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Datasheet for the decision of 13 October 2015

Case Number: T 0748/12 - 3.2.05

Application Number: 06755934.4

Publication Number: 1963721

IPC: F16J9/26

Language of the proceedings: EN

Title of invention:
Piston Ring for Internal Combustion Engines

Patent Proprietor:
Mahle International GmbH
Mahle Metal Leve S.A.

Opponent:
Federal-Mogul Burscheid GmbH

Relevant legal provisions:
EPC 1973 Art. 56, 83

Keyword:
Sufficiency of disclosure - (yes)
Inventive step - (yes)

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Case Number: T 0748/12 - 3.2.05

D E C I S I O N
of Technical Board of Appeal 3.2.05
of 13 October 2015

Appellant: (Opponent)	Federal-Mogul Burscheid GmbH Bürgermeister-Schmidt-Strasse 17 51399 Burscheid (DE)
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Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted on 12 January 2012 concerning maintenance of the European Patent No. 1963721 in amended form.**

Composition of the Board:

Chairman M. Poock
Members: S. Bridge
J. Geschwind

Summary of Facts and Submissions

I. The appeal is directed against the interlocutory decision of the opposition division stating that European patent No. 1 963 721 in amended form according to the auxiliary request meets the requirements of the European Patent Convention.

II. The opposition was filed against the patent as a whole based on the ground of opposition of lack of inventive step (Article 100(a) EPC in combination with Article 56 EPC 1973).

III. Oral proceedings were held before the board of appeal on 13 October 2015.

IV. The appellant (opponent) requested that the decision under appeal be set aside and that the patent be revoked.

V. The respondent (patent proprietor) requested that the decision under appeal be set aside and that the patent be maintained on the basis of claims 1 to 6 filed during oral proceedings before the board on 13 October 2015.

VI. Claim 1 according to the only request reads as follows:

"A piston ring for internal combustion engines, comprising:

a steel or cast iron base material; and

a coating of chromium nitride deposited by a physical vapour deposition process on a sliding surface of the piston ring,

characterised in that

the coating is composed of a columnar crystal structure of CrN with the following features:

(a) an oxygen content in solid solution of from 1.2 to 6.8 wt-%;

(b) a content of uniformly dispersed micropores of 1.5% to 4.0% in volume of the coating;

(c) an X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate in the range of 0.46 to 0.57;

(d) a Vickers hardness in the range of 1650 to 2100 HV."

VII. The following documents are referred to in the present decision:

D1: US-A-6,149,162;
D2: JP-A-8 296030 with English translation D2A;
D3: US-A-5,743,536;
D4: GB-A-2 276 176;
D5: Comparative tests of coatings GOE 240 and GOE 242, Summary page, figure pages ("*Bildtafel*") 1908 to 1911 from investigation report ("*Untersuchungs-bericht*") 2012/259 and figure pages ("*Bildtafel*") 1912 to 1915 from investigation report ("*Unter-suchungsbericht*") 2012/260, filed by the appellant with the grounds of appeal.

VIII. The arguments of the appellant in the written and oral proceedings can be summarised as follows:

The issue of sufficiency of disclosure was addressed in the contested decision (point III.6). The invention is not sufficiently disclosed, because the description does not provide indications how the claimed ranges of porosity, hardness and X-ray diffraction intensity ratios of (111) planes to (200) planes parallel to the

substrate can be obtained. If obtaining such values were to be considered obvious to the skilled person, then the subject-matter of claim 1 would lack an inventive step.

In addition, the effects of the invention (absence of cracks and spalling) are not proven for the whole of the claimed range, because according to table 1 of the patent in suit, only samples with an X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate of 0.51 were tested. Samples 10 and 11 with ratios of 0.46 and 0.57 were not tested. Furthermore, it is not plausible that a small difference in oxygen content of 0.4 or 0.5 leads to very different ratios of (111) planes to (200) planes: when comparing samples 5 and 8 or 6 and 12 in table 1, the results appear to contradict each other.

Considering document D1 as the closest prior art and, in particular, embodiment 2 (table 2) with an oxygen contents of 7%, the subject-matter of claim 1 only differs therefrom in features (c) and (d), whereby the claimed oxygen content (feature (c)) represents approximately a quarter to one third of the range disclosed in document D1. The comparative tests D5 provided by the appellant indicate that an oxygen content just outside the claimed range also solves the above problem, so that the claimed range is not inventive. The skilled person starting from embodiment 2 of document D1 will necessarily also consider providing an oxygen content around the 7% value, especially as this falls within the broader teaching of document D1 of an oxygen content of 0.5 to 20%. Thus, routine experiments and optimisation alone lead to features (c) and (d) of claim 1, because the oxygen content already effectively determines the hardness and

the X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate. The subject-matter of claim 1 is therefore not inventive.

