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Low-emitting transparent coatings based *on tin doped indiumoxide applied via a* sol–gel routine

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ABSTRACT

Outgoing from inorganic metallic precursors improved sol–gel processes for the deposition of transparent conductive oxides, such as indium tin oxide (ITO) on different substrates have been developed. Coatings could be obtained by the dip-coating method and a subsequent annealing process for glass-substrates. For the coating of temperature-sensitive substrates, such as polycarbonate for instance, outgoing from an ITO-sol via a precipitation process nanopowders could be obtained. Film deposition at temperatures below 140 °C with these dispersed nanoparticles resulted in transparent and infrared reflecting coatings. For annealed glass-substrates the lowest attained emittance in the infrared was below 0.20, whereas transparency in the visible could be kept at about 0.80.

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1. Introduction

In many practical applications beside convection and solid conduction–even at ambient temperature–the influence of thermal radiative transfer on the total heat transfer cannot be neglected. For thermal radiative exchange between surfaces the emittance of the surface is of supreme importance. Low-emitting surfaces can drastically diminish the radiative heat transfer by either reducing the emission of infrared radiation or reflecting most of the incident infrared radiation. Transparent Conductive Oxide (TCO) [1] coatings based on wide band-gap semiconductors such as non-stoichiometric or doped oxides of indium, tin and zinc, for instance indium tin oxide (ITO), antimony doped tin oxide, fluorine doped tin oxide, aluminum doped zinc oxide etc. are used in numerous electronic applications. Examples are transparent electrodes in solar cells and display devices [2–5], electrochromic systems [6], light emitting diodes, heatable layers in defrosting windows [7], electromagnetic shielding [8], antistatic applications [8,9], gas sensing [10,11] and low-emissivity glazing [12] as well as oven windows [13]. Many of these applications are especially bound to the properties of the TCO-coatings like high electrical conductivity and high transparency of light in the visible region. In this work, the high reflectance in the infrared spectral region combined with high transmittance in the visible wavelength region of sol–gel deposited coatings is of peculiar interest. In general, low-e-coatings can be divided into soft coatings and hard coatings [14–16]. "Soft"coatings, e.g. sputtered thin silver or ITO layers often show superior properties in regard to conductivity, transparency and reflectivity but their low mechanical and chemical durability makes it necessary to provide an extra protection which can be a decisive drawback for some applications. "Hard" coatings, e.g. pyrolytic deposited doped metal oxides show improved mechanical and chemical stability so that they are stable towards atmospheric conditions, chemically inert and mechanically hard. Altogether they are more stable with an improved long term behaviour. The deposition of hard coatings can take place, for instance, via Chemical Vapour Deposition, Spray Pyrolysis or–as in this work–via Sol–Gel Processing. A Sol–Gel routine based on inorganic metallic precursors has been chosen, as it requires only simple techniques and equipment for the film deposition. Here the dip-coating technique was used. Some other advantages consist in the good adherence between film and substrate, the easy control of doping levels and the deposition on complex shaped substrates. That could be the coating of an inner side of a tube, for instance, what cannot be easily achieved by other deposition techniques. The aim of this work is to give a detailed description of the sol–gel synthesis of ITO-coatings on glass with improved infrared-optical properties.

2. Experimental

For the preparation of the ITO-Sol inorganic precursors of the metals could be used. The details for the preparation of the ITO-sol are given in Fig. 1. Indium nitrate ($In(NO_3)_3$, purity: 99.99%) and hydrated stannic chloride ($SnCl_4 \cdot 5\ H_2O$, purity: 98%) in the atomic ratio 80:20 were dissolved in a mix of ethanol and acetone (50:50) which resulted a solution with a concentration of 10% Indium. In order to stabilize the

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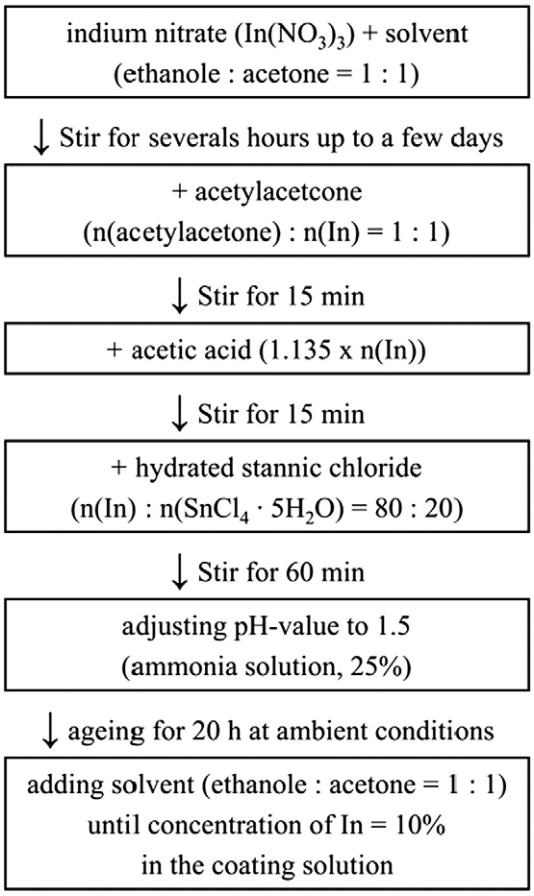

