liquid phase and thus cause shrinkage to the required high density.

The using of prior art combinations of alloying materials implies that the manufacture of a finished component, all the way from annealing to sintering, is complicated and expensive. Thus, the sintering temperature and carbon content must be carefully controlled to attain a sufficiently high density in the sintered material. The sintering temperatures used also render it impossible to perform the sintering in a belt furnace in which sintering temperatures above 1150°C normally cannot be achieved.

The object of the present invention therefore is to provide an iron-based powder which allows simple and relatively inexpensive manufacture of wear-resisting and heat-resisting components by compacting and sintering.

In particular, it should be possible to perform the sintering operation in a belt furnace, i.e. at lower temperatures than about 1150°C.

According to the invention, this object is achieved in that the iron-based powder contains, in addition to Fe, 3-15% by weight of Mo and/or 3-20% by weight of W, the total amount of Mo + W being in the range of 3-20% by weight; 0.2-1.0% by weight of P; 0.5-1.5% by weight of C, and less than 3.0% by weight of other substances.

Preferably, the powder contains no, or just a small amount of Cr and V which are sensitive to oxidation. The maximum total amount of Cr and/or V should be less than 2% by weight, preferably less than 1% by weight.

In a preferred composition, the powder contains 0.7-1.3% by weight of C, suitably however at least the amount which is required to form carbides with an included amount of Mo and W. Further, P can be included in the form of a phosphorous compound, suitably an iron phosphide, most preferably Fe<sub>3</sub>P. Finally, the amount of Mo can be 5-14% by weight, the amount of W 5-16% by weight, and the total amount of Mo + W should be in the range of 5-16% by weight.

Owing to the amount of P included, it has appeared that the inventive powder can be liquid phase sintered at the temperatures which are normally used for sintering in a belt furnace. The sintered material also has properties  
5 similar to those of high-speed steel, despite complete or substantially complete absence of Cr and, above all, V which is known to increase the heat resistance of the sintered material.

A further object of the invention is to provide a  
10 powder-metallurgically manufactured component, and this is achieved in that the component contains, in addition to Fe, 3-15% by weight of Mo and/or 3-20% by weight of W, the total amount of Mo + W being in the range of 3-20% by weight, 0.2-1.0% by weight of P, 0.5-1.5% by weight of C  
15 and less than 3.0% by weight of other substances.

Finally, one more object of the invention is to provide a method of powder-metallurgically making iron-based components, said method being characterised in that an  
20 iron-based powder is used, which contains, in addition to Fe, 3-15% by weight of Mo and/or 3-20% by weight of W, the total amount of Mo + W being in the range of 3-20% by weight, 0.2-1.0% by weight of P, 0.5-1.5% by weight of C and less than 3.0% by weight of other substances; that the  
25 powder is compacted into the desired shape, and that the compact is sintered at a temperature below about 1150°C.

In the inventive method, first a prealloyed powder can be made which consists of Fe, Mo and/or W and, optionally, C and/or P, and then the prealloyed powder thus made can be mixed with a lubricant, such as zinc stearate, and op-  
30 tionally graphite and/or P before compacting. Both P and C can thus be excluded from the prealloyed powder.

Like conventional high-speed steels, the material produced according to the invention can be used for components for use in metal-cutting, which requires excellent  
35 high-temperature strength, and for components subjected to wear, e.g. in motor-car engines.

The inventive iron-based powder is preferably made by water atomisation and is suitably soft annealed in a subsequent operation. The powder thus obtained is then mixed with graphite, P, most preferably in the form of  $\text{Fe}_3\text{P}$ , and a lubricant. Finally, compacting is effected and also liquid phase sintering at a temperature which preferably is below about  $1150^\circ\text{C}$ , thereby making it possible to use a conventional belt furnace.

By using, according to the invention, P and especially  $\text{Fe}_3\text{P}$ , the liquid phase in the compacted material is already attained at a temperature below about  $1150^\circ\text{C}$ , and the compact shrinks to a high density of the component manufactured.

The addition of P gives, in addition to the liquid phase, a solution-hardening effect in the sintered component. The amount of P, especially  $\text{Fe}_3\text{P}$ , is at the lower limit selected so that a sufficient amount of liquid phase for attaining the high density is obtained. The upper limit for the amount of P is justified by the fact that brittle phosphides tend to be formed and reduce the strength.

The amount of C should be selected so that at least a sufficient amount of carbides for improved wear resistance is formed. However, an excess amount of C should suitably be present in order to provide a sufficiently hardenable material. The presence of C is also important since it contributes to the liquid phase.

Mo and W are added to form carbides, which improves the high-temperature strength and wear resistance. Moreover, the hardenability is increased by adding Mo and W. The lower limit of Mo and W is selected in view of the fact that a sufficient amount of carbide-forming elements is required to provide the desired wear resistance and high-temperature strength.

