

**Internal distribution code:**

- (A) [ ] Publication in OJ  
(B) [ ] To Chairmen and Members  
(C) [X] To Chairmen  
(D) [ ] No distribution

**DECISION**  
**of 8 July 2003**

**Case Number:** T 0283/01 - 3.3.5  
**Application Number:** 96100118.7  
**Publication Number:** 0720969  
**IPC:** C03C 3/06  
**Language of the proceedings:** EN

**Title of invention:**

Silica glass, optical member including the same, and method  
for producing the same

**Patentee:**

NIKON CORPORATION

**Opponent:**

Heraeus Quarzglas GmbH & Go. KG

**Headword:**

Glass/NIKON

**Relevant legal provisions:**

EPC Art. 100b, 54, 56

**Keyword:**

"Sufficiency of disclosure (yes)"  
"Lack of novelty (not proved)"  
"Inventive step (yes)"

**Decisions cited:**

-

**Catchword:**

-



Case Number: T 0283/01 - 3.3.5

**DECISION**  
of the Technical Board of Appeal 3.3.5  
of 8 July 2003

**Appellant:** NIKON CORPORATION  
(Proprietor of the patent) 2-3, Marunouchi 3-chome  
Chiyoda-ku  
Tokyo 100

**Representative:** Hansen, Bernd, Dr. Dipl.-Chem.  
Hoffmann Eitle  
Patent- und Rechtsanwälte  
Arabellastrasse 4  
D-81925 München (DE)

**Respondent:** Heraeus Quarzglass GmbH & Co. KG  
(Opponent) Quarzstrasse 8  
D-63450 Hanau (DE)

**Representative:** Staudt, Armin, Dipl.-Ing.  
Patentanwälte  
Grimm & Staudt  
Edith-Stein-Strasse 22  
D-63075 Offenbach/M. (DE)

**Decision under appeal:** Interlocutory decision of the Opposition  
Division of the European Patent Office posted  
8 January 2001 concerning maintenance of  
European patent No. 0720969 in amended form.

**Composition of the Board:**

**Chairman:** R. K. Spangenberg  
**Members:** M. M. Eberhard  
J. H. Van Moer

## Summary of Facts and Submissions

I. This appeal is from the decision of the opposition division according to which European patent No. 0 720 969 in amended form meets the requirements of the CBE. The decision was based on the granted claims, as the main request, and on a set of amended claims submitted on 20 October 2000 as the auxiliary request. claims 1 and 13 of the main request read as follows:

"1. A silica glass for use with light in a wavelength region of 400 nm or shorter, having a structure determination temperature of 1200 K or lower and a hydrogen molecule concentration of  $1 \times 10^{17}$  molecules/cm<sup>3</sup> or more."

"13. A method for producing a silica glass having a structure determination temperature of 1200 K or lower and a hydrogen molecule concentration of  $1 \times 10^{17}$  molecules/cm<sup>3</sup> or more, comprising the steps of heating a silica glass ingot having a hydrogen molecule concentration of  $1 \times 10^{17}$  molecules/cm<sup>3</sup> or more to a temperature of 1200-1350 K, retaining the ingot at that temperature for a given period of time, and then, cooling the ingot to a temperature of 1000 K or lower at a temperature-lowering rate of 50K/hr or less to anneal the ingot."

II. During the opposition proceedings it was relied *inter alia* on the following documents:

E4: EP-A-0 401 845

E5: US-A-5 364 433

E6: Diffusion curve submitted by the opponent

E8: EP-A-0 483 752

E9: Physical Review B, vol. 28, 6, 15 September 1983,  
pages 3266-3271, A.E. Geissberger and  
F.L. Galeener.

In its decision the opposition division held that the requirement of sufficiency of disclosure was not met since the skilled person would not be in a position to determine the "structure determination temperature" (hereinafter SDT) of a silica glass specimen of unknown glass origin. He could only unambiguously determine (i) the minimum required molecular hydrogen concentration and (ii) the maximum allowed normalised intensity of the  $606\text{ cm}^{-1}$  Raman scattering signal. Furthermore, the silica glass according to granted claim 1 lacked novelty since both conditions (i) and (ii) were satisfied by the glass of E8.

