One step antiglare sol–gel coating for screens by sol–gel techniques

C. Schelle, M. Mennig, H. Krug, G. Jonschker, H. Schmidt *

Institut für Neue Materialien, In Stadtwald, Gebäude 43, Saarbrücken, Germany

Abstract

Anti-reflection coatings on glass substrates were prepared by the application of sol–gel coatings of silica containing crystalline tin oxide particles. The sol was coated on commercially available float glass by dip-coating and spin-coating techniques. Increasing the surface roughness of the float glass (factor of 50) by a particulate coating material resulted in anti-reflection effects comparable to frosted glass. Haze and clarity, which are a measure for the contrast and definition of an optical system, were determined and compared to the uncoated and the commercial frosted glass. The reflectivity of the float glass could be reduced from 9% to 3%, which is a factor of two better than that of glasses.

1. Introduction

Due to the difference of the index of refraction between mineral glass and air, reflection losses of about 4% for each interface of a glass have to be accepted. When mineral glass is used for glazing of paintings, the optical appearance of the painting is disturbed by unwanted reflections, which are angle dependent and which are enhanced by bright daylight or artificial illumination. A simple method to reduce these reflections is to generate a light scattering effect by roughening the surface [1]. Antiglare glazing based on this light scattering effect in general are produced by hydrofluoric acid etching or by leaching of the glass surface in alkaline solutions. Both, hydrofluoric etching as well as NaOH etching causes waste to be disposed or processed to suitable products, and waste water cleaning procedures have to be established in order to meet the regulations, leading to costly processes. To overcome these problems investigations have been conducted to develop an antiglare coating. As a coating technique, the sol–gel process is of interest, since it is possible to prepare inorganic glass-like coatings on glass surfaces [2]. However, to produce sufficient surface roughness, it was found necessary to prepare layers of several μm thickness to produce sufficient light scattering [3]. As investigations in the sol–gel field have shown [4], it is difficult to prepare films above 1 μm thickness by the alkoxy route. Modified routes as shown by Yamane [5] do not seem to result in a technique suitable for large-scale fabrication. Results reported from Mennig et al. [6], using colloidal silica as an additive seemed to be a suitable basis, but led to smooth, transparent coatings.

Based on these results, it seemed to be an interesting route to use the described colloidal sols as binder for particles to obtain an appropriate surface roughness.

* Corresponding author. Tel.: +49-681 302 5013; fax: +49-681 302 5223.

2. Experimental

To obtain sufficient roughness for reducing reflection within a sol–gel coating, two different routes are possible. To disperse sub μm particles within a sol–gel matrix or to grow particles to a size of several hundred nanometer to one micrometer during the sol preparation. The particles by themselves should not have any light absorbance within the visible region. For this reason, the usefulness of SnO_2 as particulate system was investigated since the zeta potential for SnO_2 at low pH leads to destabilization of a SnO_2 sol. SiO_2 sols, especially if coreacted with $CH_3Si(OR)_3$ can be kept stable at low pH as described in detail [6]. If it is possible, to grow the SnO_2 particles to a desired size either by growth or appropriate agglomeration, a system providing rough surfaces after coating should be possible. To obtain destabilization, nitric acid was added to a mixture of MTEOS (methyl triethoxy silane) and TEOS (tetra ethoxy silane) and SnO_2 sols prepared as described in the experimental part. Precipitation of the SnO_2 takes place through an agglomeration process. The particle size was not within the desired range through this process, so an aging step was added at 0°C. After the aging, the SnO_2 particle size seemed to be suitable, as shown in Fig. 1.

