



A contribution to climate protection - Electrochromic windows fabricated with the sol-gel technology

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were analyzed. In difference to this, the evaluation of the L spectra results in a significant systematic error (> 10% relative). It may be explained with uncertainties in the computation of the ionization cross section at the high excitation energy.

Summary

Comparative chemical analyses carried-out by standardless TEM/EDXS and SEM/EDXS analytical procedures have shown the resulting compositions of Na_3AlF_6 , ZrSiO_4 and InSb agree within acceptable confidence ranges with certified data. The evaluations of selected materials consisting of lower and higher Z components reveal the analytical strategy using the TEM is – related to the SEM – much more sensitive against imponderable parameters of excitation and propagation of X-radiation in the specimen chamber. The micro-morphological structures of the materials can be verified in the SEM more precisely. Therefore, as far as it is possible from properties of the materials and methodical circumstances the analytical SEM should be accompany spectrometric analyses in the TEM. In comparison with SEM/EDXS an advantage of the TEM consists in the possibility to evaluate higher energetic X-radiation (> 20 keV). In this

way the determination of the spectral intensities – as an important step of the quantification – becomes more certain because of the presence of energetic separated, non-overlapping spectral lines.

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Electrochromic systems - Introduction

Electrochromic (EC) windows, also called “smart windows”, change their optical properties (transmittance or reflection) in a reversible manner when a voltage is applied and a current flows through them [1, 2]. Large EC glazing are of considerable interest for architectural, automotive and aeroplane applications in order to control the solar radiation entrance to save energy costs for air conditioning in summer and for heating in winter (especially for buildings and automobiles) and also to add comfort factors like privacy and to avoid glare and fading.

Further applications for EC systems are EC displays and self-dimming rear-view mirrors for

Weight % by Element, SEM			
Spectr. Line	O K	Si K	Zr L
Mineral Data	34.91	15.32	49.77
Mean Value	36.43±4.04	15.73±0.91	47.83±3.13
Weight % by Element, Oxygen by Difference, TEM			
Spectr. Line	O K	Si K	Zr K
Mineral Data	34.91(1.0)	15.21(1.7)	49.92(6)
Mean Value	30.07(6)	19.31(7)	50.86(9)
Weight % by Oxide, SEM			
Component, Radiation	SiO ₂ , Si K	ZrO ₂ , Zr L	
Mineral Data	32.78	67.22	
Certificate	33.57	65.93 (+1.27 HfO ₂)	
Mean Value	34.31(0.05)	65.69(0.05)	
Weight % by Oxide, TEM			
Component, Radiation	SiO ₂ , Si K	ZrO ₂ , Zr K	
Mineral Data	32.73(8)	67.3(5.6)	
Mean Value	32.73(8)	67.3(5.6)	
Remark: Hf has not been detected, neither with TEM- nor SEM-EDXS!			

Table 2: Zircon, ZrSiO_4

Weight % by Element, SEM		
Spectral Line	InL	SbL
Certificate (synth. Compound)	48.51	51.46
Mean Value	49.87± 0.30	50.13± 0.30
Weight % by Element, TEM		
Spectral Line	InK	SbK
Mineral Data	50.11±0.2	49.9(0.2)
Mean Value	50.11±0.2	49.9(0.2)
Weight % by Element, TEM		
Spectral Line	InL	SbL
Mineral Data	43.6±1.8	56.4±1.8
Mean Value	43.6±1.8	56.4±1.8

Table 3: Indium Antimonide, InSb



cars, which were already on the market for several years (Gentex, Magna Donnelly). Examples for electrochromic materials are inorganic complexes (e.g. Prussian blue), organic molecules (e.g. viologens => application in EC rear-view mirrors), organic polymers (e.g. polyaniline, PEDOT) and a large amount of transition metal oxides (e.g. tungsten oxide (WO_3), niobium oxide (Nb_2O_5) and nickel oxide (NiO).

The most studied EC material is the tungsten oxide (WO_3) due to its high coloration efficiency. The cathodic electrochromic WO_3 colours reversibly from transparent to dark blue by reduction and simultaneously insertion (intercalation) of small ions (e.g. H^+ , Li^+) from the electrolyte and bleaches by oxidation and extraction of the ions.

