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Datasheet for the decision of 28 November 2007

Case Number: T 0376/06 - 3.2.07

Application Number: 99901911.0

Publication Number: 1061052

IPC: C03B 20/00

Language of the proceedings: EN

Title of invention:
Synthetic silica glass optical members and process for the production thereof

Patentee:
Asahi Glass Co., Ltd.

Opponent:
Heraeus Quarzglas GmbH & Co. KG

Headword:
-

Relevant legal provisions:
-

Relevant legal provisions (EPC 1973):
EPC Art. 54, 56, 114(2)

Keyword:
"Novelty - yes"
"Inventive step - no"
"Late filed documents - not admitted"

Decisions cited:
-

Catchword:
-

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Case Number: T 0376/06 - 3.2.07

D E C I S I O N
of the Technical Board of Appeal 3.2.07
of 28 November 2007

Appellant: (Opponent)	Heraeus Quarzglas GmbH & Co.KG Quarzstraße 8 D-63450 Hanau (DE)
Representative:	Staudt, Armin Walter Patentanwalt Auf der Mauer 8 D-63674 Altenstadt (DE)
Respondent: (Patent Proprietor)	Asahi Glass Co., Ltd. 12-1, Yurakucho 1-chome Chiyoda-ku Tokyo 100-8405 (JP)
Representative:	Müller-Boré & Partner Patentanwälte Grafinger Straße 2 D-81671 München (DE)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted 8 February 2006 concerning maintenance of European patent No. 1061052 in amended form.**

Composition of the Board:

Chairman: H. Meinders
Members: P. O'Reilly
I. Beckedorf

Summary of Facts and Submissions

I. Opposition was filed against European patent No. 1 061 052 as a whole based on Article 100(a) EPC (lack of novelty and lack of inventive step).

The opposition division decided to maintain the patent in amended form.

II. The appellant (opponent) filed an appeal against that decision.

III. The appellant requested that the decision under appeal be set aside and the patent be revoked.

The respondent (patent proprietor) requested that the appeal be dismissed. Alternatively, the respondent requested that the decision under appeal be set aside and the patent be maintained in amended form in accordance with the first, second or third auxiliary requests filed with letter of 17 October 2007.

IV. Oral proceedings were held before the Board on 28 November 2007.

V. The independent claim of the patent as maintained (main request) reads as follows:

"1. A process for producing a synthetic silica glass optical component to be used by irradiating a laser light within an ultraviolet light wavelength region, which comprises a step of treating a synthetic silica glass to which forming by heating and annealing have been applied and which has a hydrogen molecule content

of less than $1x10^{17}$ molecules/cm^3 and has an OH group concentration of at most 200 ppm and contains substantially no reduction type defects at a temperature of from 300 to 600°C in a hydrogen gas-containing atmosphere at a pressure of from 2 to 30 atms, to obtain a synthetic silica glass optical component which has a hydrogen molecule content of at least $1x10^{17}$ molecules/cm^3 and an OH Group concentration of at most 200 ppm and contains substantially no reduction type defects."

The independent claim of the first auxiliary request reads as follows (amendments when compared to claim 1 of the main request are depicted in bold or struck through):

"1. A process for producing a synthetic silica glass optical component to be used by irradiating a laser light within an ultraviolet light wavelength region, which comprises a step of treating a synthetic silica glass to which forming by heating and annealing have been applied and which has a hydrogen molecule content of less than $1x10^{17}$ molecules/cm^3 and has an OH group concentration of at most ~~200~~ **100** ppm and contains substantially no reduction type defects at a temperature of from 300 to 600°C in a hydrogen gas-containing atmosphere at a pressure of from 2 to 30 atms, to obtain a synthetic silica glass optical component which has a hydrogen molecule content of at least $1x10^{17}$ molecules/cm^3 and an OH Group concentration of at most ~~200~~ **100** ppm and contains substantially no reduction type defects."