Alternatively, starting from document D3, the subject-matter of claim 1 only differs therefrom in terms of the oxygen content. The problem to be solved is the one set out in the patent in suit (improved crack and peeling resistance) which is the same as the one addressed by document D1. In view of document D1's teaching of *preferred* (200) planes, the skilled person would not completely abandon the (111) planes disclosed in document D3 and thus, by routine optimisation alone, arrive at feature (c) as set out in claim 1. The subject-matter of claim 1 is therefore not inventive.

IX. The arguments of the respondent in the written and oral proceedings can be summarised as follows:

The skilled person is generally familiar with the physical vapour deposition (PVD) process and the patent in suit (Table 1) provides the required parameters so that the skilled person does not need more than routine experimentation to carry out the invention.

Of the cited documents D1 to D4, only the most recent document, D1, discusses all four of the parameters specified in claim 1 (features (a) to (c)). For this reason, document D1 constitutes the closest prior art.

The subject-matter of claim 1 differs therefrom in the combination of features (a) to (d). These characteristics are interdependent and cooperate to solve the problem of superior resistance to the initiation of micro-cracks and to the loss of pieces of the coating. There is no contradiction in the values presented in

table 1 of the patent in suit as explained in the description. None of the cited documents suggest the combination of features (a) to (d) as a solution to this problem. The subject-matter of claim 1 is therefore inventive.

Reasons for the Decision

1. *Sufficiency of disclosure (Article 100(b) EPC)*

1.1 The physical vapour deposition process

In claim 1 the CrN coating (obtained by a physical vapour deposition (PVD) process) is defined in terms of a particular range of values for each one of the four parameters:

(a) "*oxygen content in solid solution*" (wt-%);

(b) "*uniformly dispersed micropores*" (% in volume of the coating);

(c) "*X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate*" (a unit-less ratio);

(d) "*Vickers hardness*" (HV).

The PVD process is known as such as are the general relationships between the relevant parameters. These are as follows:

(a) "*The physical vapor deposition film of CrN with oxygen contained in a solid solution state can be covered by means of an ion plating method which uses metallic chromium as the evaporation source and uses nitrogen gas and oxygen gas as process gases*" (document D1, column 3, lines 11 to 15); "*Control of the oxygen concentration can be*

performed by adjusting the partial pressure of oxygen in the ion plating process. As the partial pressure of oxygen is raised, the oxygen concentration increases" (document D4, page 5, lines 23 to 26);

(b) "*The porosity of the coating can be controlled by adjusting the pressure used*" (document D3, column 4, lines 27 to 30);

(c) "*The preferred orientation [of the crystal planes] can be most easily controlled by means of the partial pressure of oxygen in the process gases and the bias voltage*" (document D1, column 3, lines 21 to 24);

(d) "*The coating hardness is affected by the crystal structure of the coating, the porosity, and the crystal orientation*" (document D3, column 3, lines 21 to 22).

Thus, document D3, which does not disclose a deliberate addition of oxygen, provides a coating with a crystal orientation having the (111) plane parallel to the substrate (column 2, lines 42 and 43) while document D1 discloses adding oxygen and controlling the bias voltage to obtain a coating with a preferred crystal orientation with the (200) plane parallel to the substrate (column 2, lines 62 to 64; column 3, lines 21 to 24).

In consequence, the amount of oxygen doping used in the PVD process determines feature (a) and, together with the bias voltage, feature (c), the pressure used will determine the porosity feature (b) while the hardness feature (d) is effectively dependent on features (b) and (c).

The appellant argued that general knowledge of the skilled person cannot be derived from a patent document such as documents D1 to D4. However, the statements quoted above do not relate to the specifics of the respective inventions disclosed in these documents but merely set out the generic characteristics of the PVD process and its parameters as such. In consequence, such statements can be taken as an indication of the general background knowledge of the person skilled in the art insofar as the PVD process as such is concerned. The appellant has not argued that the above knowledge of the PVD process would not be known to the skilled person.