There are three major configurations of EC windows: battery-like, solution phase and hybrid structures. A typical configuration of a battery-like electrochromic (EC) device is shown in figure 1. It consists of a five layer system: between two glass substrates, coated with a transparent electrically conducting layer (e.g. ITO or FTO), are an electrochromic coating (EC1,

e.g. WO_3), an ion-storage (IS) coating or a second EC-layer (EC2) and in between an ion conducting electrolyte (e.g. Li^+ , H^+).

After applying a voltage with negative polarization at the side of the WO_3 -layer, WO_3 is reduced and Li^+ ions diffuse into the WO_3 -layer leading to a deep colored Li_xWO_3 . After change of the polarization Li_xWO_3 is oxidized and the Li^+ -ions diffuse through the electrolyte into the ion-storage layer. The ion-storage layer should either not change its transmittance during the intercalation of ions or should have anodic electrochromic properties (EC2, as e.g. nickel oxide or Prussian blue, which colours after oxidation and bleaches after reduction), which intensify the coloration of the EC-device.

This configuration is often used for prototypes of large area EC-windows for architectural or automotive use and also the EC-windows developed in the INM have this configuration [3]). There also exist monolithic systems, in which all layers were deposited on one glass substrate e.g. by vacuum deposition method (Saint-Gobain glass [4], SAGE Electrochromics [5]).

As EC-layer commonly blue coloring WO_3 is used. In the INM-Leibniz-

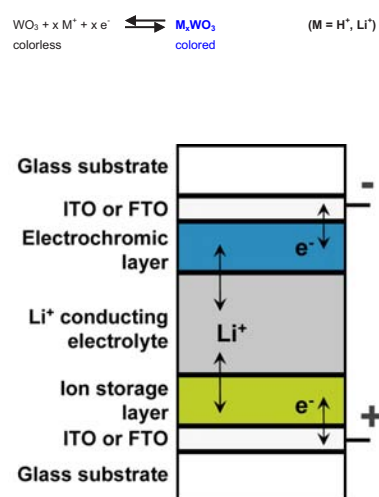


Fig. 1: Scheme of a battery-like EC-device (ITO, FTO: transparent conducting coatings of $In_2O_3:Sn$ and $SnO_2:F$)

Institut für Neue Materialien blue, grey and brown coloring EC-layers were developed based on tungsten oxide (WO_3) and niobium oxide [6] (figure 3). As counter electrode either a non-coloring ion-storage layer (E-Control [7], INM [3]) or an anodic electrochromic layer is used (Prussian blue, Gesimat [8]).

The layers of the EC-device can be deposited using different techniques:

- vacuum deposition method (e.g. E-Control; Saint Gobain glass, Sage-Glass®)
- electrodeposition method (e.g. Gesimat GmbH)
- sol-gel deposition method (INM - Leibniz-Institut für Neue Materialien)

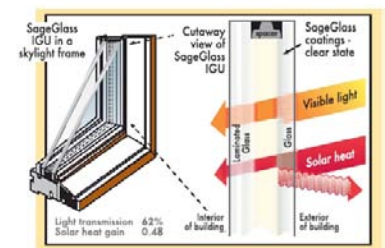
For these methods the investment for production machines is very different.

As electrolytes different materials were used: The company E-Control uses a polymer electrolyte, the INM uses an inorganic-organic composite electrolyte [3] which is thermally solidified after filling into the EC-device. The Gesimat GmbH uses an ionic conductive PVB (*polyvinylbutyral*) foil, which gives the EC-windows similar properties as laminated sheet glass (LSG) [8], which is advantageous. The advantage of the

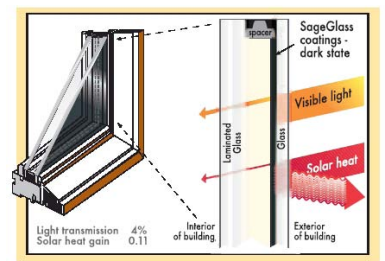
sol-gel technology used in the INM is, that this technology can be used for the deposition of coatings on bent glass [9], which is important for the fabrication of EC-windows for automotive application, and that the investment costs are relatively low.

Properties and energy saving

By applying a small voltage ($< 3 \text{ V}$) the transmission of an EC window can be changed in the visible (VIS) and near infrared (NIR) range, whereby it remains transparent. Thus colored EC-windows do not only keep out a part of the visible light but also a part of the solar heat (figure 2). The bleached and colored state and the states between are stable for a long time without a voltage supplied. Therefore only for the change of the transmission a voltage is applied and a current flows through. Due to this the energy consumption of EC windows is very small ($0.04 - 0.1 \text{ Wh/m}^2$). The switching of the EC windows can be done manually or automatically. The switching times were dependent on the size of the EC-windows and the temperature and lie in the range from 2 to 10 min, which is acceptable for architectural glazing.