By means of the invention, hardnesses and densities are attained which are on a level with those of conventional high-speed steel, and thus a corresponding wear resistance and high-temperature strength are also attained.

The invention is illustrated below by a number of Examples in which reference is made to the diagrams in Figs 1-8 in the accompanying drawings.

Example 1

5 Iron-based powders of the compositions shown in Table 1 were produced and compacted at a pressure of 589 MPa into test bars according to Swedish standard SS 11 21 23 and sintered at 1150°C for 1 hour. The values of quantity stated in Table 1 relate to % by weight.

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Table 1

| Mixture | Mo | W | P      | C | Fe      | Fig. |
|---------|----|---|--------|---|---------|------|
| 15 a    | 3  | 3 | 0-0.55 | 1 | balance | 1    |
| b       | 5  | 5 | 0-0.5  | 1 | balance | 2    |
| c       | 8  | 8 | 0-0.9  | 1 | balance | 3    |
| d       | 11 | 0 | 0-0.95 | 1 | balance | 4    |

20 Figs 1-4 show the shrinkage  $\Delta L$  in % during sintering of the compact, said shrinkage being a measure of the final density of the compact, as appears from the density values ( $\text{g/cm}^3$ ) stated under the diagrams. Figs 1-4 also show the hardness (HV10) at room temperature of the material in the sintered compact. As is apparent, an increasing amount of P results in a substantially increasing shrinkage and increasing hardness. According to the intended field of application for the finished component, the amount of P can according to the invention be selected somewhere in the range of 0.2-1.0% by weight. The lower limit can also be set at 0.3% by weight.

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Example 2

Iron-based powders of the compositions shown in Table 2 below were produced as well as compacted and sintered like in Example 1. The values of quantity stated in Table 2 relate to % by weight.

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Table 2

| Mixture | Mo | W | P   | C         | Fe      | Fig. |
|---------|----|---|-----|-----------|---------|------|
| 5 e     | 3  | 3 | 0.6 | 0.7-1.0   | balance | 5    |
| f       | 5  | 5 | 0.6 | 0.65-0.9  | balance | 6    |
| g       | 8  | 8 | 0.6 | 0.55-0.95 | balance | 7    |
| h       | 11 | 0 | 0.6 | 0.5-1.05  | balance | 8    |

10 As appears from Figs 5-8 which also show on the one  
hand the shrinkage  $\Delta L$  in % during sintering of the compact  
and the corresponding final density ( $\text{g/cm}^3$ ) and, on the  
other hand, the hardness (HV10) at room temperature of the  
material in the sintered compact, a substantially  
15 increasing shrinkage and increasing hardness are obtained  
as the amount of C increases. According to the intended  
field of application for the finished component, the amount  
of C can according to the invention suitably be selected  
somewhere in the range of 0.5-1.5% by weight, most prefer-  
20 ably in the range of 0.7-1.3% by weight.

In both Examples above, the particle size of the  
powder was smaller than 150  $\mu\text{m}$ , the average size being 70-  
80  $\mu\text{m}$ .

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## CLAIMS

1. Iron-based powder for making wear-resisting and heat-resisting components by compacting and sintering, characterised in that said powder contains, in addition to Fe, 3-15% by weight of Mo and/or 3-20% by weight of W, the total amount of Mo + W being in the range of 3-20% by weight; 0.2-1.0% by weight of P; 0.5-1.5% by weight of C, and less than 3.0% by weight of other substances.
2. The powder as claimed in claim 1, characterised in that P is included in the form of a phosphorous compound.
3. The powder as claimed in claim 2, characterised in that P is included in the form of an iron phosphide.
4. The powder as claimed in claim 3, characterised in that the iron phosphide is  $Fe_3P$ .
5. The powder as claimed in any one of claims 1-4, characterised in that C is included at least in the amount required for forming carbides containing Mo and W.
6. The powder as claimed in any one of claims 1-4, characterised in that the amount of C is 0.7-1.3% by weight.
7. The powder as claimed in any one of claims 1-6, characterised in that the amount of P is 0.3-1.0% by weight.
8. The powder as claimed in any one of claims 1-7, characterised in that said powder contains Cr and/or V in a total amount which is smaller than 2% by weight, preferably smaller than 1% by weight.
9. The powder as claimed in any one of claims 1-8, characterised in that it contains 5-14% by weight of Mo and/or 5-16% by weight of W, the total amount of MO + W being in the range of 5-16% by weight.
10. Powder-metallurgically manufactured component,

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c h a r a c t e r i s e d in that in addition to Fe, it contains 3-15% by weight of Mo and/or 3-20% by weight of W, the total amount of Mo + W being in the range of 3-20% by weight; 0.2-1.0% by weight of P; 0.5-1.5% by weight of C and less than 3.0% by weight of other substances.

