Thickness and Composition of F-doped SnO$_2$ Thin Films as Determined by Rutherford Backscattering Spectrometry

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Abstract
F-doped SnO$_2$ films were fabricated on silicon wafers by inverted pyrosol technique. The films were characterized by Rutherford backscattering spectrometry (RBS) and scanning electron microscopy (SEM) in order to determine film thickness and composition. RBS technique revealed that O/Sn ratio of the films was around 2.03-2.18.

1. Introduction
Tin oxide (SnO$_2$) is an important oxide used as an efficient dielectric material, catalytic material, sensing material and transparent conducting material. Doped SnO$_2$ films are used as transparent conductors in liquid crystal displays, thin-film solar cells and other optoelectric device applications [1]. Fluorine is a preferred dopant because it can provide high transparency and conductivity [2].

Ion beam analysis (IBA) techniques are the methods of choice to determine the thickness and the composition of thin films at near-surface region, i.e. to depths of ~ 0.5 μm [3]. Among these, Rutherford backscattering spectrometry (RBS) is the most commonly used on account of its several advantages such as a non-destructive, simple and fast method with a quantitative analysis capability. In this work we have investigated the thickness and composition of SnO$_2$ films deposited on silicon substrates.

2. Experimental
F-doped SnO$_2$ films with different F/Sn molar ratio in precursor solution were deposited on (001) silicon wafers by