Optical Properties of Silica-Copper Oxide Thin Films
Prepared by Spin Coating

Niklas Mårtensson

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Handledare
Per-Olov Käll, Kenneth Järrendahl

Examinator
Kenneth Järrendahl
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Optical properties of copper oxide nanoparticles in a silica matrix thin film have been investigated. Films were prepared on Si substrates from a sol-gel by spin coating. Four samples with different thicknesses, from 14.5-109 nm, were fabricated. Optical properties were measured with Variable Angle Spectroscopic Ellipsometry. The aim of the project was to further understanding of these films that are interesting in applications for solar absorbers as solar selective coatings. Ellipsometric angles \( \Psi \) and \( \Delta \) were measured in the wavelength range from 250-1700 nm. A dispersion model was developed and fitted to experimental data with acceptable results.

ellipsometry, solar selective absorber, spin coating, thin films, copper oxide nanoparticles, optical modeling, sol-gel
Abstract
Optical properties of copper oxide nanoparticles in a silica matrix thin film have been investigated. Films were prepared on Si substrates from a sol-gel by spin coating. Four samples with different thicknesses, from 14,5-109 nm, were fabricated. Optical properties were measured with Variable Angle Spectroscopic Ellipsometry. The aim of the project was gain to further understanding of these films that are interesting in applications for solar absorbers as solar selective coatings. Ellipsometric angles \( \psi \) and \( \Delta \) were measured in the wavelength range from 250-1700 nm. A dispersion model was developed and fitted to experimental data with acceptable results.
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1. Introduction

Over the last few years, different metal oxides have become interesting for solar absorption applications due to their suitable optical properties [1]. One promising material is copper oxide, CuO [2]. However, a good solar selective absorber has to be able to operate at high temperatures, up to at least 400 °C [2], and studies have shown that CuO films are not stable in air or vacuum at these high operational temperatures [3]. A method proposed by Barrera-Calva et al. is to enclose copper oxide nanoparticles in a silica matrix, thus improving thermal stability [1]. This study investigates the optical properties of this kind of material using spectroscopic ellipsometry. In previous studies the total reflectance properties from the complete solar absorber have been measured. In the present work the modeling gives detailed knowledge of the optical properties of the CuO film, thus making it easier to optimize the solar absorbers.

A fast and inexpensive way to chemically synthesize silica-CuO composite thin films compared to other methods, e.g. high vacuum sputtering techniques, is the bottom-up sol-gel technique. The ultimate goal for mass production purposes is to achieve effective dip coating procedures where the desired components are simply submerged into the liquid solution, thus creating a thin film upon annealing of the material. This study is based on the method described by Barrera-Calva et al. [1]. However, instead of dip coating stainless steel substrates, silicon wafer substrates were spin coated. The main reason to use silicon wafers instead of stainless steel is that the high purity of the silicon wafer crystal ensures accuracy of results and greatly facilitates the optical modeling.
2. Background and Theory

2.1 Sol-gel and spin coating
A sol-gel synthesis usually consists of two main steps. The first step is to create a colloidal solution, with a solid phase dispersed in a continuous liquid phase. The second step is to convert the solution into a gel. This is done by hydrolysis, by adding a gelling agent or by hydrothermal treatment. In this step the sol is chemically transformed into a gel which means the dispersed and continuous phases shifts places. In other words, a gel consists of liquid droplets dispersed in a continuous solid matrix. In spin coating, the more or less gelatinous solution is placed upon the substrate, which then rotates at a given speed and duration, leaving only a thin layer of solution on the substrate. This is a comparably low-cost and easy chemical technique usually done in ambient atmosphere. The procedure can, however, also be done in an artificial (e.g. inert) atmosphere.