Light transmission (TL): 62 %
Solar heat gain: 48 %



Light transmission (TL): 4 %
Solar heat gain: 11 %

Fig. 2: Functionality of EC windows ([10])



Due to the switchable transmission of the VIS- and NIR light EC windows can reduce the peak electricity demand for cooling during summer and heating during winter, thus contributing towards the reduction of greenhouse gases. A study of Lawrence Berkeley National Lab on SageGlass® EC windows for their energy saving potential as well as their effect on human factors has shown that the use of SageGlass windows results in up to 20% cooling energy savings, up to 60% reduction in lighting, and up to 30% reduction in peak electricity demand [10]. On the human factors side, the people evaluated said they overwhelmingly preferred to be in the room with SageGlass windows vs. the room with static glass windows. Better thermal insulation provided, it will also reduce the amount of space heating required during winter. Furthermore, such a glazing will facilitate better day lighting of buildings and offices which can also lead to significant cost savings due to the improvement of workers productivity [11]. Figure 3 shows the SageGlass® EC windows of this study with sensors inside the room monitoring the temperature and light transmission of the windows.

An energy life cycle inventory analysis was implemented to a 400 mm

x 400 mm prototype electrochromic window [12]. The raw material production and EC device fabrication processes were analyzed and the total embodied energy was calculated and compared with the energy savings during operation. It was found, that 0.7 years of operation are required to anticipate the embodied energy for the plain EC part.

Large area electrochromic glazing – State of the art in the architectural range

Various prototypes up to a size of 2000 mm x 1200 mm (Flabeg [13], E-Control-Glas [14]), 1200 mm x 800 mm (Firma Gesimat GmbH [8]) and 800 mm x 500 mm (INM gGmbH [15]) were fabricated and tested. The worldwide first electrochromic window for sun protection was sold by the company Flabeg in 2000. The EC-windows were installed e.g. in a glass facade of the “Stadtsparkasse Dresden” but due to switching problems of the EC-windows they were taken from the market in 2001 and taken into a “redesign phase”. The new founded company E-Control improved the EC windows and will bring their product on the market by August 2007 [16]. Recently only the EC-windows from the company Sage glass were on the market. The were

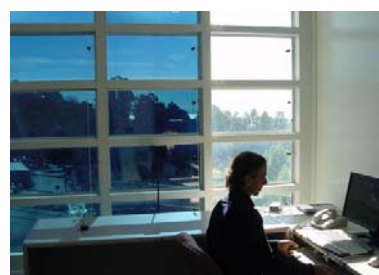


Fig. 3: SageGlass® EC windows in coloured and bleached state (with sensors inside the room monitoring the temperature and light transmission of the windows).

sold as roof windows from VELUX America up to a size of 1175 mm x 776 mm. Due to SAGE glass the high requirements for UV stability and thermal stability and cycle stability were fulfilled for the use of the windows in the architectural range.

For automotive applications Saint Gobain fabricated an electrochromic sunroof for a limited edition of Ferrari 575 which made its worldwide debut at the Detroit Motor Show in January 2005.

The price for an EC window from VELUX America is with 2000 €/m² comparably high and thus we are far away from a broad application of EC-windows in the architectural and automotive range. In order to reduce the energy consumption and the emission of greenhouse gases appreciably a broad application is preferable. Yet for a broad application the price should be drastically reduced. One way to reduce it could be the fabrication of EC-windows using the sol-gel technology of the INM.

Sol-Gel EC-windows of the INM

The EC-windows on the market and the prototypes of the industries mentioned above always color blue and use WO₃ as EC layer. The INM developed a wet coating technology for

the realization of blue, grey and brown coloring EC-windows [3, 17, 6] with the configuration shown in figure 1, in which the various colours were obtained by varying the EC-layer: The EC-layer consists of WO₃ for blue coloring windows, Nb₂O₅:Mo for grey coloring and Nb₂O₅:Li for brown coloring windows. As IS layer a non coloring CeO₂-TiO₂ layer and as electrolyte an inorganic organic composite is used.