1.2 The embodiments of the patent in suit (set out as samples 8 to 12 in table 1, page 6 of the patent as published) provide information concerning both the operating parameters (oxygen doping, pressure and bias voltage) and the resulting coating characteristics (oxygen content, porosity, hardness and the orientation and type of the crystal planes). Given the above understanding of the PVD process, the skilled person should not require more than routine experimentation to obtain the required coating when attempting to rework the embodiments of the invention, because all the relevant parameters (oxygen doping, pressure and bias voltage) are specified for samples 8 to 12 of the invention (table 1, page 6 of the patent as published).

The burden of proof for countering this prima facie evidence thus lies with the appellant. The comparative tests D5 provided on behalf of the appellant concern two coatings which do not display cracks (Summary page: "*Risse in Schicht*" - "*nein*"), have a respective oxygen content of 7.4 wt-% and 0.8 wt-% which lies outside the range claimed in claim 1 (feature (a): "*an oxygen*

content in solid solution of from 1.2 to 6.8 wt-%") and have unknown orientations and types of crystal planes. These examples thus demonstrate that coatings of unknown crystallography and oxygen contents outside the claimed invention may also constitute a solution to the problem of cracks forming in the piston ring coating. However, there are no examples which were obtained using the operational parameters values of the invention and which do not exhibit the required coating characteristics. Thus, the board has no basis for questioning the sufficiency of disclosure of the claimed invention (Articles 100(b) and 83 EPC).

2. *Inventive step (Articles 100(a) EPC in combination with Article 56 EPC 1973)*

2.1 Closest prior art

Document D1 discloses a sliding member such as a piston ring covered with a physical vapour deposition film of CrN having improved crack resistance and peeling resistance for use under harsh operating conditions and having superior durability (column 1, lines 5 to 9 and 42 to 47).

The solution according to document D1 involves a PVD film of CrN having oxygen contained in a solid solution in a range of 0.5 to 20 percent by weight, pores dispersed at a rate of 1 to 15 percent within the film, a crystal structure of CrN with a preferred (200) orientation parallel to a surface being covered and a Vickers hardness within the range of 1000 to 1800 HV (claim 2). In particular, document D1 discloses in an embodiment 2 (Table 2) a CrN coating with an oxygen content in solid solution of 7 wt-%, a porosity of 3 %, a preferred (200) orientation parallel to the surface,

a Vickers hardness of 1380 HV and crack loads greater than 150 N.

Thus, document D1 discloses, in combination, values for all of the parameters set out in features (a) to (d) of claim 1 and concerns a problem which is comparable to the one set out in the patent in suit.

In view of what is disclosed in document D3 (see point 2.5 below), the board considers that document D1 constitutes the closest prior art.

2.2 The technical problem specified in the patent in suit is to provide a piston ring with a chromium nitride coating with a crystalline structure by a physical vapour deposition process which has a superior resistance to the initiation of micro-cracks and to the loss of pieces of the coating, related to the propagation of such micro-cracks (paragraph [0012] of the patent as published).

2.3 In the judgement of the board, this problem is solved by the combination of features of claim 1, i.e. the value ranges of features (a) and (c) by which the subject-matter of claim 1 is distinguished from the piston ring of document D1, the value ranges of features (a) and (c) being themselves further limited by the value ranges of dependent features (b) and (d) (see point 2.4.1 below).

2.3.1 According to the patent in suit "*the results [of the tests of samples 1, 2, 4, 7, 8 and 12] lead to the conclusion that the most important characteristic influencing the behaviour of the chromium nitride coating concerning initiation of micro-cracks is <u>not the oxygen content</u> in the coating, <u>but the crystal</u>*

structure of it, which must have a minimum content of dense crystal planes (111) parallel to the coating surface. The oxygen content in the coating is important to cause the formation of this crystal structure. The desired crystal structure can only be obtained through a defined range of oxygen content in the coating" (paragraph [0041] of the patent as published, emphasis added by the board).

In view of the respective properties of the (111) and (200) planes (paragraphs [0014] and [0015]) the invention is "*able to provide a better compromise between these opposite effects mentioned, i. e., superior balance between internal stress absorption capacity and resistance to the initiation of superficial cracks due to external shearing stress, leading to a superior performance of this coating. Such objective was met by the balance of the relative content of (111) and (200) crystal planes on the coating*" (paragraph [0016]).

"*Surprisingly, the coatings with oxygen content between approximately 1.2 to 6.8 percent in weight of oxygen led to an intensity ratio of crystal planes (111) to (200) substantially higher than the described before, showing higher presence of crystal planes (111)*" (paragraph [0021]).