2.2 Ellipsometry
The main characterization technique used in this study was Variable Angle Spectroscopic Ellipsometry (VASE). As the name implies, it measures an electromagnetic beam (in the optical wavelength region) reflected upon a sample at different angles of incidence. The quantity measured is the complex reflection ratio \( \rho \), which is the ratio between the complex representations of the polarization states of reflected \( (\chi_r) \) and incoming \( (\chi_i) \) light.

\[
\rho = \frac{\chi_r}{\chi_i} \quad \text{(eq. 1)}
\]

When an electromagnetic wave is reflected upon a surface \( \chi \) can be expressed as

\[
\chi = \frac{E_p}{E_s}, \quad \text{(eq. 2)}
\]

Where \( E \) is the complex representation of the electric field of the electromagnetic wave, and \( s \) and \( p \) are the components of the field perpendicular and parallel to the plane of incidence respectively, commonly known as the s- and p-components of polarization [4]. Combining equations 1 and 2 gives

\[
\rho = \frac{E_{rp} E_{is}}{E_{sp} E_{ip}} \quad \text{(eq. 3)}
\]

When performing a VASE scan the values obtained are the so called ellipsometric angles \( \Psi \) and \( \Delta \), where \( \Psi \) is related to reflected amplitude and \( \Delta \) to the phase shift according to the polar equation,

\[
\rho = \tan(\Psi) e^{i\Delta} \quad \text{(eq. 4)}
\]

From these variables the complex index of refraction

\[
N(\lambda) = n(\lambda) + ik(\lambda) \quad \text{(eq. 5)}
\]
can be calculated, where \( n \) is the refractive index and \( k \) the complex extinction coefficient at a specific wavelength \( \lambda \). From \( N \) it is possible to calculate structural parameters as film thickness and porosity, as well as optical response functions such as absorbance and reflectance.

To go from \( \rho \) to \( N \) the Fresnel equations for reflection needs to be utilized [5]. The Fresnel reflection coefficients for the s- and p components, \( r_s \) and \( r_p \), in a two-phase model, that is, in an interface between two different media are

\[
\begin{align*}
    r_s &= \frac{E_{rs}}{E_{ls}} = \frac{N_0 \cos \theta_0 - N_1 \cos \theta_1}{N_0 \cos \theta_0 + N_1 \cos \theta_1} \\
    r_p &= \frac{E_{rp}}{E_{lp}} = \frac{N_1 \cos \theta_0 - N_0 \cos \theta_1}{N_1 \cos \theta_0 + N_0 \cos \theta_1}
\end{align*}
\]  

(eq. 6)

(eq. 7)

where the electromagnetic wave travels from a media 0 to a media 1 with complex refractive indices \( N_0 \) and \( N_1 \), respectively. \( \theta_0 \) is the angle of incidence, and \( \theta_1 \) the angle of refraction. However, in a general \( n \)-phase model there are multiple interfaces present. Thus, consideration has to be taken to internal reflections and phase shifts. The final reflection equations become much more complicated and the expressions for \( r_s \) and \( r_p \) in an interface between layers change, as effects from reflection in other interfaces must be accounted for, but the principle is still the same.

Instead of expressing material properties through \( n \) and \( k \), the dielectric constant can be used

\[
\varepsilon = \varepsilon_1 + i\varepsilon_2
\]  

(eq. 8)

where the constant \( \varepsilon \) is related to \( n \) and \( k \) through

\[
\begin{align*}
    \varepsilon_1 &= \eta^2 - k^2 \\
    \varepsilon_2 &= 2nk
\end{align*}
\]  

(eq. 9a)

(eq. 9b)

2.3 Optical Modeling

For this project, three different types of models were studied. The first model, Cauchy dispersion with an Urbach absorption tail, is a combination of two empirical (mathematical) models where \( n \) and \( k \) are dealt with separately. This means that they do not necessarily fulfill the so called Kramers-Kronig condition that relates the real (\( \varepsilon_1 \)) and imaginary (\( \varepsilon_2 \)) parts of the dielectric function [4]. The other two approaches, the Lorentz and Gaussian models, are more based on physical assumptions. In these models one or several oscillators are assumed at certain resonance frequencies in the electromagnetic spectra where absorption occurs and thus are related to energy absorption bands in the material. The Lorentzian model accounts for the case where we have an electron oscillating in a harmonic electromagnetic field. The Gaussian model considers a slight variance in atomic and molecular bonding energies caused by local irregularities in the sample, and assumes a Gaussian distribution of oscillators. Both these models are Kramers-Kronig consistent and are described by an amplitude (\( A \)), a peak broadening (\( B \)) and a center of energy (\( E \)) as fitting parameters. It is also not unlikely that there are resonances in frequencies far outside the measured spectrum, and these can be modeled by a constant \( \varepsilon_{\text{inf}} \) [4]. One of the main differences between the two models is that \( \varepsilon_2 \) in the Gauss model has a much narrower peak compared to the Lorentz model [4] which is shown in Fig. 1.
A method to measure how well a model fits to experimental data is by calculating the mean square error (MSE). When doing this it is important to ensure that the fit is also physically reasonable, mathematical solutions with for instance unreasonably high amplitude or with negative film thickness can be found to have the lowest MSE but must be discarded.

