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Magnetron sputter coater

Lotte Plaza, Moscow
SunGuard® HP Royal Blue 41/29
Mosprojekt 2, Kolsnitsin’s Arch. Bureau
1.1 History

The history of glass production dates back to about 5000 BC. Glass beads discovered in ancient Egypt and early Roman sites bear witness to a long tradition of drawing and moulding techniques used in glass production. For centuries, however, individual craftsmanship dominated manufacturing processes that ranged from using blowpipes and cylinder blow moulding techniques to the crown glass method. These manual production methods resulted in small quantities and small window panes which were almost exclusively used in stained glass windows in churches.

Demand for glass during the seventeenth century rose because in addition to master church builders using glass in church windows, builders of castles and stately townhouses were now discovering how to use glass to enclose spaces as well. French glassmakers first developed a glass rolling process that produced 1.20 x 2 m glass panels, a size that until then had seemed impossible. Glass production did not become industrialised until the twentieth century when 12 x 2.50 m sheets of glass later began to be mass produced on a large scale using the Lubbers and Fourcault methods of glass production, advancing to the more recent technologies developed by Libbey-Owens and Pittsburgh.

All of these methods had one distinct disadvantage: manufactured glass panels had to be ground and polished on both sides to obtain distortion-free and optically perfect mirror glass, a process that was extremely time consuming and expensive.

1.2 Float glass

Industrial glass – which today would be glass used in the automotive and construction industries – was originally manufactured using a system known as float glass. This floating process, which reached its peak in 1959, revolutionised glass production methods. Until this float process was developed, glass panes were produced by drawing or moulding molten glass, and then polishing it.

This new method allows the glass to “float”, with the molten glass spreading out evenly over the surface of a liquid tin bath. Due to the inherent surface tension of the liquid tin, and the fact that glass is only half as dense as tin, the molten glass does not sink into the tin bath, but rather floats on the surface, thereby moulding itself evenly to the surface shape of the liquid tin. This method creates absolute plane parallelism which guarantees freedom from distortion and crystal clear transparency. Reducing the temperature in the tin bath from approx. 1000 °C to approx. 600 °C turns a viscous mass of molten glass into a solid glass sheet that can be lifted right off the surface of the tin bath at the end of the floating process.

Tin is ideal for shape forming because it remains liquid throughout the entire shape forming process and does not evaporate, thanks to its low vapour pressure. In order to prevent the tin from oxidising, the floating process takes place in a protective gas atmosphere of nitrogen with a hydrogen additive.

At this point, the glass ribbon is approx. 600 °C and is cooled down to room temperature using a very precise procedure in the roller cooling channel to ensure that no permanent stress remains in the glass. This operation is extremely important for problem-free processing. The glass ribbon is still approx. 50 °C at the end of the 250 m long cooling line and a laser inspects the glass to detect faults such as inclusions, bubbles and cords. Faults are automatically registered and scrapped when blanks are later pre-cut.
Pre-cuts are usually realised at intervals of 6 metres or less, with the glass being cut perpendicular to the endless ribbon. Both edges of the ribbon are also trimmed, generally producing float glass panes of 3.21 m x 6 m which are then immediately processed or stored on frames for further processing. Longer plates of 7 or 8 m are also produced.

An average float glass line is about 600 m long and has a capacity of approx. 70,000 m² with a thickness of 4 mm.

1.2.1 Colouring

Normal float glass has a slightly greenish tint. This colouring can be mainly seen along the edge of the glass and is caused by naturally existing ferric oxide in the raw materials. By selecting extremely ferric oxide-poor raw materials, or by undergoing a chemical bleaching process, the melt can be turned into an absolutely colour-neutral, extra-white glass. GUARDIAN produces this type of glass under the name GUARDIAN UltraClear™. Interiors and special solar products are the widest areas of application.

GUARDIAN also provides GUARDIAN ExtraClear®, a third float glass alternative that distinguishes itself from the competition because of its reduced iron content. In terms of colour and spectral properties, this glass falls between the UltraClear white float and the standard Clear float. Due to its interesting combination of properties, Float ExtraClear is used as the base material for ClimaGuard® thermal insulating and SunGuard® solar control coatings. This improves the selectivity and colour neutrality, irrespective of the particular coatings, especially for glass used in facades.

In addition to these three versions of float glass, tinted glass can be produced using coloured mass. Chemical additives in the mixture allow green, grey, blue, reddish and bronze-coloured glass to be produced during certain production floating line periods. Changing glass colour in the vat naturally entails a considerable degree of effort and increased cost due to scrap and loss in productivity. It is therefore only produced for special campaigns.

1.2.2 Properties

Most of today’s glass production is float glass, with thicknesses usually ranging from 2 – 25 mm and a standard size of 3.21 x 6 m that is used for further processing. The glass has the following physical properties:

1.2.2.1 Density

The density of the material is determined by the proportion of mass to volume and is indicated using the notation “ρ”. Float glass has a factor of ρ = 2,500 kg/m³. That means that the mass for a square metre of float glass with a thickness of 1 mm is 2.5 kg.

1.2.2.2 Elasticity module

The elastic module is a material characteristic that describes the correlation between the tension and expansion when deforming a solid compound with linear elastic properties. It is designated with the formula symbol “E”. The more a material resists deformation, the higher the value of the E-module. Float glass has a value of E = 7 x 10¹⁰ Pa and is defined in EN 572-1.

