

**High-purity silica powder, process and apparatus for
producing it**

5 The invention relates to a high-purity silica powder and to a process and apparatus for producing it in a hot zone.

High-purity silica powders are employed in numerous
10 technical fields. Examples of application areas include optical fibers, quartz crucibles for pulling silicon single crystals, optoelectronics (e.g. lenses and mirrors), fillers in passive components used in electronics, and polishing suspensions for wafers
15 (chemical mechanical polishing). A high powder purity is required for the abovementioned applications.

In optical fibers made from SiO_2 for optical communications, the radiation intensity of the light as
20 information carrier should not be reduced by absorption caused by impurities, such as OH, iron and copper, or by scattering caused by bubbles, crystallization nuclei and inhomogeneities. Crystallization nuclei are formed by impurities such as calcium and magnesium.

25 In quartz glass crucibles, corrosion to the inner surface of the crucible occurs during the process of pulling silicon single crystals as a function of the number and type of impurities. Corrosion reduces the potential
30 pulling time. Moreover, each additional impurity increases the number of nuclei at which oxygen precipitations may form during cooling of the single crystal.

35 In optical glasses, by way of example, sodium and transition metals are responsible for transmission losses

in the glass. Therefore, it is necessary for the concentration of the transition metals not to exceed 100 ppb. Only this can ensure that the transmission for a wavelength of 248 nm is greater than 99.5% and for a wavelength of 193 nm is greater than 98%. Moreover, silica powders for optical fibers, quartz crucibles and glasses must be free of organic impurities, since otherwise numerous bubbles may form during the sintering step.

10

High-purity SiO_2 can also be used as a filler in epoxy resins for protecting IC chips if the concentration of the elements iron, sodium and potassium does not exceed 0.2 ppm and the concentration of the elements aluminum and titanium does not exceed 1 ppm. These elements change the coefficient of thermal expansion, the electrical conductivity and the corrosion resistance of the passive components. This can deactivate the chip protection function.

20

Polishing suspensions are used for direct polishing of semiconductor surfaces. The SiO_2 used for this purpose must not, for example, in the case of aluminum, exceed a concentration of 4 ppm.

25

A known process for producing high-purity silica powders is the hydrolysis of silicon-containing precursors. This may, for example, be carried out by SiCl_4 being hydrolyzed in water in the presence of an organic solvent (Degussa DE 3937394), by ammonium fluorosilicate being mixed firstly with ammonia water and then with hydrofluoric acid (Nissan, JP 04175218), or by silica being precipitated by the addition of a dilute mineral acid to an alkali metal silicate (Nippon, EP 9409167, University of Wuhan, CN 1188075). The silica formed is also known as precipitated silica and is used primarily as a catalyst

35

support and as an epoxy resin filler for protecting LSI and VLSI circuit devices. The abovementioned processes produce porous, bubble-containing imperfect spherical particles with poor flow properties. A further very significant drawback is that these processes are subject to purity limits, since certain impurities, such as OH, C, F, N and alkali metals, such as Na and K, are to a certain extent introduced by the process. These drawbacks lead to considerable light scattering and absorption and to a reduced mechanical and thermal stability of the application product. Therefore, this process is fundamentally unsuitable for use in the optical fiber, crucible and glass sectors.

Natural quartz is also ruled out for the above applications on account of the strict purity requirements. However, there has been no lack of attempts to achieve acceptable purity levels by the additional process step of further purification of insufficiently pure quartz. According to DE 3123024 (Siemens), natural quartz is converted into thin fibers by melting, and then these fibers are subjected to a plurality of leaching process steps using acids and bases. On account of the high surface area and small thickness of the fibers, the level of transition metal ions can be reduced to less than 1 ppm. This process is inexpensive, since the fibers are used directly for applications in the optical fibre sector. If, for further applications and shaped body geometries, in accordance with DE 3741393 (Siemens), the purified fibers are milled, converted into a slip with the aid of water, dispersants and other auxiliaries, and then a slip casting process and finally a sintering process are carried out, the ultimate result is a complex process with numerous contamination sources.

