EVONIK
INDUSTRIES

AEROXIDE® fumed aluminum oxide and AEROSIL® fumed silica in lighting applications

Technical Information 1283

AEROSIL®

Evonik. Power to create.

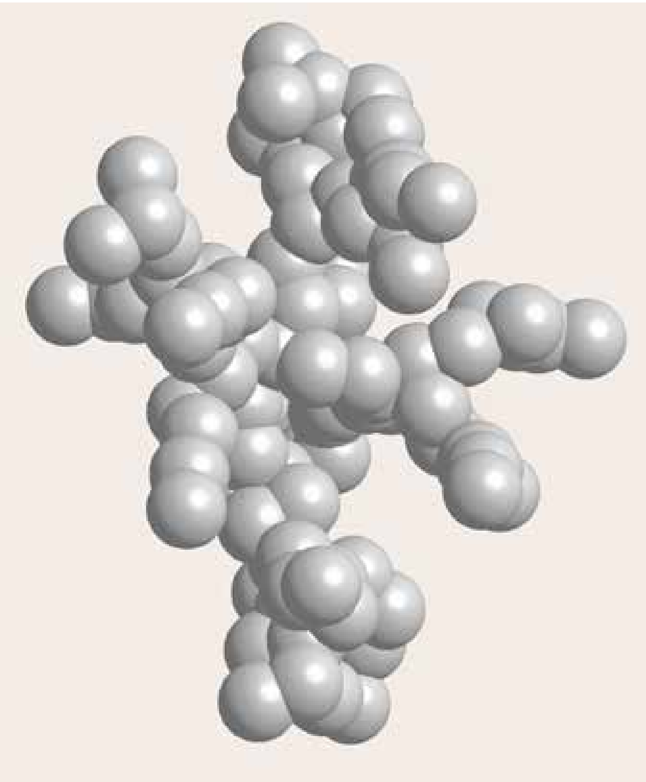
Introduction

AEROSIL® fumed silica and AEROXIDE® fumed aluminum oxide products are characterized by a number of physical-chemical properties that make them very valuable in various lighting applications. These properties include:

- extremely small primary particle size
- surface charge
- electrostatic properties
- optical properties, especially the refractive index
- secondary structure (aggregates)
- high purity
- low moisture content

AEROSIL® and AEROXIDE® products are produced in a flame process and have a unique particle structure, as illustrated in **Figure 1**.

Figure 1
Computer model for fumed oxides



With this structure, these products can perform functions in illuminants that are difficult to duplicate with other materials. This makes these products indispensable in modern lighting applications.

AEROSIL® fumed silica and AEROXIDE® fumed aluminum oxide products simultaneously perform:

- as inorganic binders
- as mercury diffusion barriers
- as matting agents
- as selective UV reflectors
- to optimize light yield
- to fine-tune the spectral light distribution
- as raw material for the manufacture of illuminants

Production and properties

AEROSIL® fumed silica is a synthetic, amorphous silicon dioxide that is produced in an oxyhydrogen gas flame through hydrolysis of chlorosilanes according to the following reaction equation:

$$SiCl_4 + 2H_2 + O_2 \rightarrow SiO_2 + 4\,HCl$$

Evonik Industries developed this production process over 60 years ago and has continuously improved it since then. Since it is produced by flame hydrolysis, AEROSIL® is considered a fumed silicon dioxide or fumed silica. Because of its fine particle structure, the product is also referred to as highly dispersed silicon dioxide. Other metal oxides, such as aluminum oxide and titanium dioxide, can also be produced using the AEROSIL® process, provided that vaporizable starting materials are available. Evonik markets these other oxides under the trade name AEROXIDE®. The reaction equation for aluminum oxide is as follows:

$$2\,AlCl_3 + 3\,H_2 + 1.5\,O_2 \rightarrow Al_2O_3 + 6\,HCl$$

Evonik also produces a number of mixed oxides.

High purity

Evonik uses very pure raw materials that are exclusively of chemical origin. AEROSIL® has an SiO_2 content of more than 99.8 wt% (loss on ignition), making it one of the purest silicas on the market. The same applies to AEROXIDE® aluminum oxide, which has a purity exceeding 99.8%. The content of heavy metals is usually below the detection limit of common analytical methods.

