Novel Material Properties Based on Flame-synthesized Nanomaterials

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Abstract

The principles of high-temperature reactive particle formation in flames are characterized by a sequence of partly interacting rate processes in the gas flow, while the necessary energy is delivered by the exothermic combustion reaction heating the flow to high temperatures. A complete description of the precursor decomposition kinetics and the subsequent oxidation/hydrolysis reactions is rarely obtained, while the properties of the products manufactured such as size, morphology, phase composition, and crystallography are decisively influenced by these parameters. A precise understanding and control of the initial steps is therefore required to open up the possibility of tuning particle properties.

In the present study, the formation of oxidic particles in flame reactors is presented. It will be shown that the stoichiometry and crystallography of oxides such as ZnO, SnO\textsubscript{2} and TiO\textsubscript{2}, and therefore their physical and chemical properties, can be adjusted depending on the reaction conditions. In addition to the synthesis of pure materials, coated particles as well as nanocomposites are accessible when a few requirements are fulfilled. In the case of immiscible oxides such as TiO\textsubscript{2} and SiO\textsubscript{2}, composites consisting of separate phases are produced, while the formation of composites from miscible compounds usually requires a two-step process that tends to produce poorly mixed materials. Nevertheless, in the case of kinetically controlled synthesis, a one-step formation of nanocomposites from miscible oxides can be realized when the kinetics of precursor decomposition and particle formation of the participating oxides are quite different. This results in materials that exhibit new properties according to the used oxides. As an example, the one-step formation of homogeneously dispersed superparamagnetic Fe\textsubscript{3}O\textsubscript{4} in fumed silica will be shown. Chemically, this material behaves like common silica but due to the superparamagnetic characteristics of the embedded iron oxide, it can be heated in a contactless manner by means of an alternating magnetic field. Applications focusing on contactless hardening and bonding become apparent.

Keywords: Flame synthesis, nanoparticles, TCO, nanocomposites, superparamagnetic Fe\textsubscript{3}O\textsubscript{4}, MagSilica®

1. Introduction

The formation of inorganic nanoparticles is used routinely today to produce a variety of bulk chemicals such as SiO\textsubscript{2}, Fe\textsubscript{3}O\textsubscript{4}, TiO\textsubscript{2}, Al\textsubscript{2}O\textsubscript{3} and ZnO, amounting to millions of tonnes a year. They are used industrially as reinforcements, pigments, stabilizers, catalysts or catalyst supports, flowing aids, and multiple other applications. The formation of nanoparticles can be divided into two generally differing methods: wet-chemical and gas-phase synthesis. While wet-chemical synthesis usually results in materials formed by thermodynamic control, gas-phase processes enable a kinetic control of nanoparticle formation. This method is therefore basically favored for the formation of metastable materials as doped nanoparticles and nanocomposites. As there is a variety of different reactor concepts such as hot-wall reactor, plasma reactor and laser reactor, the bulk synthesis for nano-sized inorganic particles is covered by flame reactors because they enable a cost-effective and versatile industrial process with high production rates. Moreover, flame reactors are designed for a broad range of operating conditions for the control of particle size, morphology and composition. Some useful studies on the flame synthesis of nanoparticles can be found in...
Due to an increased understanding of size-dependent nanoparticle properties and probable applications thereof, an increasing demand for synthesis methods for the formation of specific nanoparticles with tuned properties has evolved. As the flame reactor is the workhorse for the gas-phase formation of nanoparticles, this paper addresses different possibilities for the formation of nanomaterials with specific properties by flame-synthesis. While we are interested in a precise understanding of the physics and chemistry of the oxidic particles themselves, impurities were neglected as far as possible. Because, for instance, Spicer et al. have already studied the possibilities for the formation of SiO2/C nanoparticles in a premixed acetylene/oxygen flame, we tried to avoid uncertainties resulting from such carbon-containing flames and complex hydrocarbon flame chemistry probably interacting with particle formation. Therefore, most of the results shown were obtained from experiments carried out in a low-pressure, premixed H2/O2 flame. The particle formation and growth were observed by means of a particle mass spectrometer (PMS) for the first few centimeters downstream of the flame-stabilizing burner head. A typical result of such a PMS measurement taken during the formation of SnO2 nanoparticles is shown in Fig. 1. Both PMS measurements and TEM investigation of thermophoretically sampled particles are always found to be in good agreement with respect to the mean particle diameter $d_p$ and the standard deviation $\sigma$.