III. Both the patentee (hereinafter appellant) and the opponent filed a notice of appeal. However the opponent did not submit a statement of grounds of appeal within the four months time limit set out in Article 108 EPC. An application for re-establishment of rights in respect of this time limit was filed on 21 August 2001 simultaneously with a statement of grounds of appeal. Oral proceedings took place before this board on 3 September 2002 in which only the issue of re-establishment of rights was dealt with. The board decided that the opponent's request for

re-establishment of rights was rejected (see Interlocutory decision T 283/01 of 3 September 2002). The opponent is referred to hereinafter as the respondent.

IV. Oral proceedings were held on 8 July 2003. The appellant requested that the decision under appeal be set aside and that the patent be maintained as granted. In the alternative they requested that the patent be maintained in the amended form maintained by the opposition division. The respondent requested that the patentee's appeal be dismissed.

V. The appellant presented inter alia the following arguments:

The patent specification contained a description as to how the SDT could be determined, in particular how the calibration curve was obtained. It was clear that a calibration curve had to be prepared for each glass ingot. The respondent's assertion that it was impossible to establish a calibration curve because test samples having a SDT <1200 K could not be produced, was not correct. It was based on information concerning the method of production of the silica glass in accordance with the present invention instead of the method for establishing the calibration curve. The respondent's arguments concerning the dimensions of the glass sample and the hydrogen content of the glass were mere allegations not supported by any evidence although the burden of proof lay with the opponent. E8 did not destroy the novelty of the glass of granted claim 1. E8 taught that the ratio  $R_2$  of the Raman intensities  $I_{606}/I_{800}$  should preferably be less than 0.15; however

the SDT value of the glass could not be deduced from the said ratio. Samples of the ingot had to be quenched in liquid nitrogen to obtain a calibration curve. E8 was silent about the quenching step.  $R_2 < 0.15$  encompassed  $R = 0$  but did not disclose it. Moreover such a ratio corresponded to an ideal glass structure not obtainable in practice. Performing the teaching of example 13 of E8 did not inevitably lead to a glass having the claimed SDT. In this example the temperature was slowly and constantly decreased and the glass had thereby not the possibility to reach the equilibrium state.

Starting from E8 as the closest prior art, the problem to be solved was to provide a silica glass having a high structural stability. It should have a good balance of the properties required for a silica glass to be used with laser radiation, ie a small amount of strains, a small amount of surface dents and a small increment of the refractive index. E4 contained no incentive regarding the relationship between the SDT and the required properties, ie amount of strain, increment of refractive index and surface dents. The skilled person would have had no reason to combine the teaching of E9 with the teaching of E8. E9 was silent about the hydrogen molecule concentration, the SDT parameter or a quenching in liquid nitrogen. If the skilled person had used a lower retention temperature in the process of E8 he would not have quenched the glass in water since it was an essential feature of E8 to cool the silica glass slowly and carefully.

VI. The respondent's arguments can be summarised as follows:

The invention was not sufficiently disclosed as regards the determination of the SDT. No calibration curve was disclosed in the patent in suit and it was not possible on the basis of the information given therein to draw up a calibration curve. According to the patent in suit a sample having a SDT of 1200 K or less could not be produced with the given process since a SDT-value of 1200 K or less could not be obtained if the annealing completion temperature was higher than 1000 K. The samples used for making the calibration curve were however all heated to higher temperatures, namely 1073 to 1700 K and then quenched. Therefore, either the information in the patent in suit were not correct, or the calibration curve could not include SDT-values of 1200 K or less. It was also indicated in the patent in suit that, if the retention temperature was lower than 1200 K, the SDT could not be lowered to 1200 K or less in a limited time period. Therefore the structural equilibrium state could not be reached for test samples heated at a retention temperature  $< 1200$  K and the calibration curve could thus not include SDT-values of 1200 K or less. At the oral proceedings the respondent admitted that it was not technically impossible to determine a SDT at 1073 K but pointed out that the period of time for achieving the equilibrium state at this temperature would be extremely long. Moreover the patent in suit contained no data about the geometry of the samples used to prepare the calibration curve although the behaviour of a sample during quenching depended on the volume, form and dimensions of the sample. Dimensions as indicated on page 9 of the patent in suit seemed to be unreasonable for a sample to be

quenched and were inconsistent with the dimensions indicated in E9. As the appellant alleged that the sample used for establishing the calibration curve had the same dimension as the samples used for measuring the UV light resistance, the burden of proof rested on him for this affirmation. The description was also not sufficient as to how the hydrogen molecule content could be maintained at the same value before and after annealing. It was known that hydrogen diffused very quickly out of silica glass at high temperatures and E6 showed that after about 20 hours no measurable amount of hydrogen remained in the middle of the sample. It could also be inferred from E4 that hydrogen diffused out of the sample at temperatures between 500 and 900°C.

