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**Datasheet for the decision
of 24 May 2011**

Case Number: T 0663/08 - 3.3.05

Application Number: 01919046.1

Publication Number: 1274660

IPC: C03C 17/34

Language of the proceedings: EN

Title of invention:

Method for applying an antireflection coating to inorganic optically transparent substrates

Patentee:

Blösch Ressourcen AG

Opponent:

Satisloh AG

Headword:

Antireflection coating/BLOESCH RESSOURCEN AG

Relevant legal provisions:

EPC Art. 83, 100(b), 114(1)

Relevant legal provisions (EPC 1973):

EPC R. 55(c)

Keyword:

"Insufficiency of disclosure (yes) - gaps in information cannot be filled by general common knowledge - undue burden - research programme"

Decisions cited:

G 0010/91, T 0409/91, T 0435/91

Catchword:

-



Case Number: T 0663/08 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 24 May 2011

Appellant: Blösch Ressourcen AG
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Representative: Grünecker, Kinkeldey
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Respondent: Satisloh AG
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Representative: Oppermann, Mark
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 13 February 2008
revoking European patent No. 1274660 pursuant
to Articles 101(2) and 101(3)(b) EPC.

Composition of the Board:

Chairman: G. Raths
Members: H. Engl
D. Prietzel-Funk

Summary of Facts and Submissions

- I. European patent EP-B-1 274 660 was granted with 12 claims.

The independent claims as granted read:

"1. A method for applying an antireflection coating to a substrate of sapphire glass, which is an optically transparent, inorganic material, wherein alternating layers of different refractive indices are applied to the substrate by means of a plasma-enhanced PVD process, more particularly by so-called sputtering, the distance between the target and the substrate being chosen such that the scratching resistance of the obtained layers is similar to or higher than that of the substrate so that the light dispersion of the applied antireflection coating, measured by the "tightened Bayer test" defined in the description, is no more than twice as high as that of the uncoated sapphire glass."

"9. A watch glass of sapphire glass, wherein at least a part of the surface, preferably a magnifying lens, or the entire surface is provided with an antireflection coating produced according to the method of claim 1."

"12. A watch provided with the watch glass of claim 11."

- II. This appeal is from the decision of the opposition division posted on 13 February 2008 revoking the European patent in question.

III. The documents cited during the opposition proceedings included the following:

- A5: C.J. Mogab and E. Lugujo, "Backscattering analysis of the composition of silicon-nitride films deposited by rf reactive sputtering", J. Applied Phys., Vol. 47, No. 4, (1976), pages 1302 to 1309
- A6: Y. Hirohito et al., "Properties of silicon nitride films prepared by magnetron sputtering", Thin Solid Films 253 (1994), pages 425 to 429
- A7: Joo Han Kim and Ki Woong Chung, "Microstructure and properties of silicon nitride thin films deposited by reactive bias magnetron sputtering", J. Applied Phys., Vol. 83, No. 11, (1998), pages 5831 to 5839
- A8: JP-A-57 088 043 & English translation thereof
- A9: Opponent's Test Report No. 1 ("Versuchsbericht"), undated
- A10: ASTM Standard F 735-94

IV. The opposition division observed in the contested decision that the opposed patent disclosed neither the type of coating apparatus to be used, nor whether pulsed dc or rf sputtering was applied. Nor did the patent disclose any details concerning the flow rates of the reactant gases and the sputtering rate. The patent's lack of information regarding the operating conditions placed an undue burden on the skilled person attempting to repeat the claimed process and to obtain the desired peel- and scratch-resistant hard coatings. The patent was revoked because it did not disclose the invention in a manner sufficiently clear and complete

for it to be carried out by a person skilled in the art (Article 83 EPC).