2.4 Solar selective coatings
The long-term goal in this project is to achieve a material which absorbs all light in the solar spectral region and still has low emittance in the thermal infrared region. Emittance $\varepsilon$ is related to reflectance via the following equation,

$$A(\lambda) = \varepsilon(\lambda) = 1 - R(\lambda)$$  \hspace{1cm} (eq. 10)

Where $A$ is the absorbance, $\varepsilon$ is the emittance and $R$ the reflectance. Note that this equation is valid only for an opaque case (where there is no transmission of light). The equation tells us that reflectance ideally should be 0 in the solar spectral region and 1 at higher wavelengths.

As seen in Fig. 2, this change in reflectance should occur somewhere around 2.0-2.3 µm. This is a property that the solar selective absorber should inhibit in its whole, not the film itself. In real life energy applications it is likely that the final product consists of a stainless steel substrate (typically a pipe) coated with the absorbing solar selective layer, and on top, an antireflective layer [8, 9].

Figure 1. Comparison between Lorentz and Gaussian model. T. Tiwald [6].

Figure 2. The spectrum of the sunlight reaching the earth’s surface. [7]
3. Experimental

3.1 Synthesis
0.0423 g of CuCl₂·H₂O was dissolved in 2.3 ml EtOH. To this solution 0.0375 ml of propionic acid was added, creating a copper propionate solution with EtOH as solvent. A separate solution consisting of 0.0558 ml TEOS, tetraethyl orthosilicate, Si(OEt)₄, dissolved in 2.3 ml EtOH was prepared. The sol was then created by adding the Cu-propionate solution to the TEOS solution drop wise while stirring. 3.5 µl HCl (12 M) was added as acid catalyst. The molar ratios of the sol were 10:10:1 with respect to TEOS:Cu-propionate:HCl. This gave a sol with Cu-propionate concentration of 0.05 M in EtOH. The sol was then stirred at ambient temperature (≈20 °C) for 24 hours.

A 1.0 mm thick silicon crystal wafer cut into 2.0 x 2.0 cm plates was used as substrate. The substrate was cleaned in room temperature for 15 minutes in 2-propanol in an ultrasonication bath. The 2-propanol was then rinsed away with acetone. Spin coating was used as the deposition technique. The sol was dropped on the substrate until the surface was completely covered (approximately 0.1 ml solution per plate). The substrate was then spun at 3000 rpm for 10 seconds. Four different samples were fabricated, with 1, 2, 4 and 8 spinning cycles respectively and will be referred to as 1L, 2L, 4L and 8L from here on. Between each spinning cycle the sample was placed in an oven at 450 °C for 30 minutes, and after a final spinning cycle it was heat treated at 450 °C for 4 hours. For reference, separate 1 and 2 cycle samples (not to be confused with 1L and 2L) were also prepared as described above, but without adding hydrolyzed copper salt (CuCl₂·H₂O).

3.2 Structural Characterization
For structural characterization of the sample four different types of analysis techniques were performed: Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), Transmission Electron Microscopy (TEM) and Energy-Dispersive X-ray spectroscopy (EDX). SEM images were taken on a Gemini Leo 1550 SEM at 5 kV for sample surface overviews. TEM images were taken on a FEI Tecnai G2 at 200 kV to examine if crystalline nanoparticles could be observed. For the TEM imaging a powder was produced from the original sol by drying it out and baking it under the same conditions as the thin film samples (450 °C, 4h). Since the powder was placed on a copper grid and this gives a response indicating copper presence, the EDX was performed on a small area far from the grid lines, thus minimizing response from the grid. EDX was performed in connection with the TEM studies to obtain information about elements present. XRD measurements with a Cu-Kα source were carried out on both sample 8L and powder to determine the presence of crystalline CuO.