According to EP 0737653 (Heraeus), natural quartz is subjected to the process steps of milling, screening, preheating to 1000°C, treatment with Cl_2HCl , cooling and desorption. This time-consuming process gives purities of
5 around 70 ppb with regard to Fe. Impurities derived from alkaline-earth metals and Al, which are known to form cristobalite and therefore, for example, reduce the crucible quality, cannot be removed to this extent, since these elements form chlorides of low volatility
10 (previously: Na = 1100 ppb, K = 1050 ppb, Li = 710 ppb, Ca > 370 ppb, Al = 16 000 ppb, Fe = 410 ppb; subsequently: Na < 10 ppb, K > 80 ppb, Li = 700 ppb, Ca > 120 ppb, Al = 16 000 ppb, Fe > 30 ppb).

15 According to US 4818510 (Quartz Technology), quartz can be purified further using HF. However, HF only reacts selectively with certain elements, such as iron, with which it forms readily soluble complexes.

20 The further purification was also carried out on SiO_2 granules. According to US 6180077 and EP 1088789 (Heraeus), SiO_2 granules are produced and are purified at high temperatures by means of HCl. One advantage is that the granules have a high surface area and can therefore
25 be acted on more easily and more quickly by HCl. If the starting point is granules with a purity of Na < 50 ppb, Fe = 250 ppb, Al < 1 ppm, the further purification makes it possible to achieve very high purity levels (Na = 5 ppb, Fe = 10 ppb, Al = 15 ppb). One disadvantage
30 is that it is firstly necessary to produce highly porous silica granules (pore volume 0.5 cm^3 , pore diameter 50 nm, BET 100 m^2/g , density 0.7 g/cm^3 , granule size 180 - 500 μm), which is a time-consuming process, and these granules do not yet represent the finished products, but
35 rather, still have to be sintered. Furthermore, the high porosity conceals the latent risk of gases remaining

included during sintering following shaping, for example to form a crucible.

According to US 4956059 (Heraeus), in addition to the
5 purification gases Cl_2/HCl used at high temperatures, an electric field (typically 652 V/cm) can also be used in the further purification of silica granules. The further purification effect is stronger in the presence of the electric field, with in particular the alkali metal ions
10 which migrate well in the electric field, being affected by this. This method makes it possible to reduce the sodium level, for example from 1 ppm to 50 ppb.

According to EP 1006087 (Heraeus), the further
15 purification can be carried out in such a manner that impure powder is heated in a gas stream, with the impurities softening and forming molten agglomerates, and the powder then being guided on to an impact surface, to which only the impure molten agglomerates adhere. This
20 method only makes sense for very impure starting material powders. However, further purification with regard to high-melting oxides, such as MgO and Al_2O_3 is not possible in this way. The high quantities of gases required for this purpose represent a further drawback.

25 High purities (metal impurity levels < 1 ppm, $\text{C} < 5$ ppm, $\text{B} < 50$ ppm, $\text{P} < 10$ ppb) are achieved using the sol-gel process, in which first of all a sol and then a gel are formed from an organic silane and water. This is followed
30 by process steps of drying, calcining using inert gas and sintering (Mitsubishi, EP 0831060, EP 0801026, EP 0474158). The process is very time-consuming and is also expensive, since high-purity organosilanes act as starting materials. In general, an organic-based
35 rheological auxiliary, a dispersant and a solvent are used for the production process, with the result that the

finished product may contain black C particles and CO and CO₂ bubbles. The use of water leads to a high OH content and consequently to the formation of bubbles in the product and to the product having a low thermal stability. If this material is used in the form of silica crucibles for the production of Si single crystals using the Czochralski process, the bubbles and pores expand on account of the high temperature and the reduced pressure. During the pulling process, bubbles are responsible not only for turbulence in the silicon melt but also for the formation of crystal defects and a deterioration in the long-term stability of the crucible.