- V. With its letter stating the grounds of appeal the appellant submitted new sets of claims as auxiliary requests 1 to 3, and *inter alia* the following document:

Annex 1: Letter from Rolex SA

- VI. The independent method claim 1 of auxiliary request 1 differs from granted claim 1 (main request) in that the passage:

"the substrate is disposed in the plasma area (2) and" is inserted between the words "chosen such that" and "the scratching resistance".

The wording of claim 1 in accordance with auxiliary request 2 differs from the wording of claim 1 as granted in that the passage

"wherein the ratio of the distance d_{ST} between said substrate and the target and of the target diameter q is equal to 1 at the most if said substrate is positioned approximately opposite the center of the target, said diameter being determined in non-circular targets by the largest circle fitting on the target surface under said substrate, and if said substrate is positioned otherwise, said distance is chosen such that the plasma density at the location of the said substrate is the same as or greater than in the case of said central positioning"

is added at the end of the claim.

The wording of claim 1 in accordance with auxiliary request 3 differs from the wording of claim 1 as granted in that the passage

"wherein the ratio of the distance d_{ST} between said substrate and the target and of the target diameter q is not greater than $1/2$ (one half), preferably no greater than $1/3$ (one third) if said substrate is positioned approximately opposite the center of the target, said diameter being determined in non-circular targets by the largest circle fitting on the target surface under said substrate, and if said substrate is positioned otherwise, said distance is chosen such that the plasma density at the location of the said substrate is the same as or greater than in the case of said central positioning"

is added at the end of the claim.

VII. With its reply, the respondent filed *inter alia* the following document:

E1: Opponent's Test Report No. 2, dated
4 February 2009.

A further submission of the appellant, received with a letter dated 7 April 2011, included the following additional documents:

Annex 7: Test Report of Prof. Pulker, undated; and
Annex 9: H.K. Pulker, "Coatings on Glass", Elsevier,
Amsterdam, 1999, pages 256 to 258

VIII. Oral proceedings were held on 24 May 2011. The appellant filed

Annex 12: H.K. Pulker, "*Coatings on Glass*", second Ed., Elsevier, Amsterdam, 1999, page ix (Preface).

IX. The appellant essentially argued as follows:

Late filed ground for opposition under Article 100(b) EPC

Said ground for opposition was raised only shortly before the oral proceedings before the opposition division. It should not have been admitted by the opposition division, as it was neither *prima facie* relevant nor substantiated. The crucial objection that essential process parameters were allegedly not disclosed was raised for the first time during the oral proceedings. The appellant had no opportunity to present counter-evidence. After admission of the fresh ground of opposition, the opposition division should have adjourned the oral proceedings to safeguard the appellant's right to be heard under Article 113 EPC.

Objections under Article 100(b) EPC

The appellant stressed that the technical field of sputtering of thin films, in particular of optical coatings on glass, was known to depend to a large degree on empirics. Often it was not possible to define precise parameters and to give clear instructions which

would lead to immediate success. The appellant referred to Annex 12 (Preface of Prof. Pulker's textbook, in particular to the second paragraph thereof), stating that inexplicable failures of a coating process had often been experienced. The skilled thin-film worker was therefore used to repeated experiments and painstaking optimization of process parameters for achieving the desired goal. The teaching of the opposed patent should be judged having this in mind. The patent indeed contained all the necessary information for successfully carrying out the claimed invention. At the time of the invention, dc sputtering had already by and large replaced the more complicated rf sputtering process. Therefore, it was not necessary to specifically disclose in the patent what was a matter of course for the skilled person and also for the respondent, who had carried out its experiments using pulsed dc sputtering. It was well known in the art of plasma coating that certain coating parameters, such as flow rates, process pressure, sputter rates, sputter power etc. had to be adjusted individually to the specific sputtering apparatus in use. No undue burden was involved with such orientating experiments.

The experiments carried out by the respondent deviated from the opposed patent in several important aspects, for instance as regards the target size, the location of the substrate, the preheating, and others. In particular, preheating of the substrate to 80°C was insufficient in view of the patent's teaching that the temperature difference between the plasma and the substrate, especially at the beginning of the sputtering, should be reduced. The Bayer abrasion test "under tightened conditions" had also been modified.