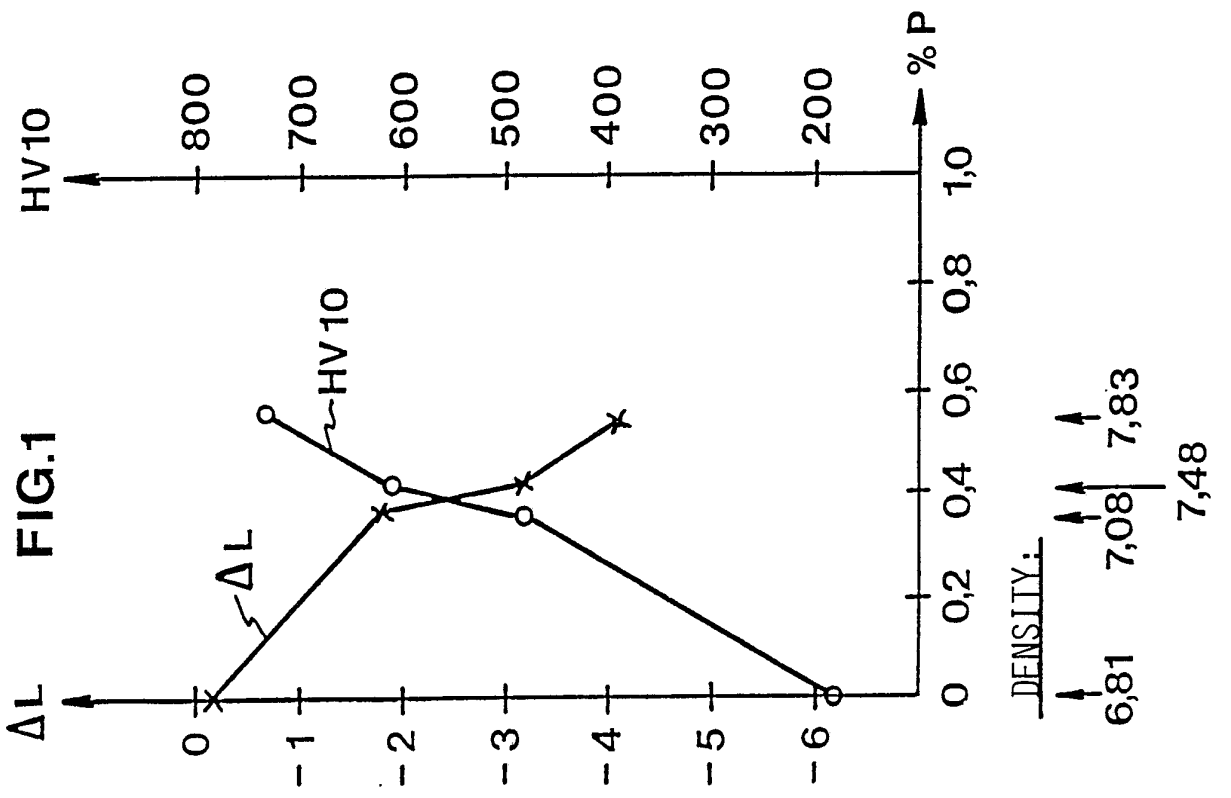
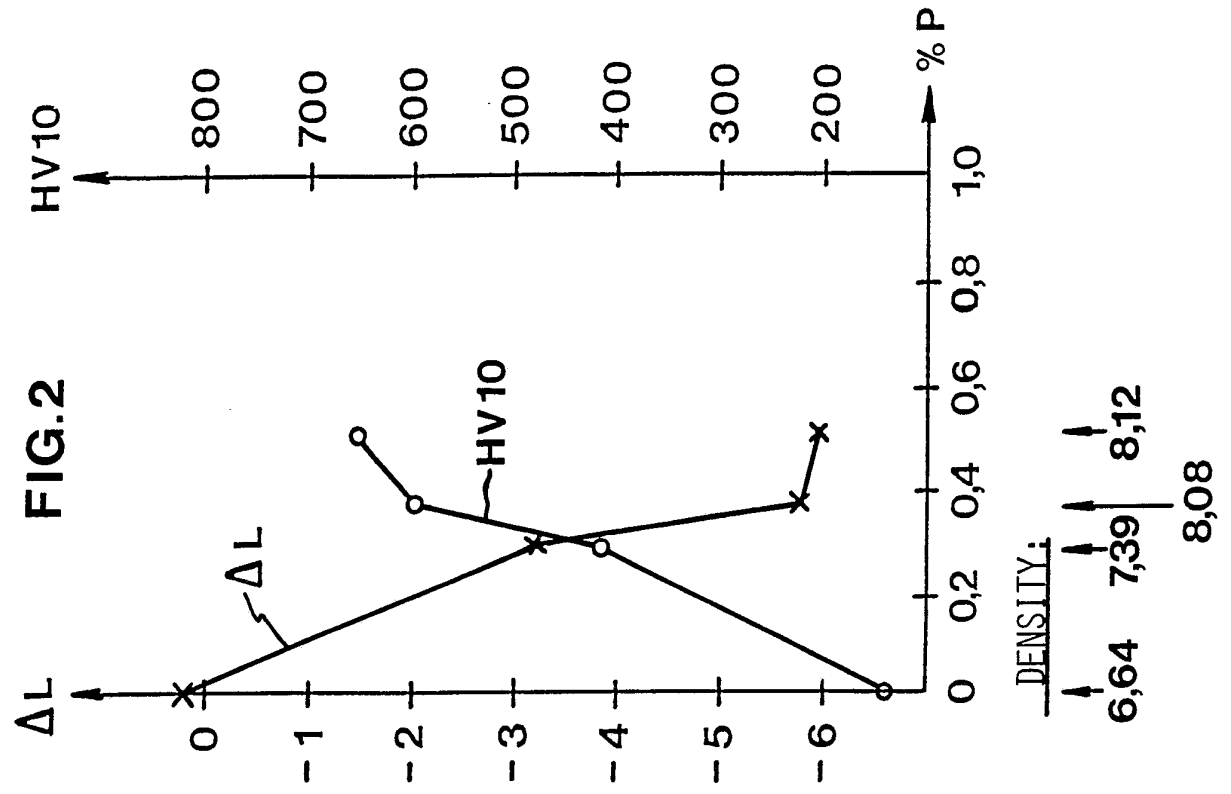
11. Method of powder-metallurgically making iron-based components, c h a r a c t e r i s e d in that an iron-based powder is used, which in addition to Fe contains 3-15% by weight of Mo and/or 3-20% by weight of W, the total amount of Mo + W being in the range of 3-20% by weight; 0.2-1.0% by weight of P; 0.5-1.5% by weight of C, and less than 3.0% by weight of other substances; that said powder is compacted into the desired shape, and that the compact is sintered at a temperature below about 1150°C.