Typically, a reasonable particle growth is observed starting from about 80 mm downstream of the burner head (flow coordinate, height above burner), as shown in Fig. 2. The specification of the PMS enables measurement of the particles’ size between 2 and 15 nm.

For further investigation, the as-prepared materials were usually sampled by thermophoretic deposition of the particles downstream of the reactor either on TEM grids or on a cooled substrate.

### 2. Oxidic Semiconductors

Semiconducting oxidic nanoparticles such as tin oxide and zinc oxide are seriously discussed as transparent conducting oxides (TCOs) for the formation of transparent conducting layers as they are used in flat panel displays and solar cells, while titania is used as the electron-conducting electrode in dye solar cells, the so-called Grätzel cells. Up to now, the market for TCOs has been dominated by indium tin oxide (ITO) and fluorine-doped tin oxide (FTO), mainly prepared by chemical vapor deposition (CVD), physical vapor deposition (PVD) and sputtering. These methods usually require expensive, low-pressure process steps and have the disadvantage that a lot of the material is not deposited on the substrates but is lost as so-called off-spray. Printing methods using nanoparticles such as ink-jet printing or roll-to-roll printing would allow minimizing the waste of the TCO, but these methods are still not able to completely resolve the issue of the high cost of ITO. In addition, the electrical as well as the optical performance of printed layers based on nanoparticles mostly do not meet the requirements for TCO layers. This is mainly due to the two following drawbacks:

1. Agglomerates and bigger nanoparticles exceeding a size of about $1/10^{9}$th of the wavelength of the visible light decrease the transparency of a
In accordance with several oxides are discussed. The influence of the flame conditions on the stoichiometric $\Phi$ ratio is defined as the mol of oxygen supplied divided by the mol of oxygen required for complete combustion. For all experiments, the fuel-to-oxygen ratio $\Phi$ is defined as:

$$\Phi = \frac{\text{mol of oxygen required for complete combustion}}{\text{mol of oxygen supplied}}$$

As a result, the adjustment of specific properties with respect to the electrical properties requires a tuning of the particle composition, especially the oxygen content during particle formation. Several authors have shown that the fuel-to-oxygen ratio of flames has an important influence on nanoparticle properties such as morphology, size, crystallinity and stoichiometry. In their works, the influence of the flame conditions on the stoichiometry and therefore on the electronic properties of several oxides are discussed. In accordance with, for all experiments the fuel-to-oxygen ratio $\Phi$ is defined as:

$$\Phi = \frac{\text{mol of oxygen required for complete combustion}}{\text{mol of oxygen supplied}}$$

This means for $\Phi < 1$ that the flame is operated at lean conditions while $\Phi > 1$ corresponds to rich conditions.

 Whereas in industrial synthesis processes $\Phi$ is usually fixed at a value around 0.5, our experiments were performed for values of $\Phi$ between 0.25 and 0.97 to investigate the influence of the fuel-to-oxygen ratio on the nanoparticles properties. Experiments performed at $\Phi$ close to one showed that almost no tin oxide or zinc oxide could be produced from organometallic precursors under this condition. Tin is known to exhibit stable oxidation states of 2 and 4, however, oxidation states that lie between 2 and 4 have been identified. X-ray diffraction (XRD) measurements of the powder received from synthesis at $\Phi = 0.85$ showed that the monoxide SnO is formed rather than the dioxide SnO$_2$. As is the case with many organometallic precursors, the Sn-precursor tetramethyltin (TMT) decomposes and forms Sn atoms within a few hundred microseconds by thermal decomposition. Thereafter, tin oxide is produced by a subsequent oxidation of the tin atoms. As studied for many metals, atomic as well as molecular oxygen plays an important role for the oxidation of metal atoms and suboxides, respectively. Therefore, the oxidation process leading to tin dioxide is expected to be incomplete because of the lack of oxidizing species. Hence, it is understandable that almost no tin oxide was found at $\Phi > 0.85$ due to the fact that mainly tin monoxide is formed. Its high vapor pressure prevents the material from condensation within the reaction chamber.

Zinc oxide is an amphoteric material that forms zinc hydroxides during particle growth within the flame and which also has a high vapor pressure like tin monoxide. As a result of the high vapor pressure of tin monoxide and zinc hydroxide at elevated temperatures, it was found that noticeable amounts of tin oxide and zinc oxide nanoparticles could only be produced when $\Phi > 0.7$.