In principle, high-purity silica is also produced by precipitation of silica from high-purity organosilanes or SiCl₄ in the presence of an oxy-fuel flame using the CVD or OVD process (Corning, US 5043002, US 5152819, EP 0471139, WO 01/17919, WO 97/30933, WO 97/22553, EP 0978486, EP 0978487, WO 00/17115). However, this process does not produce powders, but rather glass bodies having a defined, simple geometry. The simple geometries include optical glasses and lenses. Optical fibers can be obtained from the glass body produced in high-purity form by drawing. To produce glass bodies of any desired geometry from the simple glass bodies, the glass body produced must first of all be milled to form a powder, then dispersed, shaped and sintered. However, this can entail widespread contamination in particular during the milling step.

A further drawback of this process is that expensive, high-purity organosilanes, such as, for example, octamethylcyclotetrasiloxane (OMCTS), are used to achieve particularly high purities.

High-purity SiO_2 layers can also be produced by deposition on high-purity substrates (e.g. by plasma CVD/OVD, GB 2208114, EP 1069083). One drawback is that it is only possible to achieve low deposition rates of 150 nm/min (e.g. J.C. Alonso et al., J.Vac.Sci.Technol. A 13(6), 1995, pp. 2924 ff.). Coating processes entail high production costs. High purity silica powders are not obtainable by these processes.

- 10 A simple alternative process is the formation of silica in a flame. Two different approaches are known in this respect. According to JP 5-193908 (Toyota/ShinEtsu), high-purity silicon metal powder can be oxidized to form high-purity silica powder by means of a $\text{C}_3\text{H}_{2n+2}/\text{O}_2$ flame, 15 the $\text{C}_3\text{H}_{2n+2}$ being required only for ignition. However, the inventors themselves acknowledge the problem that the reaction produces a large number of unburnt particles. Full oxidation is difficult to realize unless the starting particles are very fine ($0.2 \mu\text{m}$). However, it is 20 in turn almost impossible to produce such fine Si particles in a highly pure form.

Alternatively, fumed silica can be produced from SiCl_4 in an oxyhydrogen flame in a first step by flame hydrolysis 25 and this fumed silica can be converted into fused silica by sintering in a second step.

The term fumed silica is to be understood as meaning ultrafine-particle, nanoscale powders which are produced 30 by reacting silanes in a high-temperature flame and are often greatly aggregated and agglomerated. One typical example of fumed silica is Aerosil® OX 50 produced by Degussa, with a BET surface area of $50 \text{ m}^2/\text{g}$. The term fused silica is to be understood as meaning coarser- 35 grained, spherical glass powders. One typical example of

fused silica is Excelica® SE-15 produced by Tokuyama with a mean particle size of 15 µm.

5 According to US 5,063,179 (Cabot), the second substep (= the production of fused silica) is implemented by fumed silica being dispersed in water, filtered, dried, purified further using SOCl_2 or Cl_2 and being sintered in a furnace. The concentrations of the impurities, such as Na and Fe, are then around 1 ppm (total content of
10 impurities < 50 ppm), i.e. still rather high.

According to JP 59152215 and JP 5330817 (Nippon Aerosil), in the second substep (= the production of fused silica), the fumed silica powder is transferred in dispersed form,
15 for example directly by means of a screw conveyer, into an oxyhydrogen flame and sintered to form fused silica powder.

According to JP 5301708 and JP 62-270415 (Tokuyama), to
20 produce the fused silica, high purity fumed silica is treated with H_2O vapor, cooled, fluidized and fed by means of a screw conveyer to an oxyhydrogen flame for the purpose of sintering.

25 The fused silica product obtained using the abovementioned processes contains > 1000 ppb of impurities, as a cumulative sum of the elements Cu, Fe, Ti, Al, Ca, Mg, Na, K, Ni, Cr, Li. The dispersion and conveying of the fumed silica particles in accordance
30 with the abovementioned processes is carried out, for example, with the aid of a screw conveyer. The screw is a moving part which becomes worn through contact with silica, in particular in the region of the edges. As a result, the screw contaminates the silica powder. Other
35 components of the installation are also exposed to the abrasive silica particles and therefore to heavy wear.

Mention should be made in particular of the burner nozzle, in which the velocities of the silica powders are particularly high.