The sol-gel layers were deposited on FTO-glass (K-glass) by dip-coating process and than heat treated at 240°C, 500°C, 400°C and 450°C to 500°C for WO₃, Nb₂O₅:Mo, Nb₂O₅:Li and (CeO₂)_x(TiO₂) layers respectively. After mounting of the EC devices, the electrolyte is filled into the device and solidified by thermal treatment (105°C). After cooling down the EC-windows were sealed using butyl rubber.

By applying a small voltage (up to 3 V) the transmission in the visible range can be changed continuously from 70 % down to about 25 % (T_L values) for blue coloring EC-devices and from 60 % to 20 % for grey coloring EC-devices (see figure 4, 5). Due to the change in the visible and the near infra red (NIR) range a high change of the total solar heat transmission (g-value, DIN EN 410) is

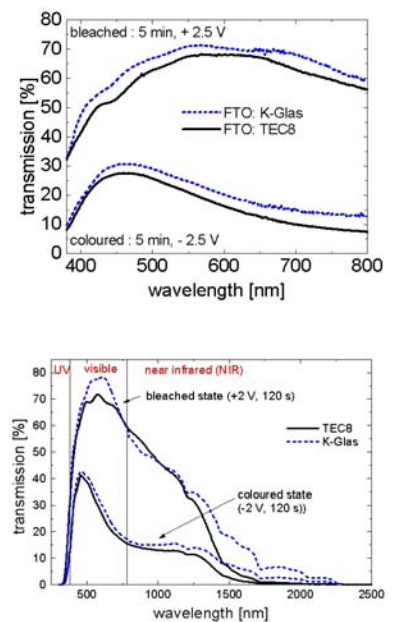


Fig. 4: UV-VIS- and UV-VIS-NIR spectra of blue coloring EC-windows with the configuration FTO / WO₃ / composite electrolyte / CeO₂-TiO₂ / FTO.



obtained varying from 42 % to 17 % so that the energy entrance into the buildings can be varied in a wide range. The switching time depends on the size of the window and lies between 2 and 5 min. The coloration is homogeneous over the whole area as shown in figure 6 where the transmission at 650 nm is shown as a function of the time during the coloration and bleaching process at different points of the EC-window (size: 500 x 800 cm). The voltage is only necessary to change the transmission of the EC window so that the energy consumption is small (0.1 Wh/m²). The windows shows an UV stability for 1000 hours (Heraeus suntest 800 W/m²), without change of the switching behaviour. The long-term switching stability could be improved from 15000 to > 60000 switching cycles for blue and grey coloring EC-devices by adding a small amount of water to the composite electrolyte [6, 18]. Prototypes up to a size of 500 cm x 800 cm and 300 cm x 400 cm were fabricated for blue and grey coloring EC windows respectively (figure 7).

Using the blue coloring EC system of the INM (tungsten oxide as EC layer) a curved EC window of the size 300 cm x 400 cm with a bending radius of 2000 mm was fabricated by bending the K-glass in an

oven at 600°C before the dip-coating process (figure 8). The dip-coating of the curved glass was successful and a homogeneous blue coloring bent EC-device was obtained.

Furthermore a patterned EC-window of 5 x 5 pixels with a size of 300 mm x 300 mm has been fabricated (figure 9). The WO₃ coating and the (CeO₂)_x(TiO₂)_{1-x} coating were patterned with the FTO coating below by laser ablation in 5 stripes. Each stripe was contacted at the FTO layer. Then the EC-device was mounted in that way that the stripes of the functional layers were perpendicular to each other in order to get a matrix display of 5 x 5 pixels (pixel size: 50 mm x 50 mm). Afterwards the electrolyte was filled in and solidified by heat treatment and the EC-window was sealed as described above.

The coloration and bleaching of the pixels were controlled by a PC program and a display interface developed at the INM (figure 9). Each pixel could be colored and bleached individually, but also different patterns as 4 different pixels together, horizontal or vertical stripes or the whole area could be colored and bleached together (figure 9). A structured EC-window could be used to show information as e.g. prizes in a shop window or a company logo in an

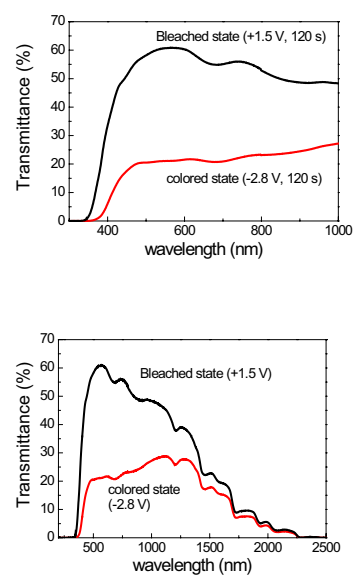


Fig. 5: UV-VIS- and UV-VIS-NIR spectra of a grey coloring EC-window with the configuration K-glass / Nb₂O₅:Mo / composite electrolyte / CeO₂-TiO₂ / K-glass.