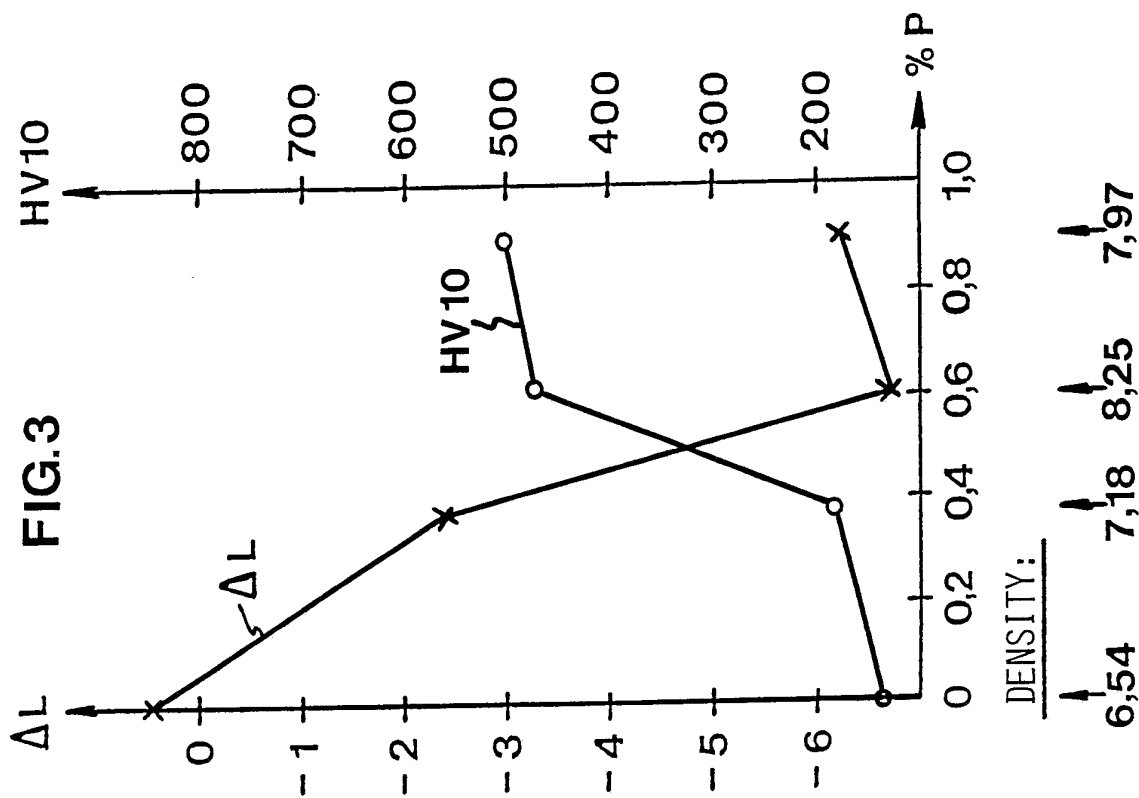
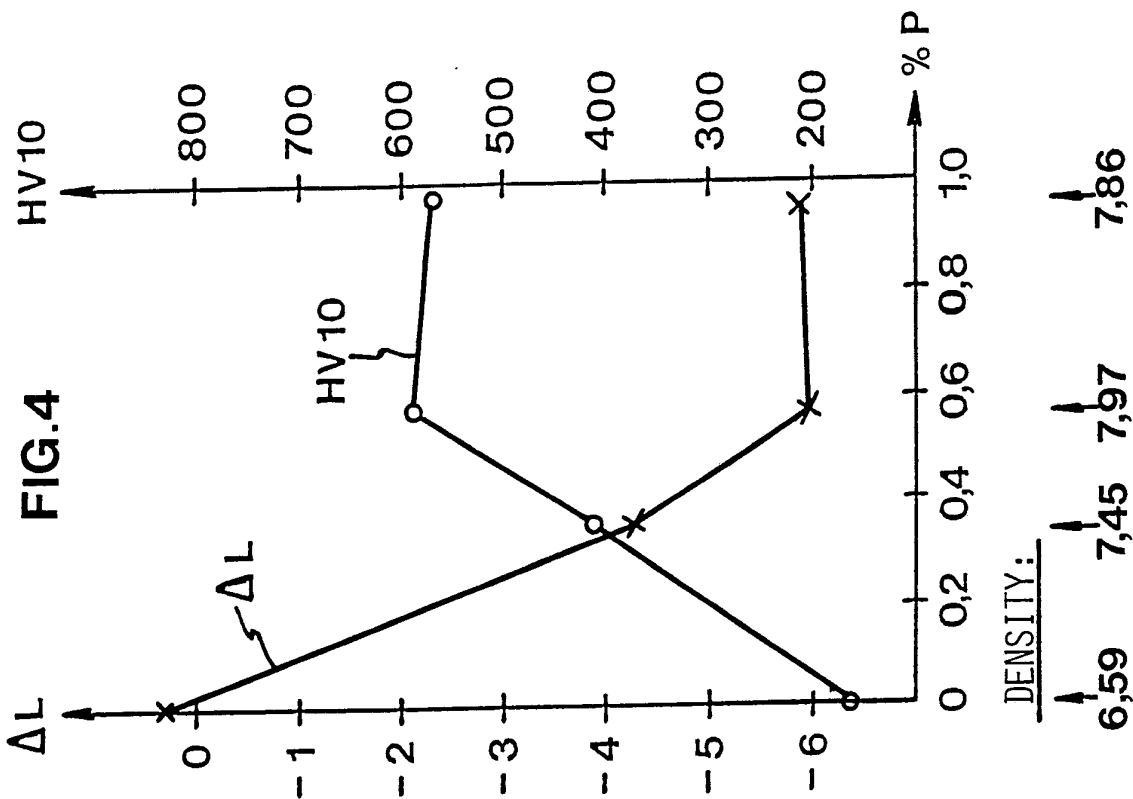
12. The method as claimed in claim 11, c h a r a c t e r i s e d in that first a powder is made, which consists of Fe, Mo and/or W, and optionally C and/or P, and that the powder made is mixed with a lubricant and, optionally, graphite and/or P before compacting.