The SnO_2 content of the sol was chosen to be about 12 wt%, which is below the percolation limit to avoid a packing which reduces the roughness. Coatings on floatglass were obtained by dipping and crackfree layers of 2 μm in thickness were obtained
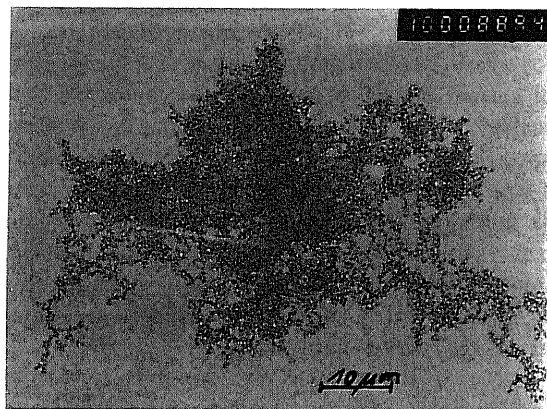
Fig. 1. TEM investigation of the particle size of the SnO_2 in the TEOS/MTEOS sol after 1 h aging at 0°C.
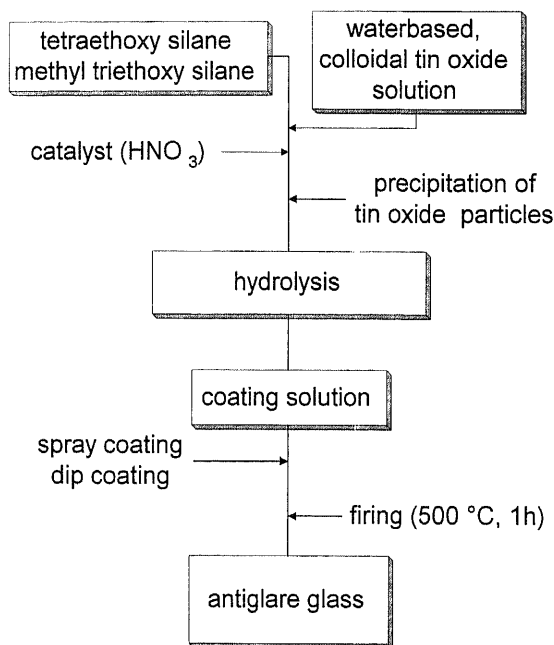
Fig. 2. Draft of the preparation of antiglare sol–gel coatings on glass.

at 500°C curing temperature. The whole process is shown in Fig. 2.

Colloidal tin oxide solution (15% solid content, 10–15 nm particle size [7]) was added to a mixture of MTEOS and TEOS as used in Ref. [6] in molar ratio of $SiO_2:SnO_2 = 15:1$. Nitric acid (65 wt%, 3 vol% of amount of TEOS) was added as catalyst for hydrolysis. Subsequently the solution was cooled in ice water and aged for 1 h.

Float glass plates (2 mm thick), of dimension 10×10 cm^2, were used as coating substrates. The glass samples were cleaned in an alcoholic solution of an alkaline glass cleaner (Hellmanex II) for 3 h, afterwards rinsed with water and dried at 80°C. The dried samples were dipped in the solutions and withdrawn at rates of about 3 mm/s or spray coated after diluting the coating sols with ethanol (1:1). The coated samples were dried at 80°C for 1 h, and heated to 500°C (1 K/min) and kept at this temperature for 1 h.