Versatile product modifications

In order to meet a wide range of technical demands, several AEROSIL® fumed silica and AEROXIDE® fumed aluminum oxide grades are available. For example, the specific surface area of AEROSIL® OX 50 amounts to only 50 m^2/g, whereas that of AEROSIL® 380 is nearly eight times higher. AEROSIL® 200, our best known product, has a specific surface area of 200 m^2/g. The surface chemistry and structure of AEROSIL® and AEROXIDE® can also be changed.

For instance, it is possible to chemically anchor dimethylsilyl, trimethylsilyl or polydimethylsiloxane groups to the surface of AEROSIL® and AEROXIDE® by chemical reaction with organosilicon compounds. Products that have undergone this surface modification include AEROSIL® R 972, AEROSIL® R 812 S and AEROXIDE® Alu C 805.

Evonik also offers fumed silica and metal oxide dispersions in water under the trade name AERODISP®.

Fine structure

AEROSIL® fumed silica and AEROXIDE® aluminum oxide are both white, fine, light powders consisting of primary particles that are approximately 7 to 40 nm in size, depending on the product grade.

The primary particles are not isolated, but linked into stable aggregates typically exceeding hundred nanometers.

The aggregates form loose, micrometer-sized agglomerates that can easily break down again in the application process.

Fluorescent lamps

This section will explain and highlight the many functions that AEROXIDE® fumed aluminum oxide performs in fluorescent lamps (energy-saving lamps, fluorescent tubes). The glow in these types of lamps is not produced by a filament, as in traditional light bulbs. Instead, the light is generated by a plasma [1], which is ignited and keeps glowing, powered by electrical current. Most fluorescent lamps use a mercury vapor plasma, which radiates at a wavelength of 254 nm. This UV light is invisible to the human eye, but can be converted into visible light by fluorescent materials ("phosphors") [2].

To generate white light, a thin layer that consists of a mixture of different fluorescent materials is applied to the inside surface of the fluorescent tube. This is the concept of the most simple and basic fluorescent tube. AEROXIDE® fumed aluminum oxide can significantly improve the performance of these types of lamps:

- The adhesion of fluorescent materials to one another and to the glass surface is inherently poor because both, the fluorescent materials and the glass surface usually have a negative surface charge. Aluminum oxide, which has a positive surface charge, can be used as an inorganic binder that is stable in this operating environment. A separate alumina layer between the glass tube and the layer of fluorescent material will not only improve adhesion but also fulfill other critical functions (see below).

- To maximize the effectiveness of fluorescent lamps, all of the UV light that is generated needs to be converted into visible light. UV radiation that is not converted will be absorbed by the glass tube and converted into heat. To prevent this, the layer of fluorescent materials could be made thicker. While this would keep the UV radiation from being absorbed by the glass, it would prevent the visible light that is generated from leaving the tube. The use of AEROXIDE® fumed aluminum oxide as a selective UV reflector offers a much better solution than increasing the thickness of the fluorescent material. In addition to its function as a selective UV reflector that permits light to pass through, it also acts as a barrier for other materials (see next paragraph).

- Without fumed aluminum oxide, small amounts of mercury continuously penetrate into the layer of fluorescent material and into the glass tube. This causes the tube to turn gray over time. This graying affects efficiency as well as light yield: First, the mercury that is required to produce the UV radiation is reduced. Secondly, as the tube turns gray, it absorbs more of the visible light, increasing the transformation of light into heat. To compensate for these losses, it is possible to increase both, the amount of mercury and the wattage of the lamp. This solution results in more heat generation and promotes the process of diffusion even more. A layer of AEROXIDE® aluminum oxide acts as an effective mercury barrier. This keeps the use of this poisonous heavy metal to a minimum, while simultaneously increasing the service life of the lamp.

These three different functions that AEROXIDE® fumed aluminum oxide simultaneously performs in fluorescent lamps, make it an indispensable component of high end fluorescent designs. While its functions as an inorganic binder and as a mercury barrier are relatively easy to understand, its special optical properties are more complex and require further explanation.

AEROXIDE® fumed alumina as selective UV reflector

Fumed Aluminum Oxide is transparent over a very wide spectral range: extending from extreme UV, through visible light, and reaching into the IR region. That means that light within these wavelengths can pass through an alumina screen without energy loss. There is no absorption and consequently,

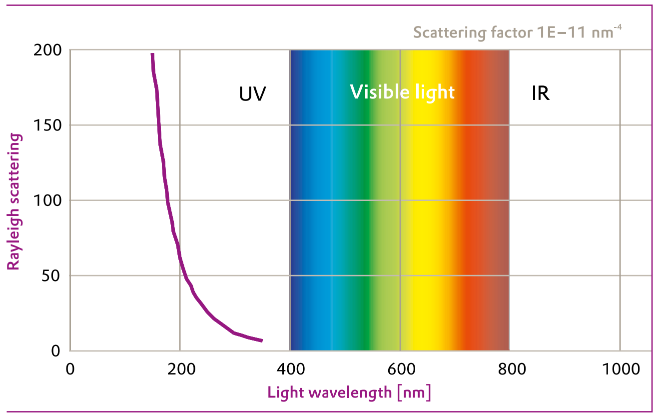
[1] A plasma is generally defined as an ionized gas consisting of free electrons and positive ions.

[2] The term "phosphor" has become standard English usage for these fluorescent materials in spite of the fact that neither the chemical element phosphorus nor the physical effect of phosphorescence is applicable. The effect that is used involves the fluorescence of special fluorescent dyes, which emit light in a particular other color when excited by UV light. As far as their chemistry is concerned, these materials usually consist of oxides, silicates or other inorganic materials that have been doped with rare-earth metals.

no transformation from light to heat. Glass, on the other hand, does absorb UV light because it consists primarily of silicon dioxide.

The fact that fumed aluminum oxide powder appears white and not translucent is a result of light scattering that occurs at the gas/particle interface. The light scattering properties, primary particle size, and aggregate structure of AEROXIDE® fumed aluminum oxide make it a nearly perfect optical material for a wavelength-selective UV reflector. Particles that are much smaller than the waveleng λ obey Rayleigh's scattering law[3], which states that the intensity of light scattered at the phase boundary is proportional to λ^{-4}. The consequence of this phenomenon is depicted in **Figure 2**.

Figure 2
Scattering behavior as a function of the wavelength of the light according to Rayleigh's scattering law



Although the differences between different wavelengths remain small in the visible range of the light, the curve rises sharply in the UV range. This means that, for the same particle size, 16 times more light is scattered at 254 nm than at 500 nm (green light) and even 100 times more than at 800 nm (red light). Considering that the light within the tube of fluorescent material will be scattered an enormous number of times before it can exit the tube, it is easy to understand why a layer of AEROXIDE® fumed aluminum oxide on the inside of the tube works like a cage that seals in the UV radiation while allowing the visible light to pass through.

The UV light is not destroyed in this process, but remains inside the tube to be transformed into visible light by the fluorescent materials. This increases the light yield and efficiency of the lamp.

[3] Rayleigh scattering is the reason why the sky appears blue. Small particles in the atmosphere scatter the blue portion of sunlight more strongly than other wavelengths (see **Figure 2**). The effect is even more pronounced when the sun is low, namely at sunrise and sunset. During those hours, the light rays are subjected to much more scattering because the increased distance through the atmosphere. When it reaches the observer, it contains a higher proportion of red light, which is scattered the least.

The special structure of AEROXIDE® fumed aluminum oxide makes it especially effective as a selective UV reflector. This structure consists of primary particles with diameters in the range of 7 to 20 nm. During production these primary particles form stable aggregates typically over 100 nm suitable for light scattering (see **Figure 1** on Page 2). When compacted in a layer, the aggregates are not destroyed and retain their structure. They form a porous network that retains much of the gas/particle interfaces for scattering, as would the individual particles. Aluminum oxide particles produced by other processes lose a much greater part of their surface area.

The effects that have been described are reinforced by the fact that the index of refraction is a function of wavelength. AEROXIDE® fumed aluminum oxide has a refractive index of 1.69 at 589 nm (Na D-line). The refractive index increases with decreasing wave length, exceeding 1.8 at a wavelength of 254 nm.

Typical construction of a fluorescent lamp with AEROXIDE® aluminum oxide

The construction illustrated in **Figure 3** has proven to effectively utilize the three functions that AEROXIDE® fumed aluminum oxide performs. First, the inside surface of the glass tube is coated with a layer of pure aluminum oxide. An aqueous dispersion is ideal for this type of application and can be produced with suitable dispersing equipment.