3.3 Optical Characterization
The optical properties of the film were measured with a J. A. Woollam VASE system in the wavelength region 250-1700 nm at three different incident angles, 60°, 65° and 70°. Measurements were also performed in the same wavelength region and with the same incident angles on a substrate with no film, i.e. with only native oxide present. This was to obtain the thickness of the native oxide layer which was then considered a fixed parameter during modeling. Once the ellipsometric angles have been measured, it is necessary to create an optical model in order to obtain valuable structural and optical information about a sample. In the present case, the basic modeling begins with building a 4-phase structure with a thick crystal silicon layer in the bottom...
followed by a thin native oxide layer (SiO$_2$) and on top of that adding a theoretical dispersion model for the properties of the film, as seen in Fig. 3.

Figure 3. A schematic view of the structural model.

For the silicon substrate and the native oxide layer existing reference material was used [10]. The top layer is described by the theoretical dispersion model, whose parameters are adjusted for best fit of the whole multi layer model to experimental data. Depending on the particular case, a more or less complex dispersion model is needed. When the modeling is done, a simulation can be made of the reflectance of a stack with the film on top of a stainless steel substrate.
4. Results

4.1 Structural properties

In Fig. 4 a SEM image of sample 1L is shown. The image shows an even particle distribution on a smooth surface. A similar distribution was seen for the 4L and 8L samples (See appendix A). In comparison, the particles are smallest for 1L and have a sharp flaky appearance. For 4L and 8L the particles are bigger and more rounded.

![SEM image of sample 1L](image)

Figure 4. SEM image from sample 1L showing particles evenly distributed across the surface.

The EDX results from the powder sample are presented in Fig. 5. This confirms the presence of copper, silicon and oxygen, as well as small traces of cobalt and nickel. Figure 6 shows the corresponding area where EDX was performed photographed in a TEM. The image shows a material with dark particles within. The particles are crystalline, which can be seen from the aligned atomic planes in the dark areas. The particles vary in size, from about 5 to 30 nm, as seen in Fig. 7.
Figure 5. EDX results from powder sample.

Figure 6. TEM image showing crystalline nanoparticles in an amorphous material.
Figure 7. TEM image showing nanoparticles 5-30 nm in size unevenly distributed in the material.

The results from the XRD measurements on sample 8L is presented in Fig. 8. The diffractogram shows a peak from the silicon substrate only and no presence of crystalline copper oxide is detected. By comparison the XRD from the dried-out powder show very clear results of CuO (Fig. 9).

Figure 8. An X-ray diffractogram from sample 8L.
4.2 Optical properties

From the measurement on the substrate with no film and fitting to reference data for Si and SiO$_2$, the native oxide layer was found to be 13.5 Å thick. All subsequent modeling of the films used this result. Fitting with Cauchy dispersion with an Urbach absorption tail was attempted on reference silica films with good results (Fig. 10) and with somewhat satisfying results for samples 1L and 2L. The noise in $\Delta$ in Fig. 10 from 1050 and above occurs when $\Delta \rightarrow 180^\circ$. However, this model could not be made to conform with results from 4L and 8L. Dispersion models with Gaussian and Lorentzian oscillators were compared and gave results closer to experimental data.

The fitting results with Gaussian (sample 1L, 2L and 8L) and Lorentzian (sample 4L) oscillators are shown in Fig. 11a-d.

Figure 9. An X-ray diffractogram from the powder. The CuO corresponding peaks are marked.

Figure 10. Experimental $\Psi,\Delta$-data from the ellipsometry measurements (red and green lines) and calculated data from the optical model (dotted line) for silica film without nanoparticles.
Figure 11. Experimental $\Psi, \Delta$-data from the ellipsometry measurements (red and green lines) and calculated data from the optical model (dotted line). a) Sample 1L, b) Sample 2L, c) Sample 4L and d) Sample 8L.

Figure 11 shows the results from the VASE measurements for the three different angles of incidence 60°, 65° and 70°, with the generated models for each sample. The best fit parameters from models corresponding to Fig. 11 can be seen in Table 1. Gaussian oscillators are utilized for samples 1L, 2L and 8L, while a Lorentzian is used for modeling sample 4L. Note that two oscillators are used for modeling of sample 8L. Important values immediately obtained are the film thickness (d) and resonance energies ($E_n$). According to the MSE results the model of sample 1L show the best fit to experimental data. The MSE values then increases with increasing thickness. Figure 12 shows the thicknesses (d) from table 1 as a function of spinning cycles. Note that a doubling in number of spinning cycles roughly corresponds to a doubling in thickness.