The thickness of the coatings was measured by profilometer scanning after producing a scratch deep enough to penetrate the coating. Profilometer analysis also was used to determine surface roughness.
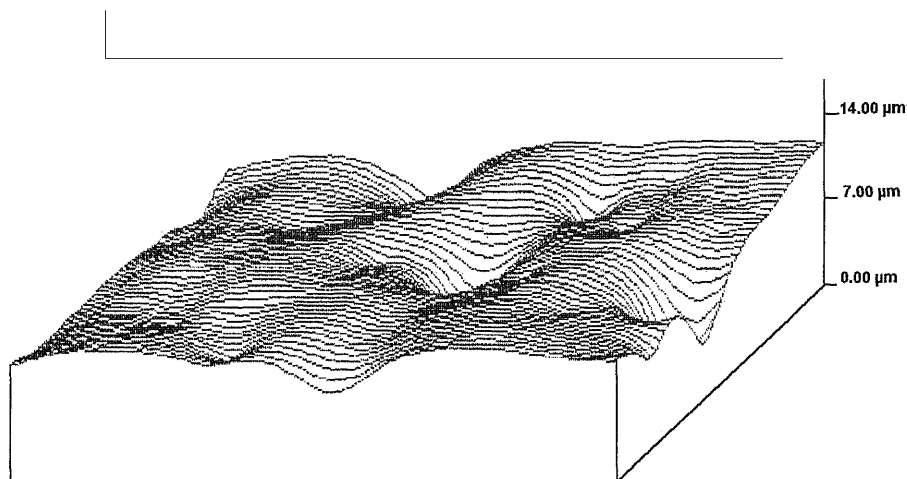
Fig. 3. 3D surface scan of state of the art antiglare glass used as reference on an area of 1 mm² obtained by profilometer scanning.

Transmittance, haze and clarity were determined by using a hazegard plus (BYK-Gardner). To investigate the directed spectral reflection a sample was mounted into a UV/VIS spectrometer (Cary UV/VIS, Varian), replacing one of the mirrors. An angle of incidence (Θ) of 7° was chosen and the wavelength of the incident beam was varied from 400 to 800 nm. Investigation of coloring index was carried out by diffuse reflection measurements, using a light beam (pulsed xenon lamp, wavelength 360 to 740 nm) at an incidence angle of 8°, with a white ceramic plate as calibration standard (Macbeth Colour-Eye 3100, Kollmorgen instruments).

Adhesion was tested by cross hatch cut and tape peeling test. Chemical resistance against organic solvents was tested (acetone, ethanol, iso- and n-propanol and common household cleaners) by exposing the coating to 1 ml of the solvent placed on the coated surface at room temperature for 5 min. After removal of the solvent the coating was investigated visually for defects.

3. Results

Light scattering antiglare coatings with a surface roughness of 1.6 μm were obtained. Figs. 3 and 4 show 3D profiling scans of the surface of the sol–gel coated glass in comparison to the surface of a commercial antiglare glass. To get a visual impression of
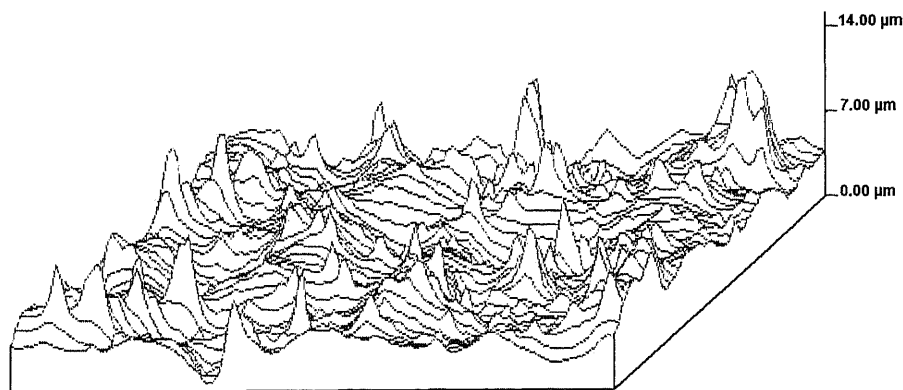
Fig. 4. 3D surface scan of sol–gel coated glass after densification at 500°C on an area of 1 mm² obtained by profilometer scanning.
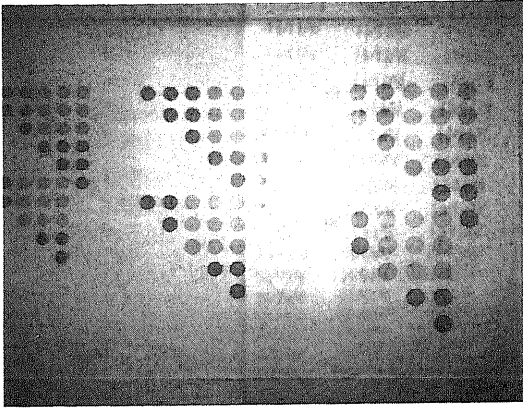
Fig. 5. Graphics behind partial coated (left) glass screen, showing strong reflection on the uncoated part. The optical impression of the picture is not disturbed by the coating.