The claimed glass lacked novelty with respect to E8 which taught that the intensity ratio  $R_2$  was lower than 0.15. This teaching encompassed the value  $R_2 = \text{zero}$  where the glass contained no structural imperfection, which therefore lay within the claimed range of SDT. Moreover, in example 13 the sample was cooled from 1200 K to 1073 K within a time period of about 13 hours. As according to the patent in suit 1 to 24 hours were necessary for achieving the equilibrium state with a retention temperature of from 1073 to 1700 K, it could be expected that the SDT would be  $< 1200$  K in example 13 of E8. Concerning inventive step, E8 dealt with the same problem as the patent in suit and taught that the Raman intensity ratio  $R_2$  should be as low as possible in order to improve the durability against laser radiation. A decrease of  $R_2$  was in fact a diminution of the amount of unstable structures and thus a low SDT. In view of this teaching it was obvious



to solve the problem of providing a glass with an improved UV-durability since E8 gave the direction to be followed, namely lower structural imperfections, in particular lower  $R_2$ . The skilled person knew in view of E9 how to decrease  $R_2$ , namely by decreasing the fictive temperature  $T_F$ . The SDT differed from  $T_F$  only in that the former was determined by quenching in liquid nitrogen instead of quenching in water. E9 further disclosed that retention temperatures as low as  $900^\circ\text{C}$  were possible, and the relaxation time necessary to achieve the equilibrium state could be inferred from Table V and equation (5). By heating the hydrogen-containing glass of E8 to a retention temperature of  $900^\circ\text{C}$  (1173 K), maintaining the glass at that temperature till the equilibrium state was obtained and then quenching the glass in water, the skilled person would automatically have arrived at a glass falling within the definition of claim 1, ie having a SDT  $<1200$  K, since the SDT cannot be higher than 1173 K. Concerning the quality criteria reported in Table 3 of the patent in suit, a comparison of example 12 with comparative examples 1, 2 and 3 showed that the properties of the glass of example 12 were not improved.

### **Reasons for the Decision**

1. The patentee's appeal is admissible.
2. The opponent filed a notice of appeal but did not submit the statement of grounds of appeal within the four months time limit set out in Article 108 EPC. At the end of the oral proceedings held on 3 September

2002 this board decided that the opponent's request for re-establishment of rights in respect of the time limit for submitting the statement of grounds of appeal was rejected (see interlocutory decision T 283/01 of 3 September 2002 for the reasons). Consequently, the opponent's appeal is not admissible (Article 108 and Rule 65 EPC).

3. *Sufficiency of disclosure*

The respondent's objections in relation to the sufficiency of disclosure were directed to both parameters stated in granted claim 1, ie the concentration of hydrogen molecules and the SDT.

- 3.1 The respondent alleged that none of the examples of the patent in suit could be reproduced since a silica glass having a hydrogen concentration of  $1.10^{17}$  molecules/cm<sup>3</sup> or more could not be obtained due to the hydrogen molecules diffusing out of the glass during the heating and annealing steps. The board observes that although the burden of proof rests on the respondent with respect to this allegation, they did not reproduce any of the examples of the patent in suit to demonstrate that the hydrogen content reported therein is actually not correct or not obtainable. The respondent's allegation is based on a simulation involving a calculation which was contested by the appellant (E6). Although it is known that hydrogen molecules diffuse out of the glass during a heating step at high temperatures, the results of the calculation and the curve in E6 are not consistent with the examples of the patent in suit. In these examples a certain amount of hydrogen escaped from the glass during the heating and