- 5 It was an object of the present invention to provide a silica powder of very high purity.

A further object of the present invention was to provide a process and apparatus for the inexpensive production of
10 the powder according to the invention.

The first object is achieved by a silica powder in which the sum of impurities is less than 500 ppb.

- 15 It is preferable for the total amount of impurities in the silica powder according to the invention to be less than 150 ppb. Particularly preferably, the sum of impurities is less than 150 ppb and the individual impurity levels are Cu < 1 ppb, Fe < 25 ppb, Ni < 2 ppb,
20 Cr < 2 ppb, Ti < 3 ppb, Al < 31 ppb, Ca < 65 ppb, Mg < 12 ppb, Na < 12 ppb, K < 6 ppb, and Li < 1 ppb and the powder is carbon-free.

The impurity levels are determined using the ICP analysis
25 method (inductively coupled plasma, appliance type ICP-MS HP4500), and the detection limit of the method is less than 1 ppb. The silica powders may be either fumed silica or fused silica.

- 30 The fumed silica particles preferably have a BET surface area of between 50 and 300 m²/g, particularly preferably between 150 and 250 m²/g. The primary particle size is between 1 nm and 1000 nm, preferably between 5 nm and 100 nm, very particularly preferably between 10 nm and
35 30 nm.

The fused silica powder preferably has a mean particle size of between 100 nm and 200 μm , particularly preferably between 1 μm and 200 μm , very particularly preferably between 5 μm and 40 μm . Furthermore the powder
5 preferably has a narrow particle size distribution, with $D(95) - D(5) < 50 \mu\text{m}$, particularly preferably $D(95) - D(5) < 35 \mu\text{m}$, e.g. with a mean particle size of $D(50) = 15 \mu\text{m}$: $D(5) = 1 \mu\text{m}$, $D(95) = 50 \mu\text{m}$, particularly preferably $D(5) = 3 \mu\text{m}$, $D(95) = 35 \mu\text{m}$ (measured using
10 CILAS 715).

The narrow particle size distribution of the product according to the invention means that additional process steps, such as screening, are not required and the powder
15 is directly suitable for further processing. Fig. 6 shows, by way of example, the very uniform particle size distribution of a fused silica powder which has been produced in accordance with Example 4.

20 The fused silica particles of the powder according to the invention preferably have a spherical morphology and are completely vitrified. Unlike powders produced using the sol-gel process, they do not include any bubbles or carbon impurities originating from the use of organic
25 solvents, dispersants and rheological agents.

The high-purity fumed silica and fused silica powders according to the invention can be used for all applications which are known from the prior art. They are
30 preferably suitable for the production of shaped bodies as described, for example, in DE 19943103 (Wacker Chemie GmbH).

A powder according to the invention can preferably be
35 produced by means of a process in which a high-purity fumed silica powder is obtained by hydrolysis of a high-

purity SiCl_4 , wherein the hydrolysis of the SiCl_4 to form the fumed silica powder is carried out in an apparatus having a metal-free surface. The hydrolysis of the high-purity SiCl_4 is carried out in a flame. The flame
5 comprises an oxygen-containing gas and a gas selected from the group consisting of hydrocarbon and hydrogen, preferably comprises air or oxygen and a gas selected from the group consisting of methane, propane and hydrogen, particularly preferably comprises oxygen and
10 hydrogen. The hydrolysis preferably takes place in an H_2/O_2 flame. Alternatively, the hydrolysis is carried out in a plasma, for example in an HF plasma.

It is preferable for the deposition of the fumed silica
15 powder also to be carried out in an apparatus with a metal-free surface.

Other suitable starting materials include silanes, organosilicon compounds and halosilanes with an impurity
20 level of < 100 ppb. SiCl_4 with an impurity level of < 100 ppb is preferably suitable and SiCl_4 with a purity as given in Table 1 is particularly preferably suitable.

A likewise high-purity fused silica powder in accordance
25 with the invention can be produced from the fumed silica powder in accordance with the invention by sintering.