Fig. 7. Directed reflection of the coated glass compared with unmodified glass and state of the art antiglare glass in dependence of the used wavelength.
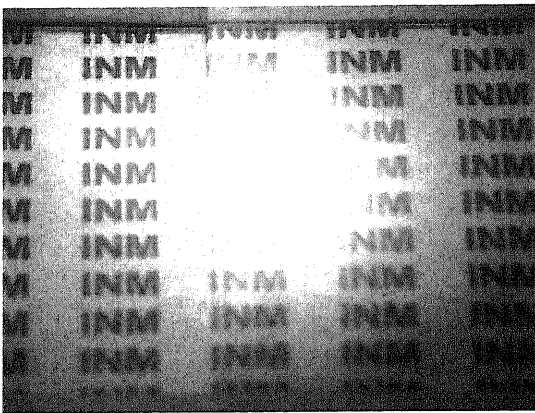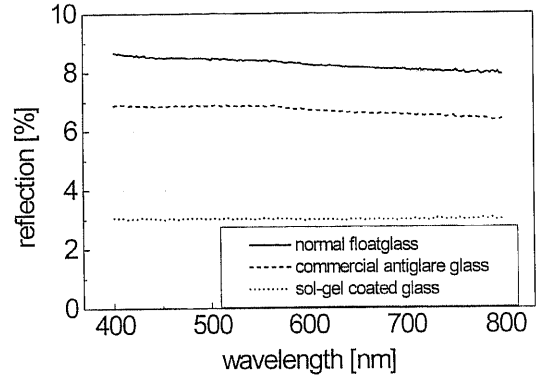

Fig. 6. Text behind partial coated (left) glass screen, showing strong reflection on the uncoated part. The image of the text is not blurred by the coating.

the antiglare effect, in Figs. 5 and 6 a graphic picture and a text are shown behind partially coated float-glass with a coating thickness of about 2 μm. No disturbance of the optical impression of text or a graphic picture is observed.

The adhesion (cross cut and tape test) classified in a scale from 0 to 5, showed 0 as the best result. Chemical resistance test showed, that after 5 min application of the chemicals as described in the experimental part, no visible damage in the coating was detected.

The transmittance, haze, clarity, reflectivity and coloring index of the coated glass in comparison to the commercial antiglare glass and to floatglass are summarized in Table 1.

The directed spectral reflections between 400 and 800 nm of the sol–gel coated glass, the commercial

Table 1
Properties of the single side sol–gel coated glass compared with commercial antiglare glass and unmodified float glass

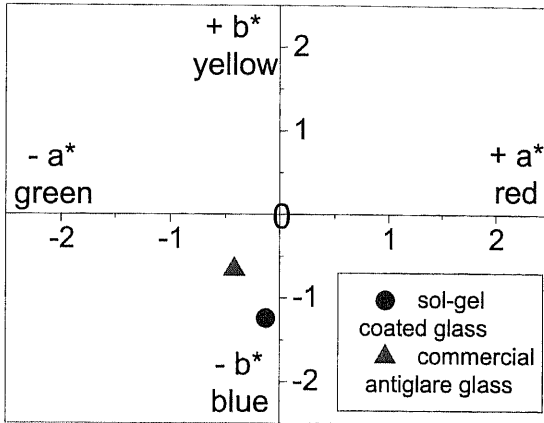| Properties	Sol–gel coated glass	Commercial antiglare glass	Unmodified float glass
Transmittance	94% ±0.05	92% ±0.05	93% ±0.05
Haze	16% ±0.3	1.5% ±0.12	0.13% ±0.02
Clarity	65% ±1.02	69% ±0.51	100% ±0.01
Directed spectral reflection	3%	6–7%	8–9%
Height root mean square	1.62 μm ±0.15	1.06 μm ±0.1	0.03 μm ±0.009