The results of these investigations show that stoichiometric $\text{H}_2/\text{O}_2$ ratios may be inadequate for a sufficient oxidation of precursor material and the formation of the respective oxides is significantly dependent on the oxidation potential of the flame. A 1-dimension simulation of the gas-phase species that appear in hydrogen/oxygen flames typically used for nanoparticle synthesis was performed with CHEMKIN’s PREMIX™. The underlying gas-phase temperature used for the calculation was measured with laser-induced fluorescence of traces of NO added to the burning gas. Despite the concentration of the educt gases hydrogen and oxygen, the diluent argon, and the product water, it was only hydrogen, oxygen, and OH radicals that showed a noteworthy concentration along the reaction path while the concentration of HO$_2$ as well as H$_2$O$_2$ is negligible. Except for molecular oxygen, species with oxidizing and reducing properties for $\Phi = 0.5$ and 0.85 are shown in Figure 3. The simulation shown in Figure 3 indicates that in the case of $\Phi = 0.5$, the concentration of the oxidizing radicals O$^-$ and OH$^-$ (filled symbols) reaches the maximum shortly after entering the reaction zone at about 20 mm height above the burner head, and the portion of the reducing gases H and H$_2$ vanishes very fast. In the case of $\Phi = 0.85$, the concentration of O/H as well as H$_2$/H$_2$O is clearly lower. A high concentration of oxidizing species is vital for the oxidation process and their concentration with respect to the height above burner is indicative of the degree of oxidation at that position. Hence, the oxidation of...
the metal is assumed to be highly sensitive to the oxidation potential at the early particle formation due to no further increase of the concentration. As for low initial concentrations of oxygen, this concentration and the residence time within the reactor isn’t sufficient for the formation of some materials with the highest possible oxidation state, and an oxygen-concentration-dependent stoichiometry is synthesized. As a result, the electrical as well as optical properties change due to the conditions during particle growth. Table 1 summarizes some of the results obtained from electrical measurements on tin oxide powders.

To measure the resistivity, a few mg of the as-prepared powders were pressed into thin pellets with a diameter of 5 mm. The thickness was measured and the AC impedance of the pellets was investigated by putting them between 2 plane platinum electrodes\(^1\). The DC conductivity was extrapolated from the low-frequency data measured, and the resistivity was calculated by means of the geometrical data.

As a second example, the insufficient oxidation during the formation of TiO\(_2\) nanoparticles via synthesis in the premixed flame reactor will be discussed. The synthesis of titania with a decreasing amount of oxygen results in colored material and the color can be tuned from white to pale yellow to pale blue. As previously reported, this change in color is strongly dependent on defects originating from oxygen vacancies that give rise to color centers\(^20, 21, 22\). It is assumed that these defects are responsible for an increased catalytic activity of those materials\(^20\).

Additionally, a high oxygen flow rate tends to favor the formation of the anatase phase, while a low oxygen flow rate promotes the formation of the rutile phase\(^23, 24\). Oxygen vacancies also play an important role in the defect luminescence of zinc oxide at around 2.25 eV, while simultaneously quenching the band gap and near-band gap luminescence in the blue around 3.25 eV. These oxygen defects can be prevented via a wet chemical route with subsequent surface termination\(^25, 26\). An extreme excess of oxygen during the formation of zinc oxide from the gas phase can nevertheless also be successfully used to produce ZnO nanoparticles that show almost no defect luminescence, as can be seen in Fig. 4.

![Fig. 3 Mole fraction of O\(^{\bullet}\), OH\(^{\bullet}\), H\(^{\bullet}\) and H\(_2\) in a H\(_2\)/O\(_2\)/Ar premixed flame calculated with PREMIX\(^{\text{TM}}\) for \(\Phi = 0.5\) (left) and 0.85 (right).](image1)

![Fig. 4 PL spectra of ZnO from the gas phase synthesized at \(\Phi = 0.5\) (red graph) and with extreme excess of oxygen.](image2)

| Stoichiometry | \(\Phi\) | Crystal structure        | Color   | Resistivity / \(\Omega\)m |
|--------------|--------|-------------------------|---------|--------------------------|
| SnO\(_2\)    | 0.25   | tetragonal (Rutile)     | white   | \(14.5 \times 10^3\)    |
| SnO\(_{1.74}\)| 0.63   | tetragonal (Rutile)     | light yellow | \(7.4 \times 10^3\)    |
| SnO\(_{1.4}\) | 0.85   | tetragonal (PbO)        | light gray | \(4.8 \times 10^3\)    |
| SnO          | 0.98   | tetragonal (PbO)        | gray    | \(0.98 \times 10^4\)    |
3. Nanocomposites

The kinetic and/or temperature control of particle formation in flame reactors not only opens a broad range with respect to their oxygen content, but also enables the formation of specific nanocomposites. Several authors have shown that, depending on flame conditions and temperature, different types of composites are accessible.\(^{27, 28}\) The authors have shown that both particles of one kind homogeneously embedded in a second material as well as homogeneous mixtures of two kinds of particles are accessible. They also found that the existence of a second phase may influence size, morphology, and crystallinity.