The respondent's experiments were thus not a correct re-working of the invention.

The respondent had apparently been successfully producing coatings according to the patent since 2005 (Annex 1).

On the contrary, new experiments of the appellant (Annex 7) clearly demonstrated that the information of the patent allowed one to obtain a satisfactory, peel- and scratch-resistant product, without undue trial and error. The results so obtained were essentially comparable to those of the opposed patent.

X. The respondent essentially argued as follows:

Late filed ground for opposition under Article 100(b) EPC

The appellant had during the oral proceedings full opportunity to present its counter-arguments. The appellant could also have requested the opposition division to adjourn the oral proceedings or to continue in writing, which it did not.

Objections under Article 100(b) EPC

The respondent maintained that essential parameters were not disclosed in the patent (see E3 and test report E1). Even when the substrate was preheated to 500°C, the coatings in accordance with the opposed patent were not obtained. Deviations in the so-called Bayer test under harsher conditions did not concern the coating process itself and were thus not relevant.

Coating not passing the standard Bayer abrasion test could not be expected to pass under stricter test conditions.

The respondent argued on the contrary that the experimental tests submitted by the appellant had been carried out under conditions which differed from those of the opposed patent. For example, the substrate was apparently not rotatably mounted during sputtering, as clearly called for in the patent. The heating by a 400W halogen, maintained during the coating process, was excessive and without a basis in the patent itself. A change of working gas during the coating was also not disclosed. For these reasons, the appellant's allegedly positive results were obtained only under process conditions which differed from the ones disclosed in the patent and which were unusual in the art. Therefore they could not support reproducibility and sufficiency of disclosure of the claimed invention.

Inventive step

The respondent maintained that sputtering of hard antireflective layers on sapphire glass substrates was known per se and so was sputtering at close target distances (A5 to A8).

XI. Requests:

The appellant requested that the decision under appeal be set aside and the patent be maintained as granted; or, in the alternative, that the patent be maintained in amended form on the basis of the claims filed with letter of 20 June 2008 as first to third auxiliary

requests, or that the case be remitted to the department of first instance for further prosecution.

The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal, although admissible, is not allowable.
2. Amendments (all requests)

The amended claims are fairly based on the originally filed application documents. The requirements of Article 123(2) EPC are met.

No objection under Article 123(3) EPC arises, as the amendments clearly limit the scope of protection conferred by the claims, having regard to the claims as granted.

Further detailed comments in this respect are unnecessary because the patent cannot be maintained for the reasons set out below.

3. Lack of sufficiency of disclosure (Article 100(b) EPC)
- 3.1 *Admissibility of the opposition ground raised under Article 100(b) EPC*

According to G 10/91 (OJ EPO 1993, page 420, Headnote I), new grounds of opposition which are not covered by the statement pursuant to Rule 55(c) EPC 1973 may not be considered by the Office of its own motion.

However, because of Article 114(1) EPC, the Opposition Division may, in exceptional cases and at its discretion, also consider other grounds for opposition which *prima facie* would seem to prejudice the maintenance of the European patent in whole or part (G 10/91, Headnote II).

The board considers that the evidence submitted by the opponent during opposition proceedings in support of its objection (E3 and test report E1) was at least *prima facie* prejudicial to the maintenance of the patent as granted. The opposition division therefore correctly exercised its discretion to admit this ground of opposition into the proceedings, although it was raised after the expiry of the opposition period.

As the ground of opposition was a subject of the first instance proceedings, the board is also empowered to investigate the matter. The opposition ground raised under Article 100(b) EPC is deemed to be admissible (Article 114(1) EPC).