The sintering of the high-purity fumed silica powder is preferably carried out in an apparatus as used to produce
30 the fumed silica powder according to the invention, in an H_2/O_2 flame or by means of an HF plasma. A controlled quantity of water can also be added to the fumed silica to control the particle sizes of the fused silica powder.

To avoid contamination from environmental elements, such as Na, K, Mg or Ca, it is preferable to work under clean room conditions and/or under a lamina flow.

- 5 The process is in this case carried out under clean room conditions from classes 100 000 to 1, preferably 10 000 to 100, particularly preferably 1000.

As an alternative to clean room conditions, the process
10 can be carried out at a pressure of between 0.913 bar and 1.513 bar, preferably between 1.013 bar and 1.413 bar, particularly preferably between 1.020 bar and 1.200 bar. The superatmospheric pressure prevents impurities from entering the installation.

15 If the powder according to the invention is produced in an H_2/O_2 flame, the apparatus according to the invention is a nozzle comprising an inner tube located in an outer tube, with a starting material selected from the group
20 consisting of $SiCl_4$, a mixture of $SiCl_4$ with O_2 , fumed silica and a mixture of fumed silica with O_2 being passed through the inner tube, wherein the inner tube consists of a silicon-containing material with silicon as the main constituent, such as for example quartz glass, fused
25 quartz, SiC , Si_3N_4 , enamel or silicon metal.

It is preferable for the inner tube to consist of a silicon-containing material with silicon as the main constituent, such as for example quartz glass, fused
30 quartz, SiC , Si_3N_4 , enamel or silicon metal, with the surface of the material having been purified using a chlorine-containing gas, such as, for example, $SOCl_2$, HCl or Cl_2 .

35 The apparatus is particularly preferably a nozzle in which the inner tube consists of quartz glass or a

material with a quartz glass surface, which has particularly preferably been purified using a chlorine-containing gas, such as, for example, SOCl_2 , HCl or Cl_2 .

5 It is particularly preferable for the entire nozzle to consist of quartz glass or a material with a quartz glass surface. The purity can be increased still further if the quartz glass or the material with the quartz glass surface has been purified using, for example, SOCl_2 , HCl
10 or Cl_2 .

If only the inner tube for the supply of fumed silica or SiCl_4 consists of quartz glass, while the remainder of the nozzle consists, for example, of steel, the purity of the
15 powder produced is slightly worse than with a nozzle made from quartz glass, but is still higher than in the case of the known silica powders.

Therefore, the invention also relates to a nozzle
20 comprising an inner tube located in an outer tube, wherein the inner tube consists of a silicon-containing material with silicon as the main constituent. This material is preferably selected from the group consisting of quartz glass, fused quartz, SiC , Si_3N_4 , enamel or
25 silicon metal.

It is preferable for the nozzle to consist of a material selected from the group consisting of quartz glass, fused quartz, SiC , Si_3N_4 , enamel or silicon metal, particularly
30 preferably of quartz glass.

The nozzle is preferably a nozzle without premixing of the fuel gases. In a nozzle of this type, the fuel gases H_2 and O_2 are fed to the combustion chamber separately. In
35 one embodiment of the nozzle according to the invention, SiCl_4 and/or fumed silica are premixed with one of the

fuel gases, preferably with O_2 , in a pilot chamber, and the mixture is then fed to the combustion chamber. The nozzle comprises an inner tube for supplying the mixture of O_2 and fumed silica ($SiCl_4$) and an outer tube for
5 supplying H_2 (Fig. 4).

In another embodiment of the nozzle according to the invention, all the reaction partners (H_2 , O_2 , $SiCl_4$ and/or fumed silica) are fed to the combustion chamber
10 separately. The nozzle comprises concentrically arranged tubes for the supply of fumed silica ($SiCl_4$), O_2 and H_2 . One possible arrangement comprises an inner tube for the supply of fumed silica ($SiCl_4$), a middle tube for the supply of O_2 and an outer tube for the supply of H_2
15 (Fig. 1).