annealing steps (about  $1 \times 10^{17}$  mol./cm<sup>3</sup> in examples 1 to 6, 9 and 11 and about  $1 \times 10^{16}$  mol./cm<sup>3</sup> in examples 7 to 8, 10, 12: compare Tables 1 and 2 of the patent in suit), however the amount of hydrogen molecules remaining in the glass after the annealing step was at least  $1 \times 10^{17}$  mol./cm<sup>3</sup>, ie the lower limit stated in claim 1. Furthermore, it is also derivable from E8 that it is possible to obtain a silica glass having a hydrogen molecule content of at least  $1 \times 10^{17}$  mol./cm<sup>3</sup> even after a heating and annealing step at high temperatures despite the diffusion of hydrogen molecules (see the examples). To support his allegation the respondent also made reference to comparative examples 12, 13 and 14 of the patent in suit in which the samples were kept at 973 K for 60 hours under vacuum in order to decrease the hydrogen molecules concentration to less than  $5 \cdot 10^{16}$  mol./cm<sup>3</sup> (comparative example 12) or less than  $1 \times 10^{16}$  mol./cm<sup>3</sup> (comparative examples 13 and 14). In comparative examples 13 and 14 the heat treatment was performed under a vacuum of  $10^{-5}$  Torr. It cannot be derived therefrom that a hydrogen concentration of  $\geq 1 \times 10^{17}$  mol./cm<sup>3</sup> is not obtainable in the examples of the patent in suit since the heat treatment in the comparative examples was performed under a high vacuum whereas the examples of the patent in suit do not involve a vacuum heat treatment. At the oral proceedings the respondent further argued that it could be inferred from E4 that hydrogen diffused out of the silica glass at temperatures from 500 to 900°C since hydrogen was introduced into the silica glass at these temperatures (see page 17, lines 25 to 27). This argument cannot be accepted by the board because it only takes into consideration the temperature used during the treatment and fully ignored the pressure. E4

in fact teaches that the ingots are heated at about 500 to 900°C for 10 to about 100 hours under a hydrogen gas atmosphere at a pressure of 10 At. In this context the board further makes reference to the second method disclosed in E4 for measuring the hydrogen concentration of the silica glass. The sample is heated to 1000°C at a heating rate of 4°C/min and maintained at 1000°C for 2 hours in a vacuum atmosphere of  $1 \times 10^{-7}$  Torr (see page 11, lines 46 to 50). The respondent has given no convincing reason why the pressure (or vacuum) would have no influence on the amount of hydrogen introduced into (or removed from) the glass. In these circumstances and in the absence of evidence that the reproduction of the examples of the patent in suit does not lead to the hydrogen content stated therein, the board is not convinced that the description is not sufficient for the skilled person to obtain a glass having the required content of hydrogen molecules.

- 3.2 Concerning the SDT parameter, the patent in suit discloses on page 3, lines 36 to 57, and page 12, lines 39 to 41, how the SDT of a glass can be obtained. It is in particular pointed out that a plurality of samples of a silica glass are retained at a plurality of temperatures within the range of 1073 to 1700 K for a period longer than the structure relaxation temperature in air, each sample thereby having the structure in the equilibrium state at the retention temperature. The samples are then quenched in liquid nitrogen in order to fix the structure thereof to the structure at the retention temperature. Thereafter a Raman measurement is effected wherein the ratio of the Raman line intensities at wavelength of 606 and 800  $\text{cm}^{-1}$ , ie the intensity ratio  $I_{606}/I_{800}$ , is determined for each

sample of the silica glass. A calibration curve is drawn up from the two variables, ie retention temperature (corresponding to the SDT) and  $I_{606}/I_{800}$ . The unknown SDT of a further sample of this silica glass is then determined using the calibration curve by plotting the measured  $I_{606}/I_{800}$  and reading the corresponding SDT. Although the patent in suit does not disclose a calibration curve, it contains sufficient information as to how such a curve can be obtained. It is not explicitly indicated in the patent in suit that a calibration curve is prepared for each different glass; however, the respondent did not give any reason at the oral proceedings why the absence of an explicit indication in this respect would result in the skilled person not being able to put into practice the teaching of the patent in suit. The patent in suit does not disclose that the same calibration curve should be used for all silica glasses whatever their method of production or their thermal history. As according to the patent in suit the SDT is a factor which expresses the structural stability of a silica glass, it is plausible that a new calibration curve has to be made for silica glasses prepared differently. This was not contested by the respondent.