Table 1. Modeling parameters for the investigated samples. Mean square error MSE, amplitude $A$, broadening $B_r$, center of energy $E_m$, constant $\varepsilon_{inf}$, type of model and thickness of film in nanometers.

<table>
<thead>
<tr>
<th>Sample</th>
<th>MSE</th>
<th>$A_1$</th>
<th>$A_2$</th>
<th>$B_{r1}$ (eV)</th>
<th>$B_{r2}$ (eV)</th>
<th>$E_{n1}$ (eV)</th>
<th>$E_{n2}$ (eV)</th>
<th>$\varepsilon_{inf}$</th>
<th>Model</th>
<th>d (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1L</td>
<td>11,098</td>
<td>0,519</td>
<td>-</td>
<td>1,735</td>
<td>-</td>
<td>3,366</td>
<td>-</td>
<td>1,898</td>
<td>Gaussian</td>
<td>14,5</td>
</tr>
<tr>
<td>2L</td>
<td>21,162</td>
<td>0,628</td>
<td>-</td>
<td>1,351</td>
<td>-</td>
<td>3,050</td>
<td>-</td>
<td>2,498</td>
<td>Gaussian</td>
<td>23,94</td>
</tr>
<tr>
<td>4L</td>
<td>22,340</td>
<td>0,410</td>
<td>-</td>
<td>3,066</td>
<td>-</td>
<td>1,460</td>
<td>-</td>
<td>1,410</td>
<td>Lorentzian</td>
<td>44,68</td>
</tr>
<tr>
<td>8L</td>
<td>33,766</td>
<td>0,237</td>
<td>0,367</td>
<td>1,022</td>
<td>5,918</td>
<td>2,800</td>
<td>1,107</td>
<td>1,043</td>
<td>Gaussian</td>
<td>109</td>
</tr>
</tbody>
</table>

Figure 12. Thickness as a function of spinning cycles.
From the fitting results the complex index of refraction $N$ is calculated for the nanoparticle films. Figure 13 and 14 show values for $n$ and $k$, respectively.

**Figure 13. Refractive index $n$ for the nanoparticle films calculated from model data.**

**Figure 14. Extinction coefficient $k$ vs. nm**

As seen in figures 13 and 14, the optical constants $n$ and $k$ have a similar appearance for sample 1L and 2L with similar peak values for $k$ at around 400 nm and same shape of the $n$-curve. The 4L sample has a low $k$-value for low wavelengths but increases with wavelength up to 1000 nm, whereas sample 8L exhibit a $k$-peak at the same wavelength as 1L and 2L, and then another local maximum at 900 nm. For both 4L and 8L $n$ is low. From the complex refractive index $N = n+ik$ simulations of the films with optical properties corresponding to samples 1L, 2L, 4L and 8L were done and the reflectance calculated, shown in Fig. 15.
a) 1L properties

b) 2L properties

c) 4L properties
d) 8L properties

Figure 15. Calculated reflectance of films on stainless steel (SS/x nm) and values of stainless steel only (SS).
Figure 15 a-d displays the simulated reflectance from a stainless steel substrate with a film on top, with same optical properties and similar thickness as 1L, 2L, 4L and 8L respectively. Reflectance is high and decreases with increasing thickness. Figure 15 e) shows a 1µm thick film with the optical properties of sample 8L and here reflectance is low.
5. Discussion