This paper will focus on the physicochemical properties of the composites. The TiO\(_2\)/SnO\(_2\) and SiO\(_2\)/Fe\(_2\)O\(_3\) systems will therefore be discussed in detail. The phase diagram of SnO\(_2\)/TiO\(_2\) shows a big miscibility gap (see Fig. 5) and in thermodynamic equilibrium, a segregation of SnO\(_2\) and TiO\(_2\) is observed. A couple of scientists have investigated the properties and main differences of SnO\(_2\)/TiO\(_2\) composite particles and films compared to mixed oxides (i.e. substitution of Ti with Sn in the TiO\(_2\) lattice and vice versa) with respect to their photocatalytic potential.\(^{29, 30, 31}\)

The photocatalytic activity of titania mainly depends on the formation of electron-hole pairs (excitons) and the strong oxidation potential of the hole. In TiO\(_2\), excitons can be created under UV irradiation, and the composite material TiO\(_2\)/SnO\(_2\) is expected to improve the transport of electrons from titania to tin oxide. The spatial separation of electrons and holes prevents the excitons from recombination and increases the photoactivity. In comparison to the composite material, the increase in photoactivity of the mixed oxides is expected to originate from an increase in band gap energy of the solid solution Ti\(_{1-x}\)Sn\(_x\)O\(_2\).

Whereas usually, different routes for the formation of composites and mixed oxides are required, flame synthesis has the ability to produce both composites as well as mixed oxides. Due to the high temperature gradient in flame reactors in the order of 10\(^4\) – 10\(^6\) K/s,\(^{33}\) phase compositions can be quenched without segregation “freezing” the actual mixture. As has been shown by Akurati et al.\(^{34}\), the flame synthesis of segregated TiO\(_2\)/SnO\(_2\) nanocomposites is accessible probably due to a relatively low temperature within the particle formation zone, while experiments in our lab for TiO\(_2\)/SnO\(_2\) mixtures ranging from 10/1 to 1/1 always showed complete solid solutions of both oxides.\(^{35}\) The XRD investigations approve this finding as the change in lattice constants of the synthesized materials behaves linearly in accordance with Vegard’s law. It was also found that with an increasing amount of tin substituting for titanium, the band gap energy for TiO\(_2\) shifts from 3.2 eV to higher values.

Moreover, the electrical properties of the materials are also in accordance with these findings, resulting in an increasing conductance when changing the composition from almost insulating TiO\(_2\) to more conducting SnO\(_2\) (Fig. 6). The results presented in Fig. 6 were obtained from impedance measurements taken at 200°C under synthetic air. They demonstrate that pure titania as well as the 10/1 mixture with tin oxide show poor conductance and DC behavior (pure electron transport) up to a few hundred hertz (G’ keeps constant with increasing frequency). With an increasing amount of tin oxide, the conductance increases by about 2 orders of magnitude and DC behavior is observed up to a few kHz.

As has been shown, temperature control as well as oxygen control can dramatically influence nanoparticle as well as nanocomposite growth and properties. Furthermore, the kinetics of precursor decomposition and particle formation also plays an important role during the formation of nanocomposites.
The measurement of the magnetic properties of pure and nanosized \(\gamma\)-Fe\(_2\)O\(_3\) (maghemite) exhibits a superparamagnetic behavior. Nevertheless, the decrease in blocking temperature with decreasing particle size wasn’t observed as expected\(^{36}\) and is explained with magnetic interactions between the particles. Thus, a spatial separation is required to obtain size-dependent magnetic properties. Superparamagnetic materials do not retain any magnetization in the absence of an externally applied magnetic field. Due to this property, superparamagnetic nanoparticles are of great interest for applications that require a switchable magnetism, for example biomedical applications such as magnetic resonance imaging, hyperthermia, separation and purification of biomolecules, and drug delivery.