The respondent argued that it was impossible to establish a calibration curve in the range 1073 K to 1200 K since according to the patent in suit (i) the SDT cannot be lowered to values  $\leq 1200$  K in a limited period when the retention temperature is lower than 1200 K and (ii) the SDT-values could not be lowered to values  $\leq 1200$  K when the annealing completion temperature was higher than 1000 K. The board cannot follow these arguments for the following reasons. The

passages of the patent in suit referred to by the respondent with respect to (i) and (ii) are namely on page 7, lines 43 to 45, and page 7, lines 53 to 55. In the first passage it is in fact stated that "when the retention temperature is lower than 1200 K, the structure determination temperature cannot be lowered to 1200K or lower in a given period, and, furthermore, annealing is insufficient and strain cannot be removed". The expression in a "given period" cannot be interpreted in this context as meaning that the time period is not a "limited period". In the next sentence of the patent in suit (page 7, lines 45 to 48) the retention time is said to be preferably a period longer than the structure relaxation time at the retention temperature, especially preferably 1 to 24 hours. The board cannot derive from the passage on page 7, lines 43 to 48, that the time period for attaining the equilibrium state at a retention temperature  $< 1200$  K would be unlimited (or infinite). According to E9 the relaxation time for the "defect-line D2" is function of the retention temperature and the level of network-terminating impurities, e.g. the OH content (see page 3270, left-hand column, second paragraph). The respondent has provided no evidence that at retention temperatures  $< 1200$  K the time periods for attaining the equilibrium state are actually unreasonably long so that the preparation of the calibration curve for the silica glass of the examples of the patent in suit would be impossible or would represent an undue burden for the skilled person. Therefore the board cannot accept the respondent's contested allegation that it would be impossible for the skilled person to establish a calibration curve within the range 1073 K to 1200 K. Turning now to the passage mentioned above under (ii),

it does not prove that a calibration curve cannot be made between 1073 K and 1200 K, since the samples for establishing the calibration curve are not subjected to the annealing treatment described in this passage (slow cooling step from the retention temperature to the annealing completion temperature) but are quenched in liquid nitrogen after having reached the equilibrium state at the retention temperature. The statement on page 7, lines 53 to 55 clearly concerns the process as defined in claim 13 of the patent in suit and not the method used to draw up the calibration curve.

- 3.3 As pointed out by the respondent, the patent in suit does not disclose the geometry and dimensions of the samples to be used for drawing up the calibration curve. The appellant's representative indicated that samples of 60 mm in diameter and 10 mm in thickness were used. These dimensions are indeed stated on page 9, lines 42 to 44, of the patent in suit; however the samples having these dimensions are heated to the retention temperature, maintained at this temperature and then annealed under the conditions reported in Table 2. It cannot be directly inferred from the last paragraph on page 9 that samples having the same dimensions were used for drawing up the calibration curve. However, even if the use of such dimensions for samples to be subjected to a quenching step in liquid nitrogen were unreasonable as argued by the respondent, it could not be deduced therefrom that the skilled person would not be able to prepare a calibration curve on the basis of the indications in the patent. Assuming for the sake of argument that the skilled person would have encountered difficulties when using samples with the said dimensions (for example a non-uniform structure

throughout the glass sample), he would, after this first failure, have immediately known on the basis of the common general knowledge how to avoid further failures. He would have decreased the dimensions of the sample to be quenched in order to reduce the temperature difference within the glass samples during the quenching step. Therefore, the respondent's conclusion that the absence of information regarding the geometry or dimensions of the samples to be quenched resulted in an insufficiency of disclosure are also not convincing. In view of the preceding considerations, it is immediately apparent that the question whether or not the burden of proof rested on the appellant for his allegation concerning the dimensions of the sample has no bearing on the outcome of the decision about sufficiency of disclosure.

It follows from the above that the subject-matter of the patent meets the requirement of sufficiency of disclosure set out in Articles 83, 100(b) EPC.

4. It was disputed that the subject-matter of claim 1 complies with the requirement of novelty with respect to E8. This document discloses a high-purity transparent synthetic silica glass for use with ultraviolet laser such as a KrF laser or an ArF excimer laser, having a hydrogen molecule concentration of  $1 \times 10^{17}$  molecules/cm<sup>3</sup>. E8 further teaches that the Raman intensity ratio  $R_2$ , ie  $I_{606}/I_{800}$ , is smaller than 0.15 (see claims 1, 2, 5 and 13; page 4, lines 5 to 20 and 36 to 39; page 7, lines 4 to 6; pages 10 and 12, example 13). However E8 is completely silent about a SDT or about a quenching step in liquid nitrogen which is necessary to determine the SDT.