1.2.2.3 Emissivity

Emissivity (ε) measures the ability of a surface to reflect absorbed heat as radiation. A precisely defined “black compound” is used as the basis for this ratio. The normal emissivity of float glass is ε = 0.89, which means 89 % of the absorbed heat is re-radiated (→ Chapter 3.3)

1.2.2.4 Compressive strength

As the term implies, this indicator demonstrates the resistance of a material to compressive stress. Glass is extremely resilient to pressure, as demonstrated by its 700 - 900 MPa. Flat glass can withstand a compressive load 10 times greater than the tensile load.
1.2.2.5  Tensile bending strength

The tensile bending strength of glass is not a specific material parameter, but rather an indicated value which, like all brittle materials, is influenced by the composition of the surface being subjected to tensile stress. Surface infractions reduce this indicated value, which is why the value of the flexural strength can only be defined using a statistically reliable value for the probability of fracture.

This definition states that the fracture probability of a bending stress of 45 MPa for float glass (EN 572-1) as per the German building regulations list may be a maximum 5% on average, based on a likelihood of 95% as determined by statistical calculation methods.

\[ \sigma = 45 \text{ MPa} \]

as measured with the double ring method in EN 1288-2.

1.2.2.6  Resistance to temperature change

The resistance of float glass to temperature differences over the glass pane surface is 40 K (Kelvin). This means that a temperature difference of up to 40 K over the glass pane has no effect. Greater differences can cause dangerous stress in the glass cross section, and this may result in glass breakage. Heating devices should therefore be kept at least 30 cm away from glazing. If this distance cannot be maintained, the installation of single-pane safety glass is recommended (\( \rightarrow \) Chapter 7.1). The same applies in the case of solid, permanent and partial shading of glazing, due, for example, to static building elements or to nearby plants.

1.2.2.7  Transformation area

The mechanical properties for float glass vary within a defined temperature range. This range is between 520 - 550 °C and should not be compared with the pre-tempering and form shaping temperature, which is about 100 °C warmer.

1.2.2.8  Softening temperature

The glass transition or softening temperature of float glass is approx. 600 °C.

1.2.2.9  Length expansion coefficient

This value indicates the minimum change in float glass when the temperature is increased. This is extremely important for joining to other materials:

\[ 9 \times 10^4 \text{ K}^{-1} \] pursuant to ISO 7991 at 20 - 300 °C

1.2.2.10  Specific heat capacity

This value determines the heat increase needed to heat 1 kg of float glass by 1 K:

\[ C = 800 \text{ J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1} \]

1.2.2.11  Heat transmittance coefficient (U value)

The value for float glass with a thickness of 4 mm is 5.8 W/m²K.

1.2.2.12  Acid resistance

Chart: Class 1 acc. to DIN 12116

1.2.2.13  Alkali resistance

Chart: Class 1-2 acc. to ISO 695

1.2.2.14  Water resistance

Chart: Hydrolytic class 3-5 acc. to ISO 719

1.2.2.15  Fresh, aggressive alkaline substances

These include substances washed out of cement which have not completely hardened and which when they come into contact with the glass, attack the silica acid structure that is part of the glass structure. This changes the surface as contact points become rougher. This effect occurs when the liquid alkaline substances dry and is completed after the cement has fully solidified. For this reason, alkaline leaching substances should never come into contact with glass or any points of contact should be removed immediately by rinsing them off with clean water.
1.3 Coatings on float glass

Industrial coatings for float glass are produced in huge quantities, primarily using 2 techniques. One is the chemical pyrolysis process, also called hardcoating. The second is a physical process called vacuum deposition or magnetron sputtering.

Depending on the coating used, materials in both methods result in a neutral and coloured appearance, whereby the coloured effects are less obvious when viewing the glass head-on and are easier to note when looking at reflections on the surface of the glass. These two technologies are base glass oriented and not to be confused with surface coating applied through spraying, rolling or imprinting processes (→ Chapter 8.2).

1.3.1 Pyrolytic method

This type of float glass coating process occurs online during glass production on the float line. At this point, the glass surface is still several hundred degrees Celsius when metal oxides are sprayed onto it. These oxides are permanently baked onto the surface and extremely hard (hardcoatings) and resistant, but their properties are very limited due to their simple structure.

Multi-layer glass systems are used to meet the higher requirements that are generally demanded today. They are produced offline under vacuum in the magnetron sputter process.

GUARDIAN therefore focuses solely on the coating technology described below.

1.3.2 Magnetron process

The magnetron process has many appellations, one of which dates back to the beginning of this technology when this process was termed softcoating, as opposed to hardcoating. Today, this definition is misleading, since extremely resistant magnetron sputter films now exist which are in all cases composed of individual ultra-thin layers of film.

No other technology is capable of coating glass so smoothly and with such outstanding optical and thermal properties.

The material (i.e. the target, which is a metal plate) that is to be deposited on the glass surface is mounted on an electrode with a high electrical potential. Electrode and target are electrically isolated from the wall of the vacuum chamber. The strong electrical field (fast electrons) ionizes the sputter gas argon. The accelerated argon ions are capable of breaking off material from the target by colliding with it, and this then comes into contact with the glass where it is deposited onto the surface.

Metals and alloys are sputtered with or without additional reactive gases (O₂ or N₂). It is now possible to deposit metals, metal oxides and metal nitrides.

1.3.2.1 Typical assembly of a Magnetron-Sputter-Coater

- e.g. silver and nickel chromium
  - Responsible for the reflection of long wave and short wave radiation
  - Strong influence on heat transfer (U-value), energy transmission (g-value) and light transmission

Protection layer:
  - Protection of the functional layer against mechanical and chemical influences

Bottom and top layer:
  - Influences the reflectance, transmittance and colour of the coating
  - Silicone nitride top layer for very high mechanical durability

Functional layer: