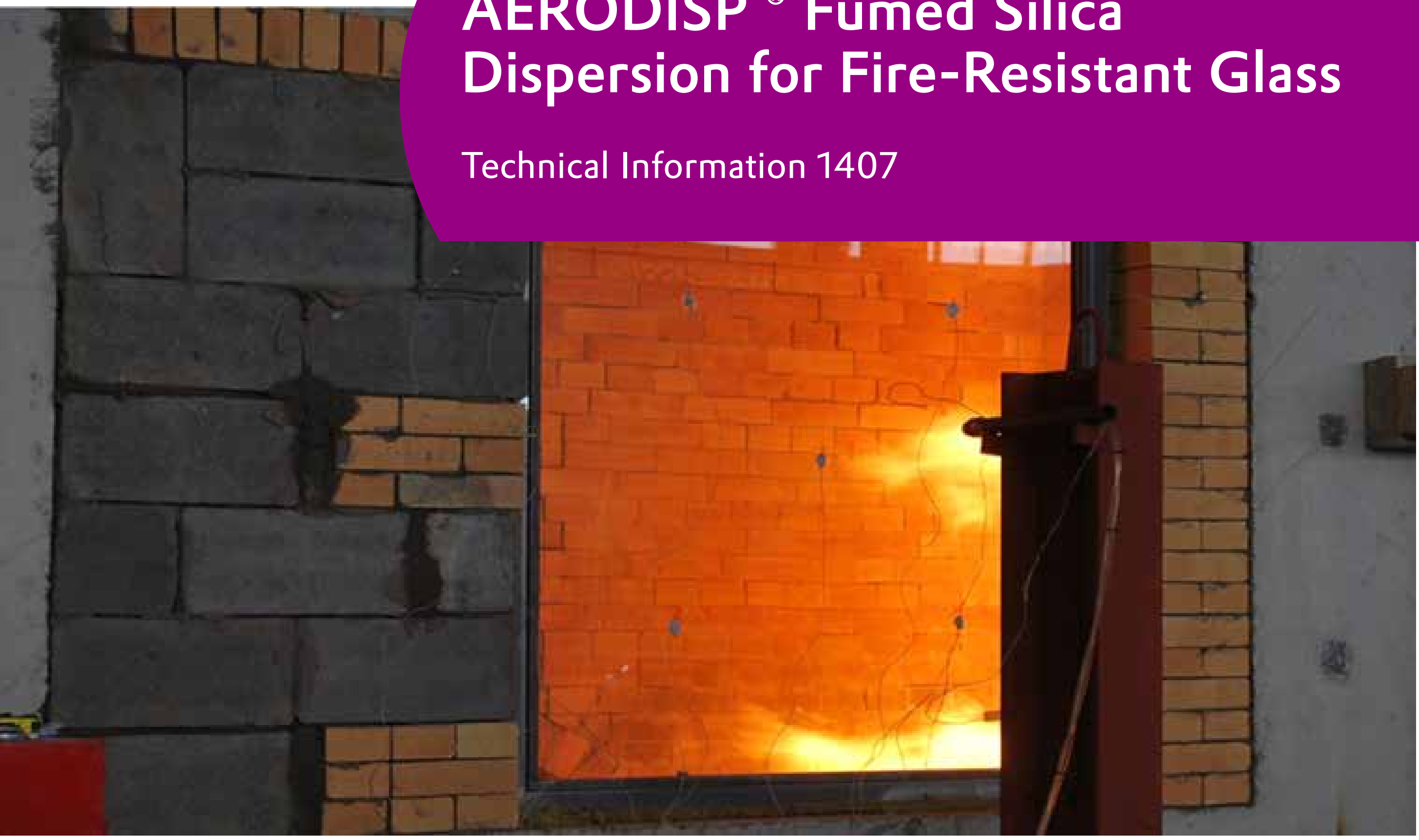
AERODISP ® Fumed Silica Dispersion for Fire-Resistant Glass

Technical Information 1407



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Introduction

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

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

The resulting silica is made of primary particles that fuse together to form aggregates which tend to clump together upon cooling, forming much larger agglomerates. Both the structure of the primary particles and the states of aggregation and agglomeration determine the overall morphology of the powder. Evonik Industries developed the process for making fumed silica already in the early 1940ies and, through extensive knowhow in the control of the process parameters acquired through the years, has been able to tailor the morphology of the silica particles to meet specific demands of a wide range of industrial applications.[1] Many applications require AEROSIL® products to be well dispersed in a liquid medium but not all customers have the necessary equipment and/or experience for making dispersions. Evonik Industries offers therefore a broad range of AERODISP® dispersions developed for specific applications, including fire-resistant glass. AERODISP® W 1244 has been developed specifically for manufacturing a potassium silicate intumescent material for use in fire-resistant glass.