The independent claim of the second auxiliary request reads as follows (amendments when compared to claim 1 of the first auxiliary request are depicted in bold or struck through):

"1. A process for producing a synthetic silica glass optical component to be used by irradiating a laser light within an ultraviolet light wavelength region, which comprises a step of treating a synthetic silica glass to which forming by heating and annealing have been applied and which has a hydrogen molecule content of less than $1x10^{17}$ molecules/cm^3 and has an OH group concentration of at most ~~100~~ **80** ppm and contains substantially no reduction type defects at a temperature of from 300 to 600°C in a hydrogen gas-containing atmosphere at a pressure of from 2 to 30 atms, to obtain a synthetic silica glass optical component which has a hydrogen molecule content of at least $1x10^{17}$ molecules/cm^3 and an OH Group concentration of at most ~~100~~ **80** ppm and contains substantially no reduction type defects."

The independent claim of the third auxiliary request reads as follows (amendments when compared to claim 1 of the second auxiliary request are depicted in bold or struck through):

"1. A process for producing a synthetic silica glass optical component to be used by irradiating a laser light within an ultraviolet light wavelength region, which comprises a step of treating a synthetic silica glass to which forming by heating and annealing have been applied and which has a hydrogen molecule content of less than $1x10^{17}$ molecules/cm^3 and has an OH group

concentration of at most ~~80~~ **30** ppm and contains substantially no reduction type defects at a temperature of from 300 to 600°C in a hydrogen gas-containing atmosphere at a pressure of from 2 to 30 atms, to obtain a synthetic silica glass optical component which has a hydrogen molecule content of at least $1x10^{17}$ molecules/cm^3 and an OH Group concentration of at most ~~80~~ **30** ppm and contains substantially no reduction type defects."

VI. The documents cited in the present decision are the following:

D1: JP-A-H06-166528
D2: EP-A-0 401 845
D8: US-A-5 679 125

VII. The arguments of the appellant may be summarised as follows:

(i) The annexes E7 and E8 are late filed by the respondent and should not be admitted into the proceedings. E7 is an article from a journal published after the priority date. E8 is a set of experimental results without any accompanying information regarding the experimental conditions under which the experiments were performed.

(ii) The subject-matter of claim 1 of the main request lacks novelty in view of the disclosure of each of D2 and D8.

In D2 Sample 20 discloses all the features of claim 1 as shown in the description on page 17,

lines 16 to 32 and Table 2B. In order to ascertain the hydrogen molecule content and OH group concentration of this sample before the hydrogen treatment the data for Sample 11 must be used since this sample had the same initial treatment as Sample 20 but did not have the hydrogen treatment.

Example 2 of D8 takes away the novelty of claim 1. In column 10, lines 11 to 32 and Table 1 the treatment applied to this example is described. The treatment and values for the hydrogen content and OH group concentration are within the limits set out in claim 1. The hydrogen content before the hydrogen treatment must have been below the limit set in the claim as is shown by the comparative examples which have a content below the limit and were not treated with hydrogen. There also will have been no hydrogen type defects since there was an oxygen treatment before the hydrogen treatment and an oxygen treatment is known to remove hydrogen type defects. The subsequent treatment with hydrogen takes place in conditions within the ranges specified in the claim so that hydrogen type defects cannot be generated by this treatment. The respondent has argued that the prior chlorine and fluorine treatments produce hydrogen type defects which cannot be removed and that the invention lies in providing a starting material which does not have hydrogen type defects. This argument cannot be followed as there are no corresponding features in the claim, which thus does not exclude such treatments.

(iii) The subject-matter of claim 1 of the main request lacks an inventive step over D8 taking account of D1. Even if it has not been proven that there are no hydrogen type defects in the glass of Example 2 of D8 either before or after the hydrogen treatment it would be obvious to provide this feature. D1 shows the importance of removing hydrogen type defects. The skilled person would know that the oxygen treatment of the glass in Example 2 of D8 reduced the hydrogen type defects and also would know that the hydrogen type defects should be avoided and thus would ensure in Example 2 that there are no hydrogen type defects. The skilled person would therefore arrive at the subject-matter of claim 1 in an obvious manner.

(iv) The subject-matter of claim 1 of each of the first to third auxiliary requests lacks an inventive step. The increasingly lower upper limit for the OH concentration as set out in these claims does not avoid that the OH concentrations disclosed in Example 2 of D8 are still within the ranges specified in these claims.