3.2 *Substantiation of the objection*

3.2.1 The requirement of sufficiency of disclosure

According to the established jurisprudence of the boards of appeal, the requirement of sufficiency of disclosure is only met provided the invention as defined in the independent claim can be performed by the person skilled in the art within the whole area claimed without the burden of an undue amount of experimentation, taking into consideration common

general knowledge and the whole information content of the patent in suit (see decision T 435/91, OJ EPO 1995, 188, point 2.2.1, third paragraph, of the reasons; and T 409/91, OJ EPO 1994, 653, point 2, first paragraph, penultimate sentence).

3.2.2 The respondent's objection

The objection raised by the respondent is that the sputtering method as disclosed in the opposed patent did not lead to the desired scratch-resistant coatings, in particular coatings on sapphire glass which satisfy the scratch-resistance criterion set out in granted claim 1 of the opposed patent. Said claim 1 calls for a coating wherein *"the scratching resistance of the obtained layers is similar to or higher than that of the substrate so that the light dispersion of the applied antireflection coating, measured by the "tightened Bayer test" defined in the description, is no more than twice as high as that of the uncoated sapphire glass"*.

The said "tightened Bayer test" is a modification of a standard test method for testing abrasion resistance of coatings using the oscillating sand method, as published in ASTM F 735-94 (2001) (A10).

The respondent argued that according to claim 1 as granted the d_{ST}/q (target distance to target size) criterion as defined in paragraph [0021] of the description was the only parameter to be observed for obtaining coatings having the scratch-resistance defined in the claim. Although the said d_{ST}/q criterion was apparently met in all cases of the working

examples 1 to 3 of the opposed patent, only the coating obtained in accordance with example 1 in fact exhibited the desired level of scratch- and abrasion-resistance.

3.2.3 The gaps in information

It was not disputed that in particular the following process and apparatus information is not disclosed in the opposed patent:

- (a) the sputtering modus (rf sputtering or dc sputtering)
- (b) the sputter voltage, current and power
- (c) the nature of the target (dielectric or non-conductive, crystalline or amorphous)
- (d) the kind of electrical connections to the electrodes (cathode grounded or not, electrodes isolated or floating)
- (e) the flow rates of the gases
- (f) the partial pressures of the working gas and the reactive gas or gases
- (g) the sputter rates
- (h) the mounting of the substrate (grounded or isolated)
- (i) the vacuum pumps used (oil-diffusion pump, turbomolecular pump or getter pump).

3.2.4 Lack of guidance

(a) The respondent argued that the opposed patent did not provide the skilled person with a sound starting point for determining the correct process parameters by trial and error. Despite all efforts, the respondent had been unable to repeat successfully example 1 of the

opposed patent (see test report A9, filed during the opposition proceedings, and E1, filed during the appeal). According to the respondent, it was common practice in the field of sputtering that essential information be disclosed (A6, page 425, right hand column to page 426, left hand column, Table 1). It was even customary to cite the manufacturer and model of the sputter apparatus used (see A5, page 1302, right hand column, last paragraph).

(b) Referring to Annexes 1 and 7, the appellant argued that determining and adjusting most of the said process parameters were in fact essential because of the different behaviour of the plasma-enhanced reactive sputtering apparatuses, but were well-known to the person skilled in the art and could be determined by routine experimentation. Choosing a pulsed dc sputtering modus would be obvious as this method was known in the art (for instance from Annex 9) to prevent target poisoning.

(c) The question to be answered by the board is thus whether the gaps in information may be filled by the general common knowledge of the skilled person, without undue burden of trial and error.

For that purpose, the respondent and the appellant filed evidence.

3.3 *Evaluation of the experimental evidence submitted by the respondent*

3.3.1 Test report A9 contains data concerning the pulsed dc sputtering of a four layer system (20 nm silicon

nitride / 20 nm silicon dioxide / 90 nm silicon nitride / 129 nm silicon dioxide) on preheated sapphire substrates of 28 mm diameter. Four runs were carried out at distances of 88 mm, 68 mm, 58 mm and 48 mm, respectively, to the target (diameter 150 mm). The process pressure was 0.005 mbar, reactive gases nitrogen and oxygen, respectively, and the working gas was Ar (15 sccm) and the sputtering power was 1500 W.