Fire-resistant glass technology overview

Classification

The most basic function of fire-resistant glass is the prevention of fast fire propagation within buildings. Maintaining integrity in case of fire for the minimum specified rated time is therefore a necessity for containing smoke and flames. This function alone however is in many cases insufficient to guarantee the safety of the building occupants. The rapid propagation of heat through the glass can lead to an increase in temperature in the adjacent area sufficient to initiate combustion of materials in the area even before the integrity of the glass fails. Fire-resistant glass technology has therefore been developed to limit also radiant and conductive heat transfer through the glass.

The legislation governing the use of fire-resistant materials in buildings varies from country to country, even within the European Union. In an effort to facilitate the comparison of the different glass classifications and governing legislations in the single member countries, the European commission has established a common classification system for fire-resistant materials (EN 13501-2) which defines three main classes of fire-resistant glass summarized in **Table 1**.

Table 1 Fire-rated glass classifications according to EN 13501-2

E	Integrity for specified rated time in minutes
EW	Integrity and radiant heat barrier for specified rated time (max 15 kW/m² at 1 m distance from glass)
EI	Integrity and insulation for specified rated time (Temperature increase above ambient of opposite side of glass): Maximum ≤180 °C, Average ≤140 °C)

Class **E** provides basic integrity for the specified rated time in minutes 30, 60, 90, etc.

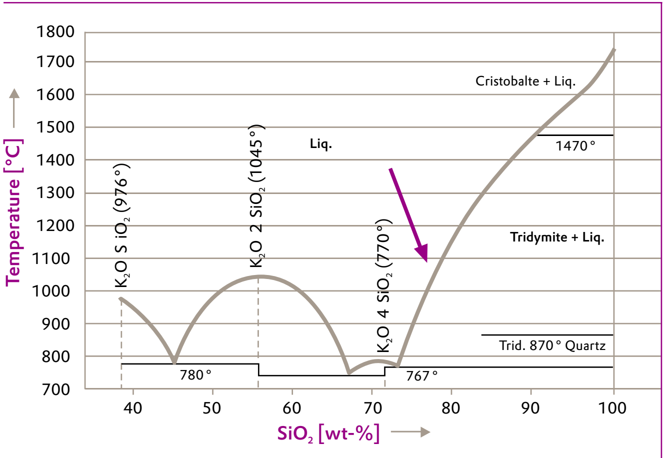
Class **EW** provides, in addition to basic integrity, protection against radiant heat transport. The maximum heat flow at 1 m distance from the glass, opposite to the fire, must not exceed of 15 KW/m² in the rated time interval of the glass.

Class **EI**, in addition to basic integrity, provides an effective insulation barrier for its entire rated resistance time in minutes. During this time interval the maximum temperature of the glass surface on the side opposite to the fire does not exceed ambient temperature by more than 180 °C. Additionally the average temperature of the glass surface on the side opposite to the fire does not exceed ambient temperature by more than 140 °C. Class **EI** fire-resistant glass provides the maximum protection in the event of fire and can be assumed to satisfy also **EW** requirements even if not specifically tested for them.

Use of alkali silicate intumescent layers

The leading technologies used to manufacture EI and EW rated fire-resistant glass are based on glass plates separated by intumescent layers, which are composed of a highly viscose solution of alkali silicates and appropriate organic additives in water.[2,3] In the event of fire the increase of temperature inside the intumescent layer leads to evaporation of water and to an increase of pressure inside the layer. As a result, the glass plate closest to the fire collapses, while the dewatered alkali silicate forms a layer of insulating foam approximately ten times thicker than the original layer. The organic additives in the interlayer carbonize under the influence of heat, providing an effective barrier to the infrared radiation. Key parameters determining the fire-resistant performance of the alkali silicate interlayer are the ratio of silica to alkali content and the overall solid content. The silica to alkali content is often referred to as silica modulus and can be expressed either as a weight or molar ratio. As can be seen in the potassium silicate phase diagram (**Figure 1**), the melting temperature of potassium silicate is strongly dependent on the silica modulus.[4] A local maximum in the melting temperature occurs when the molar silica modulus is equal to 2; however the high alkalinity of the silicate at this ratio can have a corrosive effect on the confining glass plates eventually leading to opaqueness of the window. To achieve a sufficiently high melting temperature necessary for fire-resistant glass applications, the alternative is therefore a composition in the region indicated by the arrow having a higher silica modulus.