Fig. 1. Flowchart of the ITO-sol preparation.

solution, certain amounts of acetic acid and acetylacetone as chelating agents were added. Ammonia solution (25%) was used to adjust the pH-value at 1.5. After ageing at ambient atmosphere for 20 h the stable sol was stored under exclusion of air in a dark place. Pieces of glass slides (soda lime glass, 76 mm×26 mm×1 mm) were used as

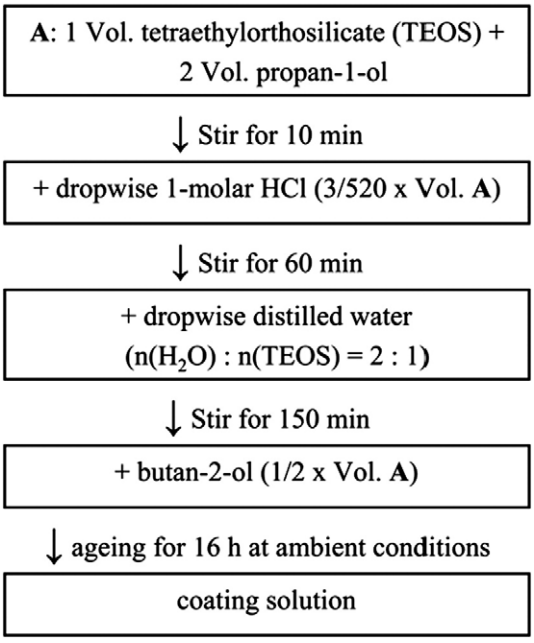

Fig. 2. Flowchart of the silica-sol preparation.

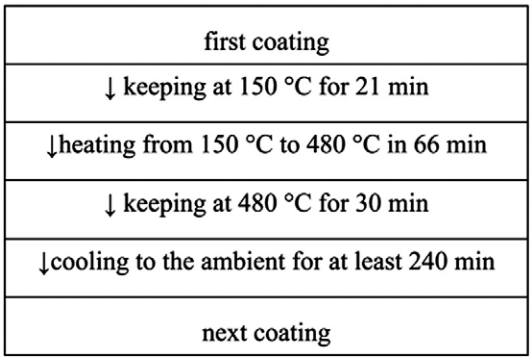

Fig. 3. Flowchart for the annealing process 1 as final treatment for the silica coating and as intermediate treatment for the ITO-coatings.

substrates, which were cleaned in an ultrasonic bath (2% extran solution in distilled water) and rinsed with distilled water and acetone. A silica barrier layer had been deposited on the glass slides in order to inhibit the diffusion of sodium ions into the functional coating [17,18]. Therefore, a silica-sol was prepared outgoing from tetraethylorthosilicate, which was mixed with propan-1-ol and butan-2-ol as solvent and hydrolysed with hydrogen chloride as catalyst. The details for the preparation are given in Fig. 2. The stable sol was stored under exclusion of air in a dark place. As deposition technique for the ITO and silica coating the dip-coating technique in a dust-free ambiance was used. The thickness of the coatings can be controlled by the withdrawal speed. A quantitative description therefore is given by the Landau–Levich equation [19]

$$d = 0.94 \cdot \frac{(\eta \cdot v)^{2/3}}{\gamma^{1/6} \cdot (\rho \cdot g)^{1/2}}, \quad (1)$$

where η means the viscosity of the sol, γ the interfacial tension between sol and air, ρ the density of the sol, g the gravity acceleration, d the thickness of the film and v the withdrawal speed. In the case of the silica layer, the optimal withdrawal speed was found to be 14 cm/min. The withdrawal speed in the case of ITO was 20 cm/min. A moderate heating of the sol to 25 °C was applied, as it is described in [20] to overcome problems caused by evaporation cooling and to obtain high quality sol–gel thin films, especially on thin or enlarged substrates.

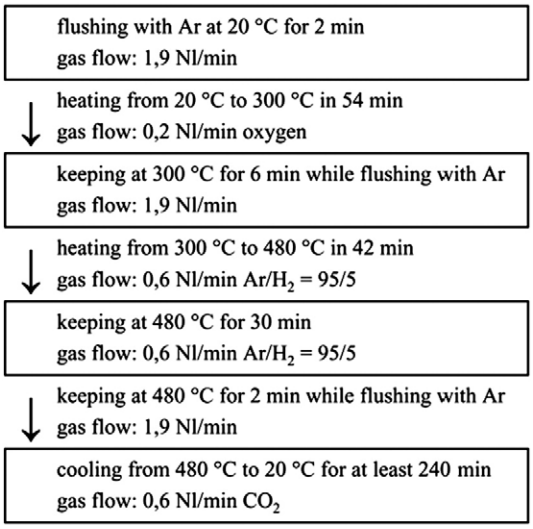

Fig. 4. Flowchart for the annealing process 2 as final treatment of the ITO-coating.

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