The respondent's arguments that the glass of claim 1 lacked novelty because  $R_2$  was a measurement of structural imperfections like the SDT and the range  $R_2 < 0.15$  encompassed the value  $R_2 = 0$  corresponding to no structural imperfection and thus lying within the claimed range of SDT, are not convincing for the following reasons. The fact that the range  $R_2 < 0.15$  encompasses the value zero does not in the present case mean that this value is disclosed. This is because  $R_2 = 0$  corresponds to an ideal glass containing no "D2 defects" (ie the defect associated with the Raman line  $606 \text{ cm}^{-1}$ ) as pointed out by the appellant and not contested by the respondent, and E8 neither expressly mentions this lower value nor discloses how this ideal silica glass without D2 defects could be produced. Therefore the range  $R_2 < 0.15$  does not destroy the novelty of the claimed glass.

The respondent further argued that the process as disclosed in example 13 of E8 would lead to a silica glass having a SDT lying within the claimed range. The board observes that the process disclosed in example 13 comprises heat treating the glass sample at  $1000^\circ\text{C}$  (ie  $1273 \text{ K}$ ) for 3 hours in an atmosphere of 90% argon and 10% hydrogen at the pressure of  $1000 \text{ kgf/cm}^2$ , then cooling the sample till  $800^\circ\text{C}$  ( $1073 \text{ K}$ ) at the slow cooling rate of about  $10^\circ\text{C/hr}$ , leaving the sample to cool down to  $500^\circ\text{C}$  while keeping the pressure at  $1000 \text{ kgf/cm}^2$ . This process differs from the process as defined in claim 13 of the patent in suit in that the sample is cooled at the slow cooling rate to  $1073 \text{ K}$  instead of  $1000 \text{ K}$  or lower. Furthermore it is not disclosed in example 13 that the starting glass has a

hydrogen molecule concentration of  $1 \times 10^{17}$  mol./cm<sup>3</sup> or more. As the process used in example 13 differs from the process of the patent in suit it cannot be concluded that it inevitably leads to a product falling within the SDT range stated in claim 1. The respondent has provided no evidence showing that the different process used in example 13 would inevitably lead to the claimed product. Instead the respondent argued that according to the patent in suit a time period of 1 to 24 hours was necessary for achieving the equilibrium state at a retention temperature of from 1073 K to 1700 K and that the silica glass obtained in example 13 could therefore be expected to have a SDT <1200 K when the sample was cooled for about 13 hours from 1200 K to 1073 K and thus maintained for about 13 hours at a temperature < 1200 K. These arguments are not convincing since the silica glass of example 13 was not maintained for about 13 hours at one **specific** retention temperature within the range 1073 K to 1200 K but was cooled at a slow cooling rate of about 10°C/hr from 1273 K to 1073 K. The respondent has not shown that an equilibrium state at a specific temperature between 1200 K and 1073 K can be reached if the glass is continuously cooled at the said cooling rate. For these reasons and in the absence of evidence from the respondent that the silica glass obtained by the process of example 13 actually exhibits a SDT of 1200K, the board considers that the claimed silica glass is new over the disclosure of E8. The claimed glass is also novel with respect to the other documents cited by the respondent. This was not in dispute so that further considerations are not necessary. Claims 7, 10 and 13, which are directed to an optical member, an exposure apparatus and the method of

preparation of the silica glass, either refer to the silica glass of claim 1 or state the hydrogen concentration and the SDT as defined in claim 1. Therefore, they also meet the novelty requirement.

5. The board concurs with the parties that E8 is the closest prior art, in particular example 13 thereof. The silica glass of E8 is used for producing optical members which are assembled in various apparatuses employing high power laser beams such as the excimer lasers, in particular the KrF and ArF excimer lasers. It has an absolute refractive index  $n_d$  of 1.460 or more, a hydrogen content of about  $5 \times 10^{16}$ , preferably  $\geq 1 \times 10^{17}$  molecules/cm<sup>3</sup>, and intensity ratios  $R_1$  (Raman intensity ratio  $I_{495}/I_{800}$ ) and  $R_2$  smaller than 0.48 and 0.15 respectively. The optical members produced from this glass are durable against high-power laser irradiation, high-power light exposure or ionizing irradiation for a long period of time (see claims 1, 2, 5 and 13; page 2, lines 5 to 9; page 3, lines 25 to 28; page 3, line 53 to page 4, line 4; page 4, lines 33 to 46; page 7, lines 4 to 6; Table 1).