VIII. The arguments of the respondent may be summarised as follows:

(i) E7 and E8 should be admitted into the proceedings.

E7 is a response to the filing of D8 by the appellant with the appeal grounds. E7 was only found a week before the oral proceedings which is why it is late filed. The document is a

publication of the appellant and it shows that the arguments of the appellant regarding D8 are not correct.

E8 is more evidence that chlorine treatment increases oxygen deficiency centres.

(ii) The subject-matter of claim 1 of the main request is novel over the disclosure of D2 and D8.

In D2 Sample 20 in fact is a number of samples since the description on page 17, lines 16 to 32 refers to ingots (plural). The description mentions a temperature range of 500 to 900°C for the hydrogen treatment. However, it is not known what the temperature was for the specific ingot of Sample 20 whose properties are shown in table 2B.

In D8 there is both a chlorine and a fluorine treatment which are known to produce hydrogen type defects. These defects cannot be removed completely so that a result of no hydrogen type defects cannot be achieved. The oxygen treatment does not remove all such defects. In accordance with the claim the starting material has no such defects and is not subject to a chlorine or fluorine treatment so that it never has any hydrogen type defects.

(iii) The subject-matter of claim 1 of the main request involves an inventive step. There is no mention in D8 of reducing hydrogen type defects. Moreover, it would not have been possible to remove all such defects because of the prior chlorine and fluorine

treatments which introduce defects which cannot be removed. It was the inventors of the present patent who first realised that it was essential to start from a glass material which has no hydrogen type defects since once such defects are present they can never be completely removed afterwards.

(iv) The subject-matter of claim 1 of each of the auxiliary requests involves an inventive step for the same reason as claim 1 of the main request.

Reasons for the Decision

1. *Admissibility of late filed evidence*

1.1 One week before the oral proceedings before the Board the respondent filed two pieces of evidence - E7 and E8. E7 is an article from a journal which was published after the priority date of the patent in suit. E8 shows the results of some experiments.

1.2 The respondent argued that E7 is a publication of the appellant and, although it is published later than the priority date, it is evidence regarding the knowledge of the skilled person.

Although this document is a publication it is in the view of the Board the equivalent to the filing of experimental results since its content is experimental results which were not in the public domain before the priority date. The filing of experimental results one week before the oral proceedings, however, sets the other party in a position in which it has little chance

to verify the results and/or file counter-evidence. The fact that the article stems from the appellant does not affect this finding since the appellant may need to make a reassessment of the results which were found seven years previously and could come to the conclusion that the experimental results are no longer valid.

With regard to the timing of the filing the respondent explained that it was a response to the filing of D8 with the appeal grounds and that the document had only just been found. The reasons why a document was filed so late play a lesser role than the rights of the other party that is confronted with the document. An admittance of the document so late in the proceedings quite clearly puts the appellant at a disadvantage irrespective of whether or not the respondent has a valid explanation for the late filing.

1.3 E8 is a table of results and a graph of these results with no indication of the experimental setup used to obtain the results. Since the validity of the test procedure used to obtain the results cannot be examined the results cannot be considered to be relevant.

1.4 The Board therefore did not admit either E7 or E8.

Main request

2. *Novelty*

2.1 The appellant argued lack of novelty of the subject-matter of claim 1 based on each of D2 and D8.

2.2 With respect to D2 the appellant argued that Sample 20 took away the novelty of claim 1 of the main request.

The description of Sample 20 is given on page 17, lines 16 to 32. There it is described how glass ingots are subjected to a treatment in an oxygen containing atmosphere at 1,100°C to remove oxygen deficiency defects - which are reduction type defects - followed by a hydrogen treatment at 10 atmospheres at a temperature of from 500°C to 900°C for from 10 to 100 hours depending upon the size of the ingot. In Table 2B values are given for various parameters of the samples including Sample 20. The OH group concentration is given as 200 ppm. The hydrogen content is given as $4x10^{18}$ molecules/cm^3. It is indicated that there are no oxygen deficiency defects.

The appellant further argued from the results of Sample 11, which did not undergo a hydrogen treatment but in the opinion of the appellant was otherwise treated similarly to Sample 20, there must have been no reduction type defects before the hydrogen treatment of the sample. It is not necessary to investigate this argument as the subject-matter of claim 1 is novel over D2 for another reason.