In the subsequently performed Bayer abrasion test according to ASTM F 735-94 (conditions "moderately tightened"), none of the layer systems passed (the sputtered layers were completely removed).

In a subsequently filed experimental report E1, similar non-satisfactory results were obtained under essentially the same process conditions as above and under varying gas pressures and substrate/ target distances (48, 58, 68, 88 and 105 mm; target diameter 150 mm) (test runs 4L W_H, 4L W_M and 4L W_S). External preheating of the substrates was carried out at 80°C for 1 hour. In five samples (4L W_S) the coating was completely removed in the subsequent modified Bayer test. None of the remaining 10 samples of coated sapphire glasses satisfied the scratch-resistance criterion as per claim 1 of the opposed patent.

3.3.2 In the board's judgment, these experiments were carried out in a manner not contestable for the following reasons:

- The coating layer system and the substrate (sapphire) are exactly the same as in example 1 of the

opposed patent, as regards the chemical nature of the layers and their respective thicknesses;

- The ratios of the substrate distances of 88 mm, 68 mm, 58 mm and 48 mm, respectively, relative to the target of diameter 150 mm, in the four test runs in accordance with A9 and the fifteen runs in accordance with E1 satisfy the conditions required by the patent (paragraph [0021]). Although the target diameter of 150 mm used by the respondent in A9 differs from the 125 mm diameter target used in example 1 of the opposed patent, this difference is in the board's view not decisive because the same ratios d_{ST}/q of target distance d_{ST} to target diameter q as called for in the opposed patent have been used. The patent itself (paragraph [0021]) states that it is the ratio of the target distance to the target diameter, which is crucial for a correct positioning of the substrate. Therefore, the absolute size of the target cannot be important as long as this ratio is observed and the substrate fits the target (which was the case, as the substrate diameter was 30 mm). The values employed by the respondent in A9 in fact fall within the preferred range of values given in the opposed patent, namely $d_{ST}/q =$ less than 1, preferably 1/2, more preferably 1/3.

- Planetary motion was used, as prescribed by the opposed patent (Figure 1 and page 6, lines 20 to 31);

- The substrate was preheated;

- The particular choices of reactive gas and working gas, of sputtering power and gas flow rate, which are

not provided in the opposed patent, are conventional in the art and must be considered adequate for the purpose. The patentee itself agreed that pulsed dc sputtering was the method of first choice (although not disclosed in the opposed patent).

- The conditions of the modified Bayer abrasion test used in A9 were in fact less severe than the test proposed in the patent itself (reduced stroke length and frequency). The appellant accepted during the oral proceedings that the test employed in A9 was significant in so far as a sample failing under the said somewhat milder test conditions would inevitably also have failed under the harsher test conditions disclosed in the opposed patent.

Therefore, the board concludes that the experiments constituted a fair attempt at reworking the opposed patent. The negative test results obtained by the respondent thus show to a high degree of certainty to the board that the gaps in information in the application could not have been filled only by general common knowledge without undue burden of trial and error.

3.4 *Evaluation of the counter-evidence filed by the appellant*

On the contrary, the experimental counter-evidence of the appellant contained in Annex 7 is not convincing. The main reason is that it deviates in important aspects from the disclosure of the opposed patent. These aspects include the following:

3.4.1 Preheating

It is reported that heating of the substrate by means of a 400 W halogen lamp positioned at a distance of 6 to 7 mm sideways of the substrate was employed. During the oral proceedings, the appellant clarified that the preheating took place in the coating apparatus itself, not externally, and that no heating had been applied during the sputtering process itself. It was estimated that such preheating would raise the substrate temperature to several hundred centigrade.