Fig. 8. The coloring index of the coated sample compared with state of the art antiglare glass shown in an a*b* color index representation.

antiglare glass and of untreated floatglass are shown in Fig. 7.

Fig. 8 shows the coloring index of the sol–gel coated glass in comparison to a commercial antiglare glass according to DIN 6174.

4. Discussion

4.1. Surface roughness

The reflectivity of a surface is determined by the surface roughness. When light is reflected on a rough surface, phase shifts depending on the surface roughness are observed.

A direct correlation between the phase shift and the root mean square height (HRMS) can be found according to Eq. (1) [3]:

$$\sigma_\Phi = \frac{2\pi}{\lambda} \sigma_h \cos \Theta, \qquad (1)$$

where σ_Φ is the phase shift, σ_h is the HRMS of the surface roughness, λ is the wavelength of the incident light and Θ is the angle of incidence. This correlation was taken for calculating the required surface roughness (HRMS), by setting the angle of incidence as 0 ($\cos \Theta = 1$). For an ideal reflective surface with a phase shift $\sigma_\Phi = 0$ and the complete information is preserved in the reflected image, a

surface roughness of $\sigma_h = 0$ is required. For an ideal scattering surface, where the information concerning the image projected onto the surface is lost in the reflected light, a phase shift of 2π is required. Therefore a HRMS of about 0.4 to 0.8 μm is necessary for the visible region from 400 to 800 nm.

First tests of the antiglare glass covering a painting showed almost no difference in the appearance compared to a commercially available HF etched one. The 3D profiling however showed that the commercial antiglare glass had a wavy surface (Fig. 3), whereas the sol–gel sample is rough (Fig. 4).

A surface roughness of $\sigma_h = 1.06$ μm was obtained for the reference, while the coated sample had a HRMS of $\sigma_h = 1.62$ μm. This value for the coated sample, exceeds the above calculated values (0.8 μm) necessary for an ideal scattering surface. Comparing these results with the roughness of an unmodified flat glass ($\sigma_h = 0.03$ μm) shows that the sol–gel coating increased the surface roughness by a factor of 50 and seemed to be in contradiction to the appearance of a painting or a written text as shown in Figs. 5 and 6.

For this reason, the optical data have been determined in comparison to the commercial glass.

4.2. Optical properties

The results in Table 1 show, that the clarity of the sol–gel sample is less and the haze is larger than that of the commercial antiglare glass, while the reflectivity of the sol–gel sample is less than that of the reference. Regarding the overall effect, however, the larger haze is compensated by the smaller reflectivity leading to a satisfying appearance of paintings or writings behind the glass. In Fig. 7 the reflection of the sol–gel coating between 400 and 800 nm wavelength is compared with untreated floatglass and commercial antiglare glass.

Coloring index measurements were made, to assure that no change in colors takes place by using the sol–gel coating in front of paintings, using state of the art techniques (DIN 6174) (Fig. 8).

The results show, that the coating almost completely compensates the green segment of the spectrum resulting from the floatglass at expense of gaining in the blue. This shift however cannot be

detected visually and does not change the appearance of a painting.

5. Conclusions

The investigations have shown, that the sol–gel process provides a way to produce an antiglare coating on top of floatglass. The chosen composition, particle size and the resulting roughness lead to an overall result suitable for applications. It can be varied to other desired properties. Since dipcoating techniques are well established in industry, the process can be scaled to a large scale coating technique.

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