Figure 1 Phase diagram of $K_2O:SiO_2$ system. Arrow indicates region of composition typically used in fire-resistant glass.



Potassium silicate and sodium silicate are the two most common alkali silicates available. However, potassium silicate has some advantages over sodium silicate for application in fire-resistant glass. It has a higher temperature resistance than a corresponding sodium silicate and, for equivalent alkalinity or silica modulus, potassium silicate also has a higher solubility making it possible to achieve an interlayer with higher solid content and/or silica module for further increasing the temperature resistance.[5]

"Cast-In-Place" manufacturing process of fire resistant glass

As the term suggests, "Cast–In-Place" refers to a process in which the alkali silicate intumescent interlayer is made by a casting process directly in the space between two glass sheets. An alkali silicate solution prepared shortly beforehand is used to fill the space between two tempered safety glass plates. The composition is defined as such that subsequent to the filling, once the silica is completely dissolved, the viscosity sharply increases conferring to the interlayer a solid aspect.

As opposed to other manufacturing techniques, in which for example a thin alkali silicate layer is deposited on a glass plate and subject to an evaporation step, the "Cast-In-Place" technology offers the advantage of:

- A simple production process
- Low investment costs for a new production startup
- Suitability for "newcomers", for small and mid-size enterprises
- No process intrinsic size limitations as with other technologies
- No waste of material because the production is done directly in the required dimensions.
- Environmental friendliness (non-toxic, non-hazardous)

Use of tempered glass plates in a "Cast-In-Place" process enables manufacturing of the largest fire-resistant glasses possible. Non tempered glass has size limitations due to the limited mechanical strength.

AEROSIL®

AEROSIL® fumed silica has unique properties as a silica source for alkali silicate intumescent interlayers for fire-resistant glass.

- The special morphology permits high silica modules and high solid content to be achieved in a process that remains easily controllable.
- The high purity ensures excellent optical characteristics of the final product
- Its dry state allows maximum flexibility in the formulation of the dispersion

The special morphology of AEROSIL® particles allows the preparation of high concentration dispersions that have relatively low viscosity at the same time. The high concentration is necessary to achieve an alkali silicate composition having both a high silica module and high solid content. The low viscosity is required on the other hand for the interlayer filling process. The available processing time is also a critical parameter in "Cast-In-Place" processes. The ideal size of the AEROSIL® particles make the dissolution process in the alkaline environment gradual, ensuring the availability of an adequate time for filling the interlayer before the solidification of the alkali silicate takes place.

The high purity of the $SiCl_4$ raw material used for the production of AEROSIL® ensures extremely low metallic impurity levels in the silica that in most cases are below the detection limits of typical analytical techniques. The high purity of the silica raw material is essential for achieving the best possible optical quality of the final window. Otherwise contaminants could in fact cause isolated scattering centers, haziness and/or a coloring of the alkali silicate interlayer.

The fact that AEROSIL® is a dry powder allows additional flexibility in the formulation because the composition of the liquid used for making the dispersion can be selected according to specific requirements and/or desired characteristics, such as a low freezing point.

AERODISP® W 1244

The unique properties of AEROSIL® influence its dispersability in water. Manufacturing of concentrated dispersions requires special know-how and appropriate equipment. The AERODISP® W 1244 dispersion is a product developed by Evonik Industries specifically for manufacturing fire-resistant glass via "Cast-In-Place" processes. It incorporates all the advantages of AEROSIL® high purity fumed silica in a ready to use optimized dispersion that greatly simplifies production procedures and minimizes initial investment costs. The dispersion already contains all the necessary components to create the infrared barrier in case of fire and to lower the freezing point of the interlayer, ensuring the transparency of the glass even in low temperature climates. The only additional component that needs to be added is potassium hydroxide (KOH), which reacts with the dispersion forming the solid like potassium silicate intumescent material. The preparation procedure of fire-resistant glass, using AERODISP® W 1244, consists of five simple steps.

1. Mixing AERODISP® W 1244 and concentrated (50 %) KOH solution in a temperature controlled environment under vacuum
2. Further degassing the mixture under vacuum and controlled temperature
3. Filling the interlayer between the glass sheets with the prepared solution
4. Sealing the window
5. Curing at 75 °C to achieve full transparency

The main equipment components required are:

- A vacuum reactor with cooling/heating mantle, temperature control and agitating body (anchor mixer)
- A vacuum pump
- Cold/warm water supply
- A curing oven
- Tools for handling the glass plates
- Equipment for dosing the liquid mixture.