The patent does not give any indication to what temperature the substrate should be preheated. It is also stated that the substrate should be preheated "*prior to the sputtering process*". There is no disclosure of internal preheating (*i.e.* heating within the sputtering apparatus). It is also noted that according to paragraph [0018] of the opposed patent, the substrates in the sputtering process of the invention reach temperatures of between 300°C and 400°C, and, in the case of isolated substrates, of up to 600°C. In view of this, internal preheating to high temperatures would appear unnecessary.

The board considers that the particular preheating step used in the appellant's experiments is not disclosed in or suggested by the opposed patent, which only briefly and generally mentions preheating as an optional measure. It is also not routine in plasma-enhanced reactive sputtering. The internal preheating by a powerful halogen lamp immediately before the sputtering, which must have led to temperatures of several hundred centigrade, may have had a decisive influence on the

adherence and the hardness of the coatings, thus partly explaining the differences between the appellant's and the respondent's experimental results.

3.4.2 Use of pure nitrogen during sputtering of silicon nitride

The opposed patent does not teach that the working gas (usually argon) should be replaced by nitrogen when sputtering certain layers. Replacing the standard working gas (Ar) during certain phases of the sputtering process could not be shown to be conventional practice. It must be assumed that the change of the working gas affected the coating properties.

3.4.3 Stationary substrate

In the appellant's experimental setup, the substrate was stationary during sputtering, as opposed to what is suggested in the patent itself (planetary motion). The board considers that the opposed patent, in dedicating one full paragraph of the otherwise succinct description and two figures (out of two) to the planetary motion of the substrate, inevitably conveys the impression of the importance of this particular feature. Therefore, the skilled person would have employed such substrate movement when trying to rework the claimed invention.

The respondent argued during the oral proceedings that such movement of the substrate could be important for layer homogeneity. The appellant maintained that moving of the substrate was not important, at least not where

only a single specimen was coated. In any event, the board considers that the respondent's experiments show that planetary motion of the substrate(s) alone does not guarantee success.

3.4.4 In view of these differences, the appellant's experimental evidence put forward as Annex 7 cannot be considered to represent a fair reproduction of the process as disclosed in the opposed patent. The results obtained thus cannot be taken into account in support of sufficiency of disclosure.

3.4.5 The appellant argued during the oral proceedings that in the field of plasma-enhanced reactive sputtering it was not practicable to give clear-cut and straightforward instructions directly leading to success. Rather, the skilled practitioner would typically arrive at the desired results by a largely empirical process of optimization. Reference was made in this context to the preface of the textbook "Coatings on Glass" by Prof. Pulker, referring to the predominantly empirical and sometimes even erratic nature of the art of thin-film deposition. The amount of process details given in the opposed patent should be judged against this background.

The board cannot, however, accept this argument. If the invention was in a technical field without a solid theoretical foundation it would have been all the more necessary to disclose all the available information which could be of use for the skilled person attempting to repeat the claimed invention.

3.5 *Conclusion*

The evidence filed by the respondent and the appellant lead to the conclusion that essential information is missing in the patent application, such that the skilled person is not in a position to carry out the claimed invention in the light of the teaching of the patent alone. The skilled person is forced to determine by trial and error from a multitude of possibilities the specific set of parameters which guarantees the desired result so as to fill in particular the gaps in information (a) to (e) cited under point 3.2.3.

Additional experimental work beyond mere routine and pure common general knowledge would be required in order to fill the gaps in information left in the patent. Carrying out a research programme of this scope would amount to an undue burden of trial and error experimentation in the sense of decision T 435/91 (*loc. cit.*).

The boards concludes that the invention was not disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art. The requirements of Article 100(b) in conjunction with Article 83 EPC are not met.

This negative finding in respect of the patent as a whole affects by its very nature all the pending requests, none of which is therefore allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

C. Vodz

G. Raths