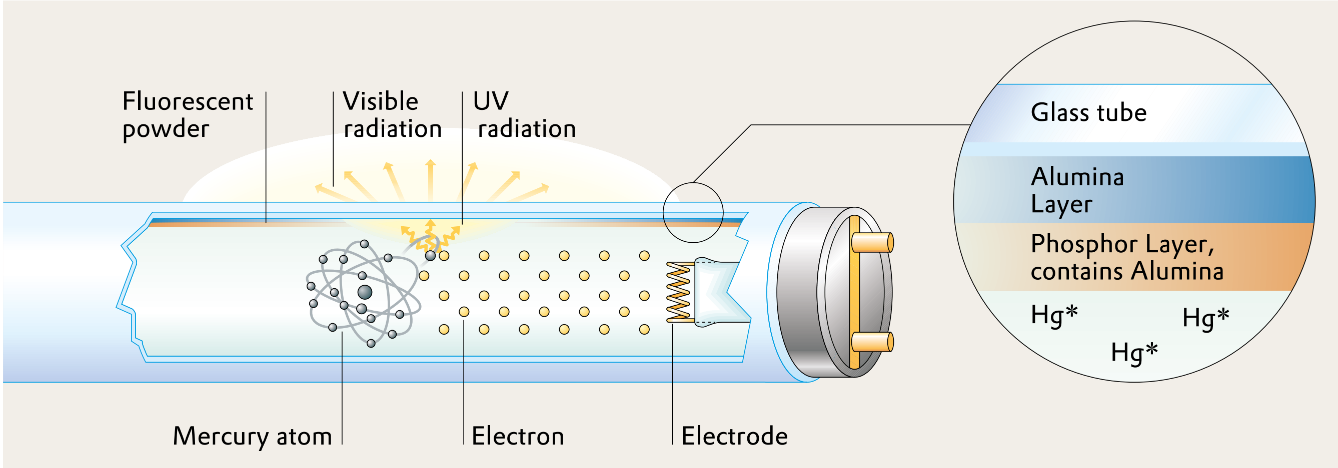
AEROXIDE® aluminum oxide is also available in form of dispersions that are easy to handle and ready to use. These are part of the AERODISP® product portfolio (see Product Overview). Depending on the type of lamp and other limitations the aluminum oxide layer should have a thickness of approximately 3 – 5 µm.

The layer of fluorescent material is applied in dispersed form as well, on top of the aluminum oxide. The fluorescent material adheres much better to the aluminum oxide than it would to a plain glass surface. To improve the cohesion of the fluorescent material layer itself, or to function as an inorganic binder, the aluminum oxide can be mixed into this layer as well. Typical additional levels are between 2 – 5 %.

Dispersions based on organic solvents (e.g. butylacetate) or water can be used for the coating process. Polymer additives (e.g. cellulose derivatives, polyethylene glycol and others) are added to the dispersions to improve processability and adhesion during the coating process itself. To achieve a uniform thickness of the coating, it is necessary to precisely adjust the rheological properties of the dispersions and to standardize the drying conditions.

The final step is to bake the layers in a furnace at temperatures close to the softening point of the glass (~ 600 °C). This causes the organic components of the layers to pyrolyze. The AEROXIDE® fumed aluminum oxide, as an inorganic binder, holds the layers together and bonds them to the glass surface. Further assembly can proceed while the tubes are still hot (approx. 500 °C).

Figure 3
Construction of a fluorescent lamp (Source: Ullmann's Encyclopedia of Industrial Chemistry)



Other applications

LED

AEROSIL® and AEROXIDE® products, are commonly used to improve properties silicone encapsulates for LED applications such as:

- prevent settling of the phoshor
  (AEROSIL® 150, AEROSIL® R 106, AEROSIL® R 812 S)
- provide mechanical strength
  (AEROSIL® 150, AEROSIL® R 106, AEROSIL® R 812 S, AEROSIL® R 8200)
- improve heat expansion properties
  (AEROXIDE® Alu C)
- increase refractive index
  (AEROXIDE® TiO_2 P25, AEROXIDE® Alu C)
- reduce moisture & sulfur permeability
  (AEROSIL® R 812 S, AEROSIL® R 8200)

Such properties are important to protect the fragile chip and wires from external conditions, such as air or moisture and improve the durability of LED module. Additionally, AEROSIL® grades are commonly added to silicone or epoxy glues, for LED-assembling. AEROSIL® 200 and AEROSIL® R 202 are very efficient rheological agents to achieve thixotropic properties of silicone and epoxy glue, respectively.