While iron silicates such as the Fe\(_2\)SiO\(_4\) spinel that are found in nature usually have evolved under high pressure and in the presence of further cations such as Mg or Ca, it is not expected that iron silicates are formed by clean flame synthesis. This fact was used to engineer a material consisting of iron oxide homogeneously dispersed in silica. As the kinetic coefficients for the decomposition of the precursor iron pentacarbonyl (Fe(CO)\(_5\)) as well as for the oxidation of iron atoms reveal a very fast kinetic with respect to the formation of iron oxide\(^{37}\), the reaction rate coefficient of the matrix material source precursor must be larger compared to that of iron pentacarboxyl. Thus, the precursors tetramethylsilane (Si(CH\(_3\))\(_4\)) and hexamethyldisiloxane (HMDSO) were chosen as a source precursor for the silica matrix\(^{38,39}\). As the dilution of Fe\(_2\)O\(_3\) in the silica matrix can be adjusted by means of the corresponding amount of precursor gases, their mean spatial arrangement is directly accessible during the synthesis, while the concentration of the precursors is proportional to the particle size. Fig. 8 shows a representative TEM image of an Fe\(_2\)O\(_3\)/SiO\(_2\) composite material illustrating the distribution of iron oxide within the silica matrix. The inset illustrates very clearly that the iron oxide nanoparticles are completely incorporated into the silica matrix. Thus, the chemistry of this “functionalized” silica is no different to that of common fumed silica.

SQUID measurements of the samples affirm that the iron oxide is superparamagnetic and from XRD measurements, a crystalline iron oxide phase is confirmed. From XRD investigations alone, it is not possible to distinguish between maghemite and magnetite (Fe\(_3\)O\(_4\)). However, Mössbauer spectroscopy at \(T = 4.2\) K on pure iron oxide nanoparticles which are prepared under otherwise comparable conditions had revealed a single sextet with homogeneous line broadening, indicating that the particles are single-phase maghemite\(^{36}\). Furthermore, since the composites were synthesized at \(\Phi = 0.4\), the full oxidation of iron resulting in Fe\(_2\)O\(_3\) is expected.

From a couple of experiments with various precursor concentrations, Fe\(_2\)O\(_3\)/SiO\(_2\) nanocomposites with similar loading but different size of Fe\(_2\)O\(_3\) ranging from 9 to 28 nm in diameter were obtained. As expected for these spatially separated \(\gamma\)-Fe\(_2\)O\(_3\) nanocrystals, a strong relation between the iron oxide particle size and magnetization was found, as can be seen from Fig. 9.
Within the size range of iron oxide particles that have been prepared, the bulk value for the saturation magnetization of $\gamma - \text{Fe}_2\text{O}_3$ is not reached. Therefore, if a higher magnetization is required, materials with a higher saturation are needed. Iron oxides make this possible with the aid of nanotechnology. While bulk magnetite $\text{Fe}_3\text{O}_4$ exhibits ferromagnetism at room temperature, nanosized magnetite switches to superparamagnetism with a size-dependent blocking temperature. In comparison to maghemite, the saturation magnetism of magnetite is significantly higher, achieving 92 emu/g. Hence, the tuning of the oxidation properties within a flame reactor and therefore tuning the stoichiometry by adjusting $\Phi$ again becomes important to force the formation of nanosized magnetite. The Evonik Degussa company has used this chance of product design to develop a superparamagnetic iron oxide/silica nanocomposite called MagSilica®. It consists of nanosized magnetite and maghemite with iron oxide nanoparticles sized in the range of 5-40 nm. The saturation magnetization of this material is higher than that of a pure Fe:O$_2$/SiO$_2$ nanocomposite. Due to the fact that flame reactors enable a cost-effective and versatile industrial process with high production rates, first attempts are made to use this material in large-scale applications such as ferrofluids and for the reinforcement of polymers and adhesives. Due to its superparamagnetic properties, the nanocomposite can be processed and manipulated by means of an alternating electromagnetic field, enabling heating and hardening of materials that usually contain fumed silica as additives. Contactless hardening, bonding and heating of materials containing the composite instead of classic fumed silica become apparent.

4. Summary

Flame synthesis is a cheap and established technology for the high-volume production of oxidic materials for bulk goods. It nevertheless still has the potential to produce sophisticated, engineered and tuned materials with specific properties. Together with the possibilities in tuning the oxygen content of flame-made materials and the composition and morphology of composites, a broad parameter range is accessible for a distinct formation of specific materials which meet the requirements of users in catalysis, optics and electronics.

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