The description on page 17, lines 16 to 32 of D2 concerns a number of glass ingots. The treatment of these ingots varies with regard to the temperature and duration of the hydrogen treatment depending upon the size of the ingots. One of these ingots constitutes Sample 20. However, since there were a plurality of ingots it is not possible to know if the ingot whose values are listed in Table 2B under Sample 20 is one

which was treated with hydrogen at less than 600°C which is the upper limit specified in claim 1 for this treatment. Therefore, there is nothing in the document to show that the Sample 20 whose properties are set out in Table 2B was treated by a process within the scope of claim 1.

The disclosure of D2 therefore does not take away the novelty of claim 1.

2.3 With respect to D8 the appellant argued that Example 2 took away the novelty of claim 1 of the main request.

Example 2 is described in column 10, lines 11 to 32. The treatment of Example 2 is linked (see column 10, line 11) to the treatment of Example 1 which is described in column 8, lines 61 to column 9, line 53. The difference between these examples is that in Example 1 a treatment in the presence of oxygen was not performed (see column 8, lines 63 to 65).

In the examples a soot preform is first made. This is then dehydrated in a chlorine atmosphere and doped with fluorine in a silicon fluoride atmosphere. In Example 2 the resultant sample was oxygen treated at 1,050°C. The sample was then treated in a hydrogen atmosphere at 400°C at 6.0 atmospheres (see Table 1). Before the hydrogen treatment the OH group concentration was 75 ppb and after the treatment the OH group concentration was 1 ppm (see column 10, lines 30 to 32). After the hydrogen treatment the hydrogen content was $7x10^{17}$ molecules/cm^3 (see column 10, line 29).

2.4 The parties disputed whether the features of claim 1 whereby there are no reduction type defects both before and after the hydrogen treatment and whereby the OH concentration is less than 200 ppm at the start of the process are disclosed in D8. In addition, the hydrogen content before the hydrogen treatment was disputed.

With regard to the claimed range of the OH concentration and to the absence of reduction type defects the respondent argued that these features applied to what it considered to be the starting material for the process. The respondent further argued that this starting material was one that had never had any reduction type defects. The Board cannot agree with the respondent on this point. The claim is quite clearly directed to a process which "comprises" certain steps. This means that the process must have the specified steps though it may in addition have other steps. This interpretation of the word "comprises" is at the basis of claim drafting in the English language. When this meaning is not the intended meaning then different terminology must be used in the claim. The claim therefore merely specifies that there is at least a heating and annealing step and a subsequent treatment with hydrogen under the specified conditions. In addition, the claim specifies certain properties of the glass regarding OH group concentration, hydrogen molecule content and the absence of reduction type defects both before and after the hydrogen treatment.

2.5 In order to know the hydrogen content of Examples 1 and 2 before the hydrogen treatment recourse must be had to the comparative examples. Comparative Examples 1, 2 and 3 are based on Example 1 though without any hydrogen

treatment. They each show a hydrogen content of $1x10^{16}$ molecules/cm^3 (see column 10, lines 5, 44 and 55). Since these comparative examples are otherwise the same as Example 1 and Example 2 (apart from the oxygen treatment) they show the value of the hydrogen content before the hydrogen treatment, which is below $1x10^{17}$ molecules/cm^3 as required by claim 1.

2.6 Claim 1 specifies the OH group concentration before the hydrogen treatment as being below 200 ppm. In this respect the Board does not follow the argument of the respondent regarding the point in the process when there are no reduction type defects and the OH group concentration is at most 200 ppm. The claim quite clearly specifies that it is the silica glass having no reduction type defects and an OH group concentration of at most 200 ppm which is treated with hydrogen. There is nothing in the claim to exclude any particular prior treatments such as with chlorine or fluorine so long as the requirement of no reduction type defects when treated with hydrogen is kept. The argument of the respondent that there were **never** any reduction type defects is thus not based on the claim under consideration. In Example 2 of D8 the OH group concentration before the hydrogen treatment is given as 75 ppb. The Board is therefore of the opinion that the feature of the claim that the OH group concentration is at most 200 ppm is disclosed in D8.