SAVOSIL™ Silica Glass is the ideal material for High Brightness LED (HB LED) and UV-LED. The unique SiVARA™ Sol-Gel Technology allows formation of reproducible complex geometry in sizes starting from 3 mm diameter. Therefore, allowing virtual complete versatility in LED design. Silica glass offers numerous advantages over traditional technology such as:

- Excellent light direction
- Enhanced efficiency
- Exceptional strength to protect the package
- High durability under action of UV-irradiation and/or elevated tempereture

Superior physical and chemical properties to improve reliability issues of high power LED caused by the environment such as UV radiation and moisture. More information on SAVOSIL™ Silica Glass can be found at www.savosil.com.

Highly purified raw material

Since it is produced in a manufacturing process that starts out with high-purity chlorosilanes, AEROSIL® fumed silica is the product best suited for the production of fluorescent materials based on silicates. We offer it in a form that is particularly easy to use, AERODISP® W 7215 S, an aqueous dispersion whose particularly gentle dispersion process has been optimized toward high purity. The iron content of this dispersion is below 1 ppm (parts per million) and the content of all other trace metals combined is likewise below 1 ppm.

Incandescent lamps

Traditional incandescent lamps can also make use of the properties of fumed oxides. Internal frosting with ultrafine metal oxide particles is increasingly being used, replacing the traditional frosting with hydrofluoric acid. Costs and environmental concerns are the key drivers for this development. Electrostatic coating of the glass bulb while it is still hot is the primary process used for application.

AEROSIL® fumed silica can be used for this purpose either by itself or in combination with colored pigments. The very low moisture content of AEROSIL® products facilitates the process and also extends the service life of the bulbs. AEROSIL® COK 84, a mixture of fumed silica and fumed alumina with special electrostatic properties, is particularly easy to process.

Product overview, physical-chemical data

Powdered AEROXIDE® fumed aluminum oxides for fluorescent lamps

Product name	AEROXIDE® Alu 65	AEROXIDE® Alu C	AEROXIDE® Alu 130
BET Surface Area	65 m^2/g	100 m^2/g	130 m^2/g
X-Ray Form (ca.)	Θ and δ, little γ	33 % δ, 66 % γ	γ
Spec. Gravity	approx. 3.2 g/cm^3 (depending on Ignition loss)		
pH (4 % aq. Slurry)	4.5–6	4.5–5.5	4.4–5.4
Ignition Loss 2 hrs 1000 °C	< 3.0 wt. %		
Refractive Index, η	1.69		

Aqueous dispersions based on AEROXIDE® fumed aluminum oxide powder

Product name	AERODISP® W 440	AERODISP® W 630	AERODISP® W 925
Solids Content	40 %	30 %	25 %
pH-value	3–5	3–5	3–5
Appearance	White, milky and low viscous liquids		
Median Aggregate Size	~ 0.20 µm	~ 0.14 µm	~ 0.11 µm
Surface Charge	+ Cationic (positively charged)		

AEROSIL® fumed silica products for the electrostatic internal frosting of fi lament lamps

Product name	AEROSIL® 200	AEROSIL® R 972	AEROSIL® COK 84
BET Surface Area	200 m^2/g	110 m^2/g	185 m^2/g
Chem. Composition	SiO_2	SiO_2	SiO_2/Al_2O_3
Carbon Content	-	0.6 - 1.2 wt. %	-
pH (4 % aq. Slurry)	3.7–4.7	3.6–5.5	3.6–4.3
Ignition Loss 2 hrs 1000 °C	≤ 1.0 wt. %	≤ 2.0 wt. %	≤ 1.0 wt. %

Literature

1 W. J. van den Hoek *et al.* (Philips Lighting B. V.), "Lamps" in Ullmann's Encyclopedia of Industrial Chemistry, 10th edition (2002), DOI: .1002/14356007.a15_115

2 M. Zachau, A. Konrad (OSRAM GmbH), „Nanomaterials for Lighting", Solid State Phenomena Vols. 99-100 (2004), pp. 13-18.

3 R. H. French *et al.* „Optical Properties of Aluminum Oxide…", J. Am. Ceram. Soc. Vol. 81 (1998), pp. 2549-2557.

4 Technical Bulletin Fine Particles, No. 56 „Highly Dispersed Metallic Oxides Produced by the AEROSIL® Process", Company Publication (Evonik).

The data represent typical values and not production parameters.

We also have numerous other products not listed here that may also be suitable for lighting applications. Information about these products can be found in our Product Overview and in our technical publications. Please ask us about the most recent products from research and development. Our web site is also a source for the current information **www.aerosil.com**.

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