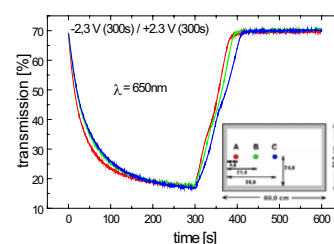


Fig. 6: Transmittance of a blue coloring EC-device (size 500 mm x 800 mm) at wavelength 650 nm as a function of the time during the coloration and bleaching process, measured at different places of the window (see inset).

office building. Because only for the change of the transmission energy is necessary, the information could be shown for a long time with low energy consumption.

The brown coloring EC windows with $\text{Nb}_2\text{O}_5:\text{Li}$ as EC-layer showed only a small transmission change from 72 % to 50 % and are therefore of small interest for the application [6]. Another form of brown coloring EC devices was studied with nickel oxide titanium oxide layers as EC-layers, $(\text{CeO}_2)_x(\text{TiO}_2)_{1-x}$ as ion-storage layer and 1 mol/l KOH in water. These windows showed a transmittance change from 65 % to 35 % (-1 V, 1 min/ +1.2 V, 1 min), but the long-term stability was relatively small with 7000 cycles [19].

Furthermore an ion-storage layer was developed which can be hardened at low temperature by UV treatment. This is advantageous, because the normally used $\text{CeO}_2\text{-TiO}_2$ IS-layer has to be heat treated at high temperatures (450 to 550 °C), which leads to a high energy consumption during the fabrication process and makes the layer not useful for the deposition on plastic substrates. The low temperature deposition was realized by development of $\text{CeO}_2\text{-TiO}_2$ nanoparticles, which can be redispersed in a solvent and after mixture with a

binder and a UV initiator the dispersion could be deposited by dip-coating and hardened by UV-treatment. Windows using this IS-layer showed a transmission change from 70 % to 30 % after 13 min [20], which shows that the kinetics is not as fast as for the standard heat treated $\text{CeO}_2\text{-TiO}_2$ layer, but a first step to fabricate EC-windows on plastic substrates has been done. The fabrication of EC-foil by the roll-to-roll process is an aim, which could be reached by development of UV-curable EC- and IS-layers and an UV-curable electrolyte

In cooperation with the university of Sao Carlos, Brasil (PROBRAL) the development of new cost-efficient electrolytes for EC-windows based on renewable primary products were done, whereby gelatine, starch and agar-agar were used for gelification and LiClO_4 , LiClO_3 or acetic acid were used for ion conduction. First results of EC-windows using the new electrolytes in EC windows of the configuration shown in figure 1 using WO_3 as EC-layer and $\text{CeO}_2\text{-TiO}_2$ as IS layer showed that a small transmission change (from about 60 % to 40 %) could be obtained. Further work has to be done to improve these results.



Fig. 7: Bleached and grey colored EC window of the INM (size 400 mm x 300 mm). The transmission in the visible range (TL value, DIN EN 410) can be varied from 60 % (bleached) to 20 % (grey colored, voltage < 3 V, switching time 2-3 min).



Outlook and comparison with other switchable glazings

The results show that the sol-gel technology is an alternative for the fabrication of EC windows on glass. It is suggested, that the fabrication of EC-windows with the sol-gel technology could reduce the price of EC-windows. However before the production process could start, some stability tests and climate change tests have to be done.

Another possibility for the fabrication of low cost EC-devices is the development of EC-foil which can be laminated on the architectural glazing of the buildings. A first step for fabrication of EC-windows on plastic foil has been done, but further work is necessary as the development of an UV-treatable EC-layer, a better UV-curable IS layer and a UV-curable electrolyte. An EC-foil could then be fabricated fast and cheap by roll-to-roll process and laminated on glass of different sizes and probably also on curved glass. This would reduce the price of EC-devices for architectural and automotive glazing drastically. Problems to be solved are the contact of the foil after the fabrication and the sealing.