"*The present invention demonstrates that a coating having a specific balance between the incidence of crystal plane (111) and crystal plane (200) presents superior functional behavior and that only a narrow range, of approximately 1.2 to 6.8 percent in weight of oxygen, is able to provide a coating with crystal structure objected by the present invention*" (paragraph [0023]).

2.3.2 Thus, the range of oxygen content in solid solution of 1.2 to 6.8 wt-% corresponds to samples 8 to 12 in which the X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate is comparable to that of the samples 8 and 12 in which the coating showed no cracks or spalling. This X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate is distinguished from that of samples 1, 2, 4 and 7 in which the coating failed and which had ratios of (111) to (200) planes of at most 0.33 (see table 1).

2.3.3 The patent in suit explains that "*the transition of the crystal structure of a chromium nitride CrN coating with predominance of crystal plane (200) parallel to the surface to a crystal structure with an optimized balance between crystal planes (111) and (200) is not reached with a theoretical and precise oxygen content in the coating. Likewise, experimental techniques have certain dispersion, being necessary to define a functional range of oxygen for the achievement of the product of the present invention as approximately 1.2 percent in weight to approximately 6.8 percent in weight of oxygen in the coating*". Paragraph [0024] further compares samples 6 and 12 and 5 and 8 in which, although the oxygen content varies by 0.4 or 0.5, the corresponding ratio of (111) planes to (200) planes respectively varies from 0.04 to 0.51 and from 0.30 to 0.51.

The appellant refers to these same sample comparisons to argue that the resulting ratio of (111) planes to (200) planes is "*not plausible*". As the patent in suit has already explained this behaviour to be surprising (paragraph [0021]), the burden of proof lies with the

appellant. Since the appellant's argumentation did not go beyond questioning the values in the patent in suit, the board has no basis for questioning the results disclosed therein.

2.3.4 The appellant contests the technical effects achieved by the invention, in that, according to table 1, only samples 8 and 12 (with an X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate of 0.51) have been tested for cracks and spalling. This leads the appellant to conclude that the invention has not been demonstrated across all of the claimed range of oxygen values.

This approach ignores that the technical insight on which the invention is based is that the "*balance between the incidence of crystal plane (111) and crystal plane (200) presents superior functional behavior and that only a narrow range, of approximately 1.2 to 6.8 percent in weight of oxygen, is able to provide a coating with crystal structure objected by the present invention*" (paragraph [0023]). Thus, the respondent is entitled to claim the range of 1.2 to 6.8 percent in weight of oxygen which provides such a ratio of (111) to (200) planes as demonstrated in table 1 for samples 8, 9 10, 11 and 12 which have ratios of (111) planes to (200) planes respectively of 0.51, 0.50, 0.57, 0.47 and 0.51. As already noted above, these ratios of (111) planes to (200) planes are distinguished from those of the comparative examples 1 to 7 which culminate at 0.33 in sample 2.

The comparative tests D5 provided on behalf of the appellant concern coatings which do not exhibit cracks and do not indicate the X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the

substrate. As already noted above, the comparative tests D5 thus do not demonstrate that a coating falling within the ranges claimed in features (a) to (d) are subject to cracking and spalling. Therefore, there is no basis for the board to question the technical effect achieved by the invention.

2.4 Non-obviousness

2.4.1 To be suitable for use on the sliding surface of a piston ring, the coating porosity and hardness (features (b) and (d)) must also be within generally known working ranges, for known reasons, i.e. because:

- Increasing porosity decreases resistance to abrasion (document D1, column 1, lines 63 to 65; document D3, column 3, lines 14 to 16; column 13,lines 32 to 35); Less porosity leads to peeling (document D3, column 3, lines 12 to 14; column 14, lines 22 to 27) i.e. an absence of crack suppression (document D1, column 1, lines 62 and 63); and
- "*The wear resistance declines when the Vicker's hardness is less than 1000. The wear of the mating member increases when the Vicker's hardness is greater than 1800*" (document D1, column 1, lines 57 to 60).

The board notes, however, that features (a) to (d) are not independent, because they are all directly or indirectly dependent on the oxygen content (see point 1.1 above). Thus, although the skilled person is generally aware that certain ranges of porosity and hardness are necessary for a piston ring, these have to be achieved in the context of features (a) and (c) to obtain a piston ring according to the invention.