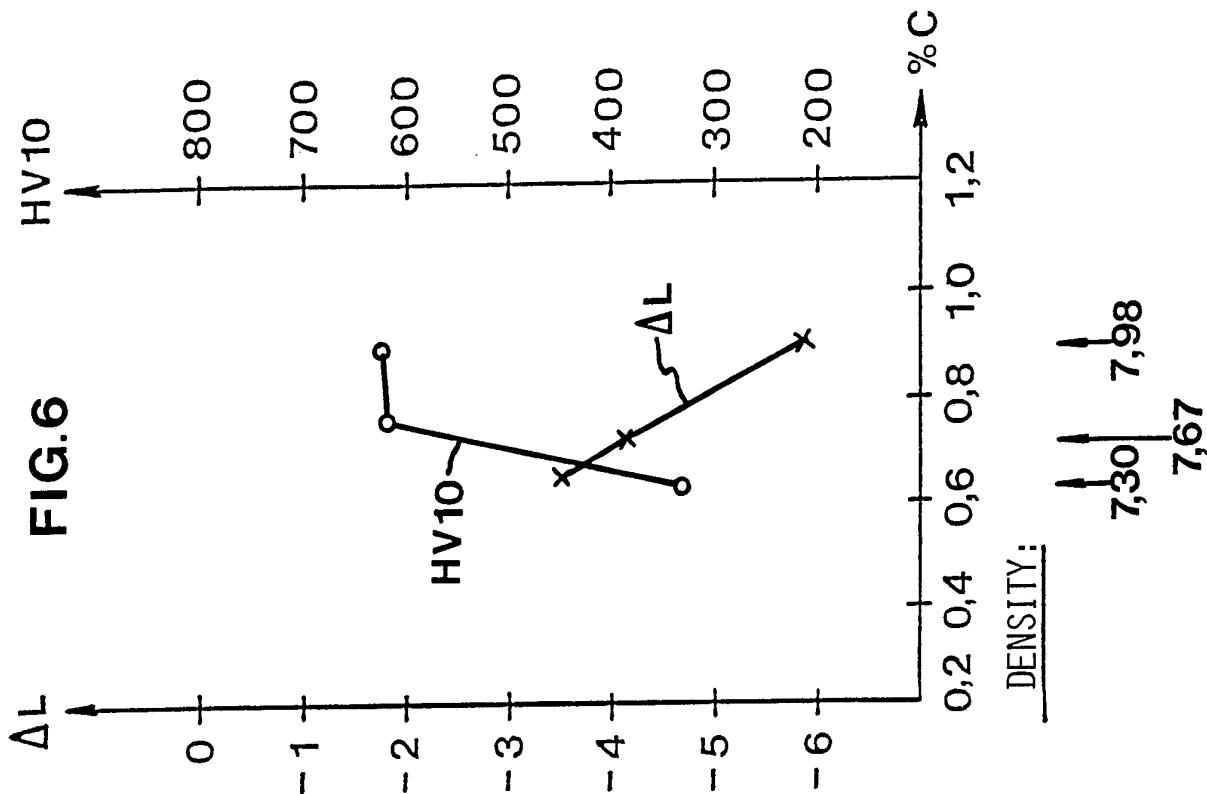
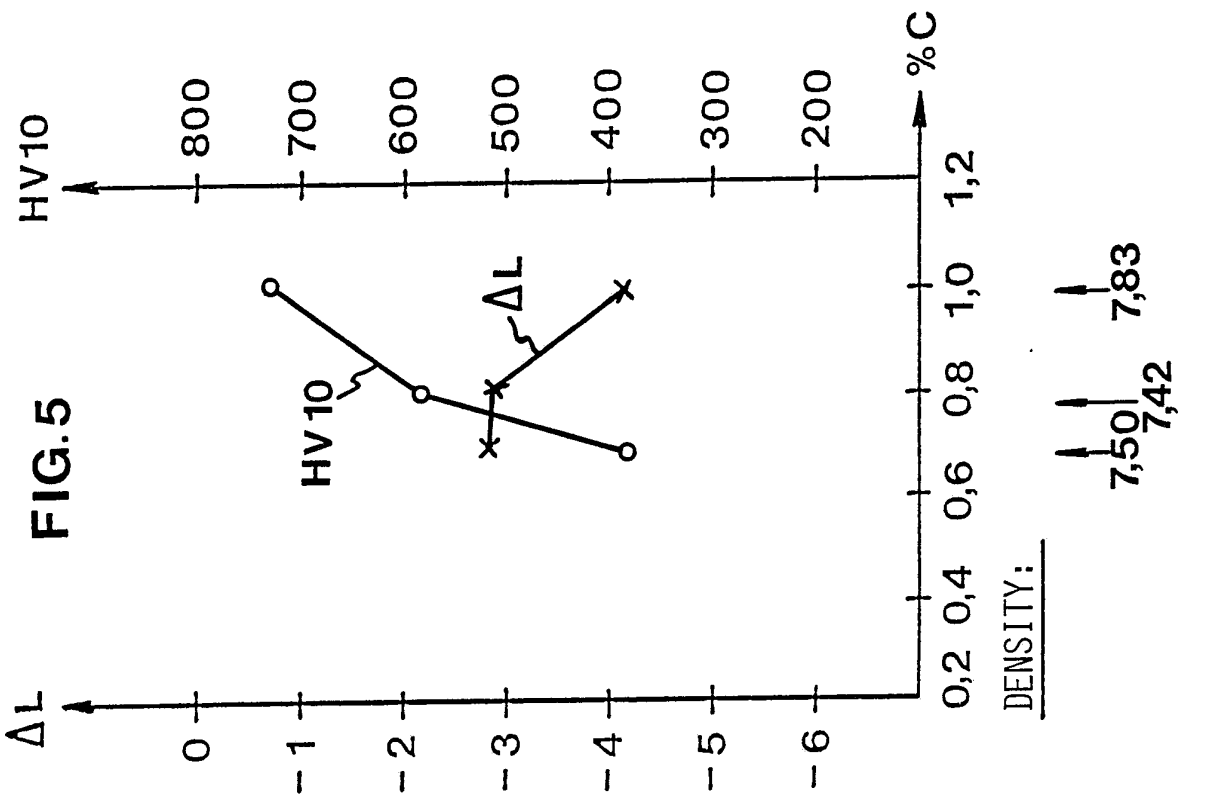
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