Starting from this prior art, the technical problem to be solved by the patent in suit can be seen in providing a silica glass suitable for use as an optical member, which has an improved resistance to ultraviolet light, in particular a silica glass in which the generation of structural defects such as E'-centers and NBOH-centers is inhibited, the increase of refractive index and the occurrence of dents and strain being sufficiently inhibited even after irradiation with UV-light of short wavelength and high output or excimer

laser beam for a long time (see page 3, lines 5 to 8 of the patent in suit).

It is proposed to solve this problem by the silica glass as defined in claim 1 which has a STD of 1200 K or lower and a hydrogen molecule concentration of  $\geq 1 \times 10^{17}$  molecules/cm<sup>3</sup>. In view in particular of the examples and comparative examples of the patent in suit which show that a silica glass having a hydrogen content and/or a STD lying inside the claimed ranges exhibit improved properties (increment of refractive index, amount of surface dents, maximum amount of strain, absorption coefficient) compared to a silica glass whose hydrogen content and/or STD are outside the claimed ranges, it is credible that the problem stated above has actually been solved. The respondent argued that the values for the increment of refractive index, amount of surface dent and maximum amount of strain in comparative example 3 are identical to those of example 12 (an example according to the invention) although the glass of comparative example 3 has both a STD and a hydrogen molecule content falling outside the claimed ranges. In comparative examples 1 and 2 having a STD < 1200 K the values for the increment of refractive index were even better than in example 12. These arguments cannot challenge the preceding conclusion because they are based on a comparison of glasses which in fact are not comparable, the glass of example 12 containing an amount of chlorine of 150 ppm whereas the glasses of the comparative examples 1, 2 and 3 contain only 8 ppm chlorine. As pointed out by the appellant example 12 should be compared with comparative example 8 whose chlorine content is also

150 ppm since high chlorine concentrations affect negatively the excimer laser resistance.

5.1 Document E8 itself deals with the problems of degradation and durability arising when silica glasses are irradiated with high-power laser beams for a long period of time. E8 addresses the problem of breakage of bonds between silicon and oxygen atoms, formation of the E'-centers and NBOH-centers. According to E8 when the silica glass has not reached the equilibrium state during the fabrication process an unstable structure containing 3-member rings and 4-member rings is produced, which is measured by the Raman intensity ratios  $R_1$  and  $R_2$ . These unstable structures are believed to contribute to the decrease of durability against laser irradiation. The formation of these unstable rings is decreased by subjecting the silica glass to a heat treatment under a high pressure atmosphere of  $1000 \text{ kgf/cm}^2$ , which increases the absolute refractive index to  $> 1.460$ . The desired durability against laser irradiation is achieved with a glass having an absolute refractive index  $n_d$  of  $\geq 1.460$  at 589 nm and further containing hydrogen at a concentration of  $\geq 5 \times 10^{16}$ , preferably  $\geq 1 \times 10^{17}$  molecules/cm<sup>3</sup>. By increasing the absolute refractive index to a level of at least 1.460 the  $R_1$  and  $R_2$  values can be reduced to  $R_1 < 0.55$  and  $R_2 < 0.17$ , preferably  $R_1 < 0.48$  and  $R_2 < 0.15$  (see page 2, lines 51 to 55; page 3, line 25 to page 4, line 46; page 6, lines 45 to 48). Thus it can be inferred from E8 that lower intensity ratios  $R_1$  and  $R_2$  lead to a glass having a better durability against laser irradiation and that the lower intensity ratios can be achieved by increasing the absolute refractive index to the level indicated above. Assuming that the skilled person

confronted with the problem stated above would have contemplated decreasing  $R_2$  to values lower than those disclosed in Table 1 ( $R_2=0.10$  is the lowest value), then he would have been confronted with the problem of how  $R_2$  could be decreased. E8 suggests increasing the absolute refractive index, in particular by treating the glass ingot under a high-pressure atmosphere. Another suggestion as to how  $R_2$  might be decreased is not disclosed in E8. However the respondent has not shown that the skilled person would arrive at a product falling within the definition of claim 1 if he had followed this direction. The board further observes that E8 contains no information which could point towards the SDT parameter or the quenching step in liquid nitrogen, which is necessary to establish the calibration curve as explained above. Therefore the respondent's argument that the claimed silica glass was obvious in view of the teaching of E8 alone cannot be followed by the board.