The fitting results for the reference silica films without nanoparticles were very good (MSE=2) with a Cauchy-Urbach approach. However, this model was unable to describe the samples with nanoparticles accurately. This clearly indicates that the nanoparticles add a complexity to the optical response. Since we expect to obtain a material consisting of nanoparticles distributed in an amorphous silica matrix, the Gaussian model could be expected to be the most suitable one [6]. Also, the particles are very different in size, from about 5 to 30 nm, and appear to be unevenly distributed in the silica matrix, which further motivates that the Gaussian oscillator is best suited for modeling and analysis of this type of material [11]. As described in section 2.3, the Gauss and Lorentz oscillators have a similar appearance, but the Gaussian dispersion model was found to have the best fit for all samples except 4L, where the Lorentzian approach gave the better fit. When looking at Fig. 14, 1L and 2L look very similar with absorption peaks around 400 nm. The 4L sample tells a different story showing a broad peak around 1200 nm. The 8L sample seems to be a combination of the two above, with two distinct peaks, one close to the 1L and 2L samples and one close to the 4L sample. This may indicate the presence of two different phases of copper oxide, CuO and Cu$_2$O, where CuO has an absorption peak at 1240 nm and Cu$_2$O at 390 nm [10]. This would mean that samples 1L and 2L contains only the Cu$_2$O phase, 4L the CuO phase and 8L both phases. The XRD of sample 8L gave no indication of Cu-particles. This does not necessarily mean that the film does not contain any crystalline nanoparticles, it can be that the result is not observable due to low particle concentration or high peak broadening due to small particle size. However, the XRD of the powder in Fig. 9 shows a very clear presence of CuO but no presence of Cu$_2$O. There could still be Cu$_2$O in the powder and films, but as the XRD tells us, if that is the case, it cannot be crystalline. The TEM images in combination with EDX analysis shows an amorphous material with crystalline nanoparticles consisting of Si, O and Cu. The response to copper is very strong and it is most likely that the amorphous material is SiO$_2$ and the particles CuO, since crystalline CuO was detected in the XRD measurement. However further investigation using X-ray Photoelectron Spectroscopy (XPS) must be carried out to be able to make a more reliable conclusion about the components in the film.

The existing models for samples 1L-8L display reasonable optical properties and fittings are fairly good. From table 1 and Fig. 11 it can be seen that the obtained layer thickness roughly doubles with doubled number of spinning cycles which is also very reasonable. Figure 13 displays the refractive index $n$ which is low for 4L and 8L, especially in the lower wavelengths. This indicates that samples 2L and 1L are denser than 4L and 8L. The low refractive index for samples 4L and 8L can be due to voids most likely created from the evaporation of solvents during the annealing process. It can also be due to surface roughness, which is supported by the SEM images in Fig. 4 and appendix A, that shows what appears to be large (1-10 µm) “islands” of agglomerated particles on a homogenous surface. To account for this, a model including roughness must be developed. The model should be an effective media approximation model that assumes a mixture of voids and the actual media. A purely speculative theory is that the islands are similar to the presumed silica matrix with copper CuO or Cu$_2$O seen in the TEM images, and the smooth surface contains very little or no particles. The films needs to be analyzed using EDX to gain more knowledge about this.
As described in section 2.3, reflectance should theoretically be equal to zero in the displayed waveband for optimum absorbance. The calculated reflectance curves in Fig. 15 a-d show high reflectances, up to 75 %, and could not be used as solar selective coatings. However, reflectance decreases with increase in film thickness and number of spinning cycles, and if a 1 µm thick film is assumed to have the optical properties of sample 8L reflectance drops drastically to values below 5 % up to a wavelength of 1000 nm and below 15 % for 1000-1700 nm. These values look promising for solar selective absorbers.
6. Conclusions and Future Work

Optical properties of silica-copper oxide composite thin films grown from precursor sol-gel with the spin coating method have been measured and analyzed. A fairly accurate model has been developed, but needs to be improved. In order to improve the optical modeling, the thicknesses of the films can be measured with an independent technique to eliminate it as a fitting parameter in the optical analysis. The improved model should be an effective media approximation model that accounts for voids and with roughness included. Furthermore, the size and shape of the nanoparticles must be taken into consideration. The films display properties that are not suitable for solar selective absorbers, but if film thickness can be increased to the micrometer size while optical properties $n$ and $k$ are the same as for sample 8L, results are of interest and further investigation should be done. More research needs to be done on the synthesis and spin coating process to ensure reproducibility and continuity in the fabrication of films. Also, measurements should be done in the thermal IR waveband from 2 to 35 µm to obtain information about reflectance and, consequently, emittance.
7. Acknowledgements

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References


Appendix A

SEM images of a) 1L, b) 4L and c) 8L.

a)
Appendix B

TEM images of dried out powder made from experimental synthesis.