2.4.2 Document D1 also refers to other orientations of crystal planes parallel to the substrate: "*The film having a crystal structure of CrN with a preferred orientation of (111), (311), or (220) is brittle in comparison with the film with the preferred orientation (200)*" (document D1, column 2, lines 12 to 15). Although comparative examples 2 and 3 (document D1, column 5, table 2) exhibit no "*preferred orientation*" of the crystal plane, no conclusions are drawn from this in document D1, other than that "*the preferred orientation of the crystal structure of CrN deposited in the physical vapor deposition process may undergo complex fluctuations due to factors such as the process gas and the bias voltage*" (column 3, lines 18 to 21).

It was further argued on behalf of the appellant, that the "*preferred orientation (200)*" (column 2, lines 62 to 64) of the crystal planes parallel to the substrate according to document D1 could be interpreted as allowing for almost up to 50% of differently oriented crystal planes. However, such a definition of "*preferred orientation (200)*" is not explicitly provided in document D1, nor does it follow that the other crystal planes are necessarily all of (111) orientation, because document D1 also mentions further crystal orientations such as (311) and (220) and teaches away from them, because these orientations are deemed to be more brittle (column 2, lines 12 to 15).

Thus, document D1 does not hint at feature (c) even though the contemplated range of 0.5 to 20% oxygen content encompasses feature (a).

2.4.3 Document D2A discloses a coating comprising a mixture of CrN and Cr_2N which involves mixtures of (111) and (200) planes for CrN and of (002) and (112) planes for

Cr_2N. However, none of the examples involve an X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate in the range of 0.46 to 0.57. Document D2A further implies that the presence of Cr_2N affects the crystals of CrN (paragraph [0009]). Thus, there is no suggestion of feature (c) in document D2A.

Document D3 does not involve the addition of oxygen and finds that a (111) plane parallel to the substrate provides "*superior*" abrasion resistance. Thus, insofar as the (111) plane is concerned, there is a contradiction with document D1 (which considers the (111) plane too brittle - (Document D1, column 2, lines 12 to 15)) thus leaving the skilled person without any clear indication of what to do with respect to these (111) planes.

Document D4 does not discuss crystal planes, but recommends a Vickers hardness range of 1600 to 2200 (page 9, lines 17 to 21).

2.4.4 None of the cited documents discloses the X-ray diffraction intensity ratio of (111) planes to (200) planes parallel to the substrate (feature (c)) as such or in combination with the other features ((a), (b) and (d)) of claim 1.

Hence, it was not obvious for the skilled person, starting from the piston ring of document D1, to arrive at the one of claim 1.

2.5 Document D3 as alternative starting point for inventive step

The appellant considered document D3 as an alternative starting point for investigating the issue of inventive step.

Document D3 concerns a piston ring with a chromium nitride based coating, having good abrasion, baking and peeling resistance for use in an internal combustion engine (column 1, lines 9 to 13; column 2, lines 23 to 28). The disclosed solution involves a coating with a porosity of from 1.5% to 20% and a micro-hardness of from 600 HmV to 1000 HmV and the crystals of the coating may be oriented with the (111) face parallel to the surface (column 2, lines 37 to 43).

As was advanced on behalf of the appellant, the skilled person seeking to solve the problem of improved crack and peeling resistance would consider document D1, which addresses this problem and proposes a coating with a preferred (200) crystal orientation.

The further argument advanced on behalf of the appellant, that the fact that document D1 considers a "*preferred*" orientation (200) implies that other orientations may be present, does not appear to have an explicit basis in document D1, which does not provide such a definition for the term "*preferred orientation*". Furthermore, it is not clear why the skilled person would arrive at a coating in which both (111) and (220) planes are present in a ratio in the range of 0.46 to 0.57, since neither document D1 nor document D3 suggest a coating with both crystal plane orientations. In addition, such a combination would leave the skilled person with the further problem of reconciling the

teaching of document D3 which does not involve the addition of oxygen with that of document D1 where oxygen is added to achieve a "*preferred orientation (200)*". In addition, as already noted above, insofar as the (111) plane is concerned, there is a contradiction with document D1 (which considers the (111) plane too brittle - (Document D1, column 2, lines 12 to 15)) thus leaving the skilled person without any clear indication of what to do with respect to these (111) planes.

Hence, it was also not obvious for the skilled person starting from the piston ring of document D3 to arrive at the one of claim 1.

2.6 The subject-matter of claim 1 is therefore based on an inventive step (Article 56 EPC 1973).

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the department of first instance with the order to maintain the patent on the basis of the following documents:
   - claims 1 to 6 filed during the oral proceedings of 13 October 2015;
   - description as amended during the first instance proceedings;
   - figures 1 to 8 of the patent as granted.

The Registrar: The Chairman:

D. Meyfarth M. Poock

Decision electronically authenticated