5.2 E9 is a scientific article published about 8 years before the filing date of E8 and which discloses studies of the first-order Raman spectra of vitreous  $\text{SiO}_2$  as a function of the sample fictive temperature  $T_F$  for  $T_F$  values from  $900^\circ\text{C}$  to  $1550^\circ\text{C}$  (see page 3266, abstract). E9 does not deal with the problem of improving the resistance of the silica glasses to irradiation with ultraviolet light, in particular with excimer laser beams, for a long time. E9 is silent about the hydrogen molecule content of the studied silica glasses which was clearly not considered as an important parameter. According to E9 the intensities of the defect-lines D1 and D2, which are assigned to the four- and threefold ring defects, increase with

increasing the fictive temperature (see page 3267, left-hand column, 1st paragraph). The respondent's arguments that the skilled person would have combined the teaching of E8 and E9, and would thus have arrived without inventive skill at a glass falling within the definition of claim 1 are not convincing for the following reasons. Although E9 teaches that the intensity of the defect-line D2, and thus the normalised intensity  $R_2$ , can be decreased by decreasing the fictive temperature, the board observes that the silica glasses of E9 do not have to fulfil any additional requirement concerning the amount of hydrogen molecules and the refractive index. Neither E8 nor E9 contains information suggesting that by decreasing the heating temperature to values lower than 1000°C (the lowest limit disclosed in E8) the features considered to be essential in E8 for achieving the desired durability against high-power laser irradiation would still be obtainable. Furthermore, the method described in E9 for studying the influence of  $T_f$  on the defect-lines D1 and D2, namely the heat treatment at the fictive temperature until the equilibrium state is reached and the subsequent rapid quenching to room temperature, is very different from the process described in example 13 of E8 for obtaining a glass suitable as an optical member for use with high-power laser beams and which has to fulfil severe optical requirements. The latter process includes in particular a very slow and careful cooling step while maintaining the glass under high pressure, which is totally in contradiction with the rapid quenching to room temperature disclosed in E9. The skilled person would have expected that performing a rapid quenching to room temperature instead of the slow and careful annealing

of E8 would lead to an optical member not satisfying the required severe optical properties. Under these circumstances the board cannot follow the respondent's argument that the skilled person would have decreased the temperature of the heat treatment to 900°C in the process of E8 and then rapidly quenched the glass in water in order to reduce the R<sub>2</sub> ratio and solve the problem stated above.

- 5.3 E4 deals with the problem of improving the resistance of silica glass optical members to optical deterioration upon exposure to a high-power ultraviolet laser beam. The solution disclosed in E4 involves making a high-purity silica glass optical member which is free from striae in at least one direction corresponding to the incident light and which contains OH groups in an amount of at least 50 wt. ppm and sufficient hydrogen to inhibit decreases in light transmittance due to exposure to said ultraviolet light, the hydrogen content being preferably at least  $5 \times 10^{16}$  mol./cm<sup>3</sup> (see page 4, lines 43 to 46; page 5, lines 6 to 11; claims 1, 2 and 26. This document which is also silent about the SDT parameter or a quenching step in liquid nitrogen contains no information pointing to the claimed subject-matter. The same remark applies to E5 which discloses an optical member made of a synthetic silica glass having a hydrogen molecule content  $\leq 1 \times 10^{16}$  molecules/cm<sup>3</sup>, a OH group-content from 10 to 100 ppm, a chlorine content < 200 ppm in order to avoid a reduction of the ultraviolet transmission when the optical member is irradiated with ultraviolet rays, e.g. excimer lasers (see column 3, lines 44 to 59; column 4, lines 48 to 56). The remaining documents cited by the opponent are less relevant and contain no



additional information which, in combination with the preceding documents, could render the claimed subject-matter obvious.

5.4 It follows from the above that the respondent's arguments concerning inventive step could not convince the board that the silica glass according to claim 1 does not involve an inventive step. The subject-matter of this claim thus meets the requirement of inventive step set out in Articles 52(1) and 56 EPC.

6. Claims 7 and 10 concern an optical member and an exposure apparatus comprising a silica glass according to claim 1. Claim 13 is directed to a method for producing a silica glass having both the SDT and the hydrogen content stated in claim 1. Therefore they derive their patentability from that of claim 1. Claims 1, 7, 10 and 13 being allowable, the same applies to the dependent claims appended thereto.

**Order**

**For these reasons it is decided that:**

1. The decision under appeal is set aside.
2. The patent is maintained as granted.

The Registrar:

The Chairman:



U. Bultmann



R. Spangenberg

MES

1987.D