Beside electrochromic systems described above, there are other switchab-

le glazings as e.g. gasochromic [21], photochromic and thermochromic coatings and suspended particle devices (SPD) and polymer dispersed liquid crystal (PDLC)-systems. The photochromic and thermochromic coatings color after irradiation or after increase of temperature respectively and cannot be influenced actively. The gasochromic systems consist of a WO_3 layer which colours after reduction with hydrogen gas. Problems are here the tightness of the system. The PDLC and SPD systems are already on the market (PDLC: SGG PRIVA-LITE, Saint-Gobain Glass [22], SPD: Research Frontiers [23]). The functionality of the devices depends on the orientation of liquid crystals or optical anisotropic particles in an electrical field after applying a voltage. Without a voltage applied they are opaque (PDLC) or dark blue colored (SPD) and only with an applied voltage the systems are transparent and clear, whereby the voltage is about 100 V for SPD devices. This leads to a higher energy consumption than EC-devices need and in addition no change of the solar heat transmission (g-value) is obtained, because only the visible part of the spectrum is change. Thus no energy saving effect can be obtained using SPD or PDLC systems.



Fig. 8: Curved blue colored EC window of the INM (size 400 mm x 300 mm, curve radius 2000 mm). The transmission in the visible range (TL value, DIN EN 410) can be varied from 70 % (bleached) to 25 % (blue colored, voltage < 3 V, switching time 2-3 min).

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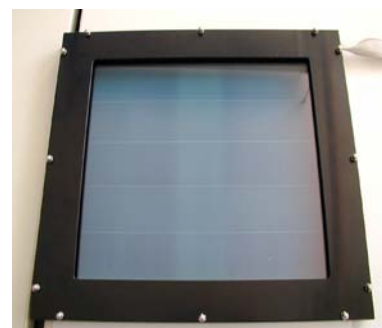
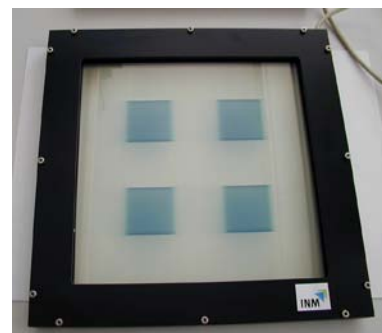
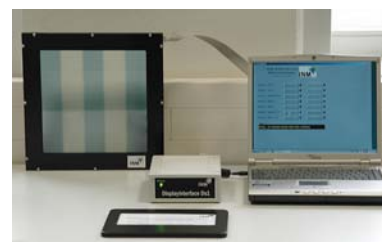


Fig. 9: Patterned electrochromic window with 5 x 5 pixels, whereby each pixel can be colored and bleached individually and different structures can be colored and bleached. Shown: EC-Display with control unit (laptop and display interface) and three colored stripes, four colored pixels and whole area colored.



Plasma-assisted Modulation of Morphology and Composition in Tin Oxide Nanostructures for Sensing Applications



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Detecting small quantities of gases and chemicals is becoming increasingly important for consumer, health and security applications such as monitoring the ecological constituents and to control the concentration of toxic and hazardous gases.[1] Nanostructures are especially attractive for detector and quantifier applications, particularly due to their high surface-to-volume ratio and higher sensitivity towards surface reactions, which results in charge penetration layers being comparable to nanostructure dimensions.[2] Tin oxide (SnO₂) represents the class of IV-VI com-

pound semiconductors with a wide band gap (3.6-4.0 eV) at room temperature and intrinsic n-type electrical conductivity.[3] Given their low electrical resistivity (10⁻²-10⁻⁴ Ωcm), high chemical resistance, thermal stability and mechanical strength,[4] SnO₂ nanostructures offer promising potential for improved chemical sensing behaviour due to its redox switching between different states, which facilitates a reversible transformation of the surface composition from Sn⁴⁺ cations on the surface into a reduced surface with Sn²⁺ cations depending on the oxygen chemical potential of the system.[5]

Tin oxide nanostructures have been synthesized by a number of methods such as chemical vapor transport at high temperatures,[6] thermal evaporation of tin oxide powders[7] and plasma enhanced chemical vapor deposition.[8] Although a large body of data is available on the synthesis of tin oxide nanostructures (particles, films, nanowires and nanobelts) in pure and doped compositions, synthetic pathways for their controlled growth and modification remains an overarching task. We have recently reported a molecule-based chemical vapor deposition (CVD) process for the synthesis of tin oxide and other semiconductor nanowires.[9-11]