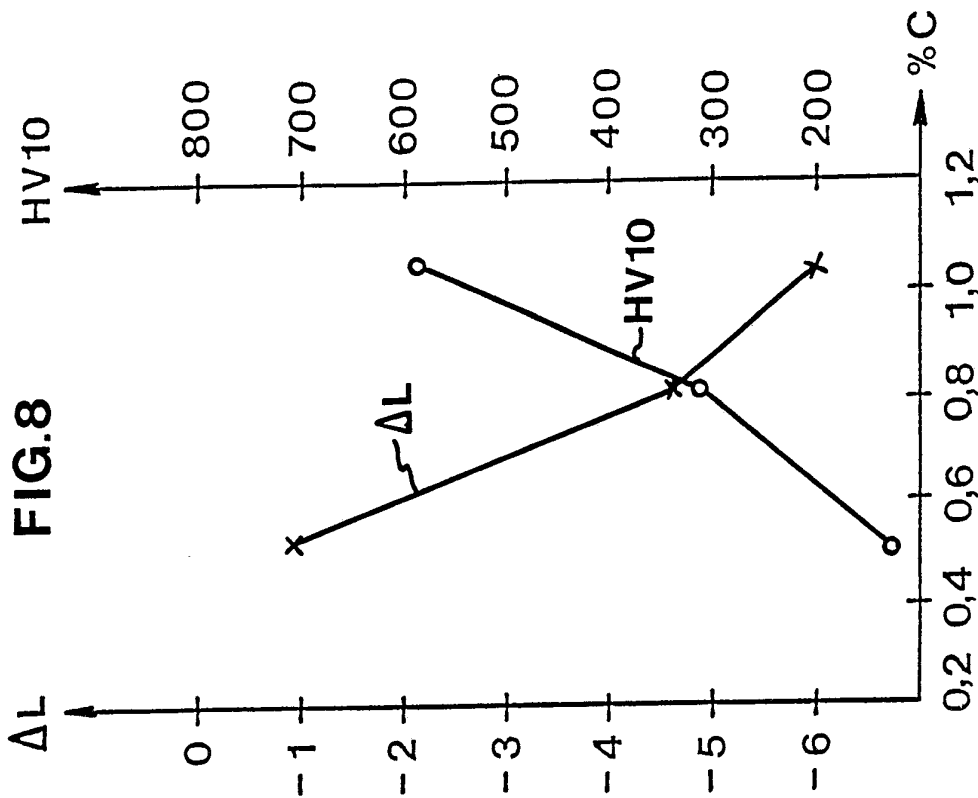
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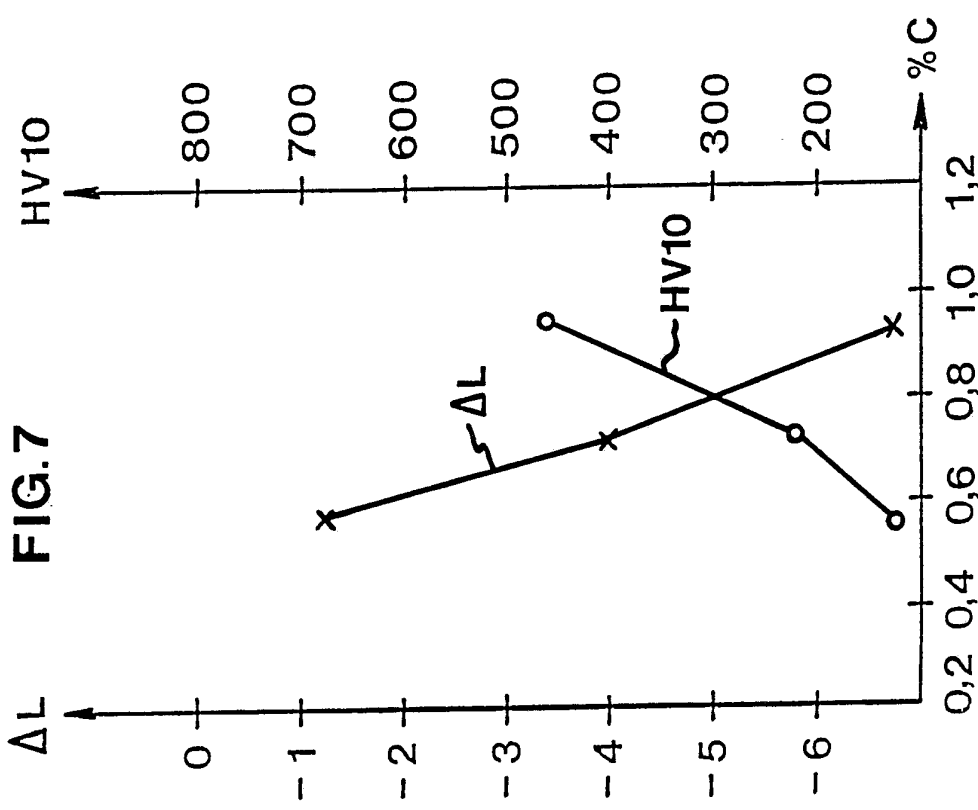