It is preferable for a burner for producing the powder according to the invention by means of H_2/O_2 flame to comprise a plurality of the nozzles according to the
20 invention. The burner delivers a powder with a narrow particle size distribution in the case of one nozzle (Fig. 1), a particularly narrow particle size distribution with a plurality of nozzles in which the starting materials are supplied through three concentric tubes
25 (Fig. 2), and a very particularly narrow particle size distribution with a plurality of nozzles and an O_2 /fumed silica premixing chamber with the starting materials being supplied through two concentric tubes (Fig. 3). This arrangement allows a particularly homogeneous
30 distribution of the $SiCl_4$ (or of the fumed silica powder when producing fused silica powder) in the flame.

Therefore, the invention also relates to a burner which includes 1 to 30, preferably 6 to 13, particularly
35 preferably 7 nozzles according to the invention. That surface of the burner which faces the combustion chamber

preferably likewise consists of quartz glass. A burner with 7 nozzles of this type, according to the invention, is illustrated in Fig. 4. Fig. 3 diagrammatically depicts a plan view of a burner of this type. Figure 2
5 diagrammatically depicts a plan view of a burner with 7 nozzles in which all 3 starting materials, as described above, are guided separately into the combustion chamber.

The dispersion of the fumed silica in the flame is
10 improved still further in the variant of the nozzle according to the invention in which O_2 and fumed silica powder are premixed before being fed to the combustion chamber.

15 If the powder according to the invention is produced in a plasma, the apparatus according to the invention is a plasma torch comprising a powder nozzle, an intermediate tube and an outer tube (Fig. 4), with the powder nozzle, the intermediate tube and the outer tube having a surface
20 made from a silicon-containing material with silicon as the main constituent. It is preferable for the surface to consist of a material selected from the group consisting of quartz glass, fused quartz, SiC , Si_3N_4 , enamel or silicon metal. It is preferable for the surface to be
25 purified using a gas, such as $SOCl_2$, Cl_2 or HCl . $SiCl_4$ or the fumed silica powder is metered in via the powder nozzle, the plasma gas O_2 is metered in via the intermediate tube and the shrouding gas mixture O_2 and H_2 is introduced via the outer tube.

30

It is particularly preferable to use a plasma torch in which the powder nozzle, the intermediate tube and the outer tube have a surface made from quartz glass.

35 It is especially preferable for the plasma torch to have a surface made from quartz glass.

High-purity powders can be produced directly using the apparatuses according to the invention. The further purification process step which is usually required is avoided. Fumed and fused silica powders of extremely high purities (Table 1), which have not been achieved using conventional processes, can be produced using a nozzle according to the invention. The purity can be increased still further by combustion in a nozzle made from quartz glass under clean room conditions. Furthermore, it is advantageous if all the surfaces of the installation for producing the fumed or fused silica powder according to the invention which come into contact with a starting material in powder form, or the product according to the invention, are designed to be free from contamination. Therefore, an apparatus for producing a silica powder according to the invention is preferably distinguished by the fact that all the surfaces that come into contact with the silica powder are metal-free. An installation for producing a silica powder is known to comprise a) a metering apparatus, b) a burner, c) a combustion chamber, d) a cyclone and e) a silo. In the case of fumed silica production, a fluidized bed is generally also connected between the cyclone and the silo.

The materials which have been mentioned for the nozzle according to the invention, preferably also form the surface of the metering, the combustion chamber, the cyclone, the fluidized bed and the silo. In another embodiment, the metering apparatus and the silo may also have a pure plastic surface. The plastics may, for example be PFA (perfluoroalkoxy copolymer), PTFE (polytetrafluoroethylene), Halar® E-CTFE, GFP (glass fiber-reinforced polyester resin) and PP (polypropylene). In the metering region, it is preferable for the silica powders to be conveyed without moving parts, for example to use pneumatic conveying by means of compressed air.