The excellent performance of AERODISP® W 1244 based fire-resistant glass has been proven during numerous certification tests. All standard EI and EW classes can be easily achieved by choice of appropriate thickness of intumescent layer or by increase of numbers of intumescent layers. It has also been demonstrated, that the overall thickness of AERODISP® W 1244 based fire-resistant glass is lower than that of other fire-resistant glasses of the same fire-resistant class. This is an additional advantage for manufacturers of fire-resistant constructions, since lower thickness reduces the overall construction weight and opens new opportunities for fire-resistant constructions.

An illustrative example is shown in **Figure 2** in which an EI 30 rated fire-resistant window containing a single 6 mm thick potassium silicate based intumescent layer prepared with AERODISP® W 1244 is subjected to a testing procedure to verify its EI rating. The temperature inside the oven during the certification test is close to 1000 °C with a standardized heating profile and temperature distribution inside. The photographs have been made after varying time intervals from the beginning of the test. They document the typical evolution of a fire resistant glass, based on AERODISP® technology, under fire. The last photograph shows a cross-sectional view of the expanded intumescent layer after the test. The thickness is approximately 10 times that of the original layer. Measuring sensors on the window confirm that the window actually met the EI conditions for 39 minutes, in total 9 minutes more than its rating. In a similar test carried out on an EI 45 rated fire-resistant window containing a single 8 mm thick potassium silicate based intumescent layer prepared with AERODISP® W 1244 the EI conditions were met for 58 minutes, in total 13 minutes more than its rating.

Figure 2
Example of test done on EI 30 rated fire-resistant glass containing a single 6 mm thick potassium silicate based intumescent interlayer prepared with AERODISP® W 1244 dispersion. The photo sequence shows different stages of the test:
a after 1 minute **b** 20 minutes **c** 35 minutes **d** cross-sectional view of expanded interlayer.
The glass actually satisfied the EI conditions for 39 minutes (9 longer than its rating)

FAQs

1 *Does tempered glass have to be used in "Cast-In-Place" processes?*

Answer:
Tempered glass is highly recommended because of a better mechanical stability. Larger glass surfaces are possible, and the glass is much more resistant to damage in everyday conditions than with normal glass.

2 *Does Evonik provide a guideline formulation for using AERODISP® W 1244 and other technical instructions in general for the manufacturing of the fire-resistant glass?*

Answer:
Yes, Evonik can provide a guideline formulation with procedures and also provide a description of the equipment necessary for setting up a production line. We support our customers in setting up the production process, even if they have little or no previous experience with this technology.

3 *Is fire-resistant glass manufactured with AERODISP® W 1244 UV stable?*

Answer:
Yes, fire resistant glass interlayers made with AERODISP® W 1244 are mainly an inorganic composition, which is UV stable.

4 *What is the allowed temperature range for the fire-resistant glass made with AERODISP® W 1244?*

Answer:
The glass remains transparent and stable within the temperature range of approximately −20 °C to 60 °C.

5 *In case of fire what will be the expansion factor of the interlayer?*

Answer:
The Interlayer will expand by a factor of 10 forming insulating foam.

6 *What is the largest fire-resistant glass size that can be made using AERODISP® W 1244?*

Answer:
There is no intrinsic size limitation. The limitations are generally imposed by the available sizes of the tempered glass and the availability of the suitable equipment for handling the large glass sizes.

References

1 Technical Bulletin Fine Particles No. 11 Basic Characteristics of AEROSIL® Fumed Silica, Company Publication (Evonik)

2 A Guide to Best Practice in the Specification and Use of Fire-Resistant Glazed Systems, Publication of Glass and Glazing Federation

3 The Role of Intumescent Materials in Timber and Metal Based Fire Resisting Glazing Systems, publication of Intumescent Fire Seals Association

4 Lagaly, G., Tufar, W., Minihan, A. and Lovell, A. 2000. Silicates. Ullmann's Encyclopedia of Industrial Chemistry

5 Sodium and Potassium silicates, Company Publication PQ Europe

Physical Chemical Data

Property	AERODISP® W 1244
Solid Content (%)	App. 48 %
pH	10.6–11.2
Dynamic viscosity	< 200 mPa·s
Density	1.4–1.5 g/ml
Stabilization	KOH

All data in this table represent typical values, not specified parameters

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