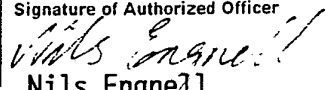
DENSITY:  
6,88 7,67 7,97



DENSITY:  
6,85 7,50 8,25

# INTERNATIONAL SEARCH REPORT

International Application No **PCT/SE 91/00331**

|   |   |                                     |
|---|---|-------------------------------------|
| <b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>6</sup>   |   |                                     |
| According to International Patent Classification (IPC) or to both National Classification and IPC   |   |                                     |
| IPC5: C 22 C 33/02  |   |                                     |
| <b>II. FIELDS SEARCHED</b>  |   |                                     |
| Minimum Documentation Searched <sup>7</sup>   |   |                                     |
| Classification System   | Classification Symbols  |                                     |
| IPC5  | B 22 F; C 22 C  |                                     |
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| SE,DK,FI,NO classes as above  |   |                                     |
| <b>III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup></b>   |   |                                     |
| Category *  | Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>            | Relevant to Claim No. <sup>13</sup> |
| A   | SE, B, 416824 (ROBERT BOSCH GMBH)<br>9 February 1981,<br>see the whole document<br>--                                     | 1-12                                |
| A   | WO, A1, 8803961 (MANGANESE BRONZE LIMITED)<br>2 June 1988, see page 8 - page 12;<br>page 22; page 26 - page 27<br>--      | 1-12                                |
| A   | DE, A1, 2428091 (TOYOTA JIDOSHA KOGYO K.K.)<br>16 January 1975, see page 3 - page 5;<br>page 11<br>--                     | 1-12                                |
| A   | GB, A, 2073247 (TOYOTA JIDOSHA KOGYO KABUSHIKI<br>KAISHA) 14 October 1981,<br>see page 2, line 16 - page 3, line 45<br>-- | 1-12                                |
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| <b>IV. CERTIFICATION</b>  |   |                                     |
| Date of the Actual Completion of the International Search   | Date of Mailing of this International Search Report   |                                     |
| 2nd August 1991   | 1991-08-02  |                                     |
| International Searching Authority   | Signature of Authorized Officer   |                                     |
| SWEDISH PATENT OFFICE   | <br>Nils Engnell                      |                                     |

| III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET) |   |                      |
|--|---|----------------------|
| Category *   | Citation of Document, with indication, where appropriate, of the relevant passages  | Relevant to Claim No |
| A  | US, A, 4696696 (YOSHIKI FUJITA ET AL)<br>29 September 1987, see column 1,<br>line 22 - line 34; column 1,<br>line 59 - column 2, line 39<br>column 4, tables 1 and 2<br>--<br>----- | 1-12                 |

ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.PCT/SE 91/00331

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the Swedish Patent Office EDP file on 91-06-27  
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| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|--|------------------|-------------------------|------------------|
| SE-B- 416824                           | 81-02-09         | AT-B- 361959            | 81-04-10         |
|  |                  | CH-A- 628089            | 82-02-15         |
|  |                  | DE-A-C- 2613255         | 77-10-13         |
|  |                  | FR-A-B- 2345526         | 77-10-21         |
|  |                  | GB-A- 1510455           | 78-05-10         |
|  |                  | JP-C- 1267536           | 85-06-10         |
|  |                  | JP-A- 52117814          | 77-10-03         |
|  |                  | SE-A- 7703382           | 77-09-28         |
|  |                  | US-A- 4128420           | 78-12-05         |
|  |                  | WO-A1- 8803961          | 88-06-02         |
| GB-A-B- 2197663                        | 88-05-25         |                         |                  |
| JP-T- 2500755                          | 90-03-15         |                         |                  |
| US-A- 4964908                          | 90-10-23         |                         |                  |
| DE-A1- 2428091                         | 75-01-16         | GB-A- 1428584           | 76-03-17         |
|  |                  | JP-C- 1094729           | 82-04-27         |
|  |                  | JP-A- 50013207          | 75-02-12         |
|  |                  | JP-B- 56038672          | 81-09-08         |
|  |                  | US-A- 3977838           | 76-08-31         |
| GB-A- 2073247                          | 81-10-14         | DE-A-C- 3048035         | 81-09-24         |
|  |                  | JP-C- 1242387           | 84-11-26         |
|  |                  | JP-A- 56123353          | 81-09-28         |
|  |                  | JP-B- 59018463          | 84-04-27         |
|  |                  | US-A- 4388114           | 83-06-14         |
| US-A- 4696696                          | 87-09-29         | DE-A-C- 3619664         | 86-12-18         |
|  |                  | GB-A-B- 2176803         | 87-01-07         |
|  |                  | JP-A- 61291950          | 86-12-22         |