In the drawing:

5 Fig. 1: shows the burner outlet as a 3-tube burner nozzle without premixing of O_2 with $SiCl_4$ or fumed silica,

10 Fig. 2: shows the burner outlet comprising 7 nozzles without premixing of O_2 with $SiCl_4$ or fumed silica,

Fig. 3: shows the burner outlet comprising 7 nozzles with premixing of O_2 with $SiCl_4$ or fumed silica,

15 Fig. 4: shows the burner comprising 7 quartz glass nozzles with premixing of O_2 with $SiCl_4$ or fumed silica,

Fig. 5: shows the plasma torch,

20 Fig. 6: shows fused silica powder from Example 4.

The following examples serve to further explain the invention.

25 Example 1: Production of a fumed silica powder from $SiCl_4$ by means of oxyhydrogen flame without clean room conditions

30 To produce a fumed silica powder from $SiCl_4$, the reaction partners $SiCl_4$, O_2 and H_2 are passed into the combustion chamber by means of a quartz glass nozzle without premixing. The reaction is carried out using 16.6 g/min of $SiCl_4$ + 6.3 l/min of O_2 + 8.9 l/min of H_2 . The combustion chamber is operated at a pressure of 20 mbar
35 above atmospheric pressure. Table 1 shows the analytical results.

Example 2: Production of a fumed silica powder from SiCl_4 by means of oxyhydrogen flame using clean room conditions

To produce a fumed silica powder from SiCl_4 , the reaction
5 partners SiCl_4 , O_2 and H_2 are passed into the combustion
chamber by means of a quartz glass nozzle without
premixing. The reaction is carried out using 16.6 g/min
of SiCl_4 + 6.3 l/min of O_2 + 8.9 l/min of H_2 . The entire
installation is in a clean room belonging to clean room
10 class 10 000. Table 1 shows the analytical results.

Example 3: Production of a fused silica powder from a
fumed silica powder by means of oxyhydrogen flame without
clean room conditions

15 To produce fused silica powder from fumed silica powder,
the reaction partners fumed silica, O_2 and H_2 are passed
into the combustion chamber by means of a quartz glass
nozzle without premixing. The reaction is carried out
20 using 180 l/min of H_2 + 90 l/min of O_2 + 60.3 g/min of
fumed silica powder. The combustion chamber is operated
at a pressure of 40 mbar above atmospheric pressure.
Table 1 shows the analytical results.

25 Example 4: Production of fused silica powder from fumed
silica powder by means of oxyhydrogen flame under clean
room conditions

To produce fused silica powder from fumed silica powder,
30 the reaction partners fumed silica powder, O_2 and H_2 are
passed into the combustion chamber by means of a quartz
glass nozzle with premixing. The reaction is carried out
using 180 l/min of H_2 + 90 l/min of O_2 + 60.3 g/min of
fumed silica powder. The entire installation is in a
35 clean room belonging to clean room class 10 000. Table 1
shows the analytical results.

Example 5: Production of fused silica powder from fumed silica powder by means of HF plasma under clean room conditions

5

To produce fused silica powder from fumed silica powder, the reaction partners fumed silica powder, air and H₂ are passed into the combustion chamber via a torch comprising quartz glass cylinders. The reaction is carried out using
10 45 l/min of O₂ as the central plasma gas, 90 l/min of O₂ and 25 l/min of H₂ as the shrouding gas and 15 kg/h of fumed silica powder, metered in via the powder nozzle. The pressure in the combustion chamber is 300 torr, and the total power of the HF plasma is 90 kW. In the present
15 case, the plasma is an HF plasma in accordance with the principle of solid state technology, with which the person skilled in the art will be familiar. The entire installation is in a clean room belonging to clean room class 10 000. Table 1 shows the analytical results.

20

Example 6: Production of fused silica powder from fumed silica powder by means of oxyhydrogen flame under clean room conditions using standard nozzle, not made from quartz glass

25

To produce fused silica powder from fumed silica powder, the reaction partners fumed silica powder, O₂ and H₂ are passed into the combustion chamber by means of a stainless steel nozzle with premixing. The reaction is
30 carried out using 180 l/min of H₂ + 90 l/min of O₂ + 60.3 g/min of fumed silica powder. The entire installation is in a clean room belonging to clean room class 10 000. Table 1 shows the analytical results.