2.7 The argument of the appellant regarding the absence of reduction type defects is based first of all on the treatment with oxygen and secondly on the test results shown in figure 4. With regard to the oxygen treatment the Board notes that from D2 (see page 17, lines 18

to 19) an oxygen treatment at 1,050°C anneals the glass and reduces oxygen deficiency defects which are reduction type defects. However, it cannot be concluded without further evidence that the oxygen treatment set out in D8 results in no reduction type defects. The further evidence is considered by the appellant to come from the results in figure 4 which the appellant considers shows that the glass after the hydrogen treatment had no reduction type defects and hence also must have had no reduction type defects before the treatment. Figure 4 is basically aimed at showing the lack of growth of defects during irradiation by an ArF laser in the glass of Examples 1 and 2 compared to the Comparative Examples 1 to 4. The scale of the graph, in particular the ordinate axis, is apparently chosen to allow the results to just fit on the graph. This means that the comparisons of the absorption may be arbitrary. Such an arbitrary scale is not suitable to prove that there are no reduction type defects, but just that there are less than in the comparative examples. The Board concludes that the features of claim 1 whereby the glass has no reduction type defects both before and after the hydrogen treatment is not disclosed in D8.

The disclosure of D8 therefore does not take away the novelty of claim 1.

2.8 Therefore, the subject-matter of claim 1 of the main request is novel in the sense of Article 54 EPC.

3. *Inventive step*

3.1 The appellant considered that the closest prior art document is D8 and the Board agrees with the appellant in this respect.

As explained above with respect to novelty the subject-matter of claim 1 is distinguished over the disclosure of D8 in that there are no reduction type defects both before and after the hydrogen treatment.

3.2 The problem to be solved would therefore be to improve the transmittance of KrF and ArF excimer lasers in glass (see paragraphs [0003] and [0005] of the patent in suit).

3.3 The solution to the problem is obvious to the skilled person.

Already from D8 the fact that the glass in Example 2 is treated with oxygen, which is known to reduce the presence of reduction type defects, is a clear hint to the skilled person towards their elimination.

It is furthermore known from D1 that reduction type defects cause problems for the use of KrF and ArF excimer lasers (see page 5, paragraph [0005] and page 6, lines 1 to 7). D1 recommends an oxidation treatment to reduce the reduction type defects (see page 6, paragraphs [0010] and [0011]) just as does D8. It is clear from D1 that the reduction type defects should be minimised before a hydrogen treatment (see paragraph [0012]) is carried out.

The skilled person is hence aware of the need to minimise reduction type defects so as to improve the laser transmission. It was thus clearly preferable for the skilled person that there should be no reduction type defects both before and after the hydrogen treatment. It would therefore be obvious to the skilled person to provide this feature in Example 2 of D8.

3.4 Therefore, the subject-matter of claim 1 of the main request does not involve an inventive step in the sense of Article 56 EPC.

Auxiliary requests

4. *Compliance with Article 123(2) EPC*

Since the requests are not allowable for other reasons (see below) it is not necessary to examine them for compliance with Article 123(2) EPC.

5. *Inventive step*

5.1 Claim 1 of each auxiliary request differs from claim 1 of the main request in that the upper limit for the parts per million (ppm) of the OH group concentration is lower. However, the argument which leads to the conclusion that claim 1 of the main request lacked an inventive step starting from Example 2 of D8 also applies to these requests. In that example the concentration of the OH group is 75 ppb before the treatment with hydrogen gas and 1 ppm afterwards. These values lie within the ranges specified in the claims 1 of each auxiliary request, just as they lie within the ranges specified in claim 1 of main request. Therefore,

the subject-matter claim 1 of each auxiliary request is distinguished over the disclosure of D8 by the same features as claim 1 of the main request. The subject-matter of claim 1 of each of these auxiliary requests hence lacks an inventive step for the same reasons as claim 1 of the main request.

5.2 Therefore, the subject-matter of the claim 1 of each of the auxiliary requests does not involve an inventive step in the sense of Article 56 EPC.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar: The Chairman:

D. Sauter H. Meinders