Comparative example 7: Production of fused silica from fumed silica by means of oxyhydrogen flame in accordance with patent JP 59152215

- 5 The high-purity fumed silica powder is passed into an oxygen stream via a screw conveyer and then passed into the burner tube. The burner comprises 3 tubes, with 7.6 m³/h of H₂ being introduced into the combustion chamber via the inner and outer tubes, while the middle
- 10 tube contains 3.8 m³/h of O₂ and 1.8 kg/h of fumed silica powder. Table 1 shows the analytical results.

Table 1

Impurity levels in the product produced in the respective examples and of the SiCl_4 used, in ppb, determined using ICP/MS.

5

Ex.	Cu	Fe	Ti	Al	Ca	Mg	Na	K	Ni	Cr	Li
1	<1	22	2	24	54	9	8	5	2	2	<1
2	<1	10	<1	10	11	2	4	1	<1	<1	<1
3	<1	25	2	31	64	11	11	5	2	2	<1
4	<1	10	<1	9	13	3	5	1	<1	<1	<1
5	<1	12	<1	15	14	3	6	1	<1	<1	<1
6	<1	250	4	63	15	7	7	2	43	27	<1
7	4	730	<1	62	66	134	19	9	167	235	<1
SiCl_4	<1	10	<1	3	8	<1	3	2	<1	<1	<1

Patent claims

1. A silica powder in which the sum of impurities is
5 less than 500 ppb.
2. A silica powder in which the sum of impurities is less than 150 ppb.
- 10 3. The silica powder as claimed in claim 1, wherein the sum of impurities is less than 150 ppb and the individual impurity levels are Cu < 1 ppb, Fe < 25 ppb, Ni < 2 ppb, Cr < 2 ppb, Ti < 3 ppb, Al < 31 ppb, and Ca < 65 ppb, Mg < 12 ppb, Na < 12 ppb, K < 6 ppb, Li < 1 ppb and the
15 powder is carbon-free.
4. The silica powder as claimed in claim 1, 2 or 3, wherein the powder is a fumed silica powder and has a BET surface area of between 50 and 300 m²/g, preferably
20 between 150 and 250 m²/g.
5. The silica powder as claimed in claim 1, 2 or 3, wherein the powder is a fused silica powder and has a mean particle size of between 100 nm and 200 µm, preferably between 1 µm and 200 µm, very particularly preferably between 5 µm and 40 µm.
25
6. The silica powder as claimed in claim 5, which has a narrow particle size distribution with D(95) - D(5) < 50 µm, preferably D(95) - D(5) < 35 µm.
30
7. The silica powder as claimed in claim 5 or 6, which has a spherical morphology and is completely vitrified.
- 35 8. A process for producing the silica powder as claimed in one of claims 1 to 4, in which a high-purity fumed

silica powder is obtained by flame hydrolysis of high-purity SiCl_4 , wherein the flame hydrolysis of the SiCl_4 is carried out in an apparatus which has a metal-free surface.

5

9. A process for producing the silica powder as claimed in one of claims 5 to 7, in which a high-purity fused silica powder is obtained by sintering a high-purity fumed silica powder, wherein the sintering of the fumed silica powder is carried out in an apparatus with a metal-free surface.

10. The process as claimed in claim 8 or 9, which is carried out under clean room conditions.

15

11. The process as claimed in claim 10, which uses clean room conditions from classes 100 000 to 1, preferably 10 000 to 100, particularly preferably 1000.

12. The process as claimed in claim 8 or 9, which is carried out at a pressure of between 0.913 bar and 1.513 bar, preferably between 1.013 bar and 1.413 bar, particularly preferably between 1.020 bar and 1.200 bar.

13. A nozzle comprising an inner tube located in an outer tube, wherein the inner tube consists of silicon-containing material with silicon as the main constituent.

14. A plasma torch comprising a powder nozzle, an intermediate tube and an outer tube, wherein the powder nozzle, the intermediate tube and the outer tube have a surface made from a silicon-containing material with silicon as the main constituent.

15. An apparatus for producing the silica powder as claimed in one of claims 1 to 7, wherein all the surfaces

which come into contact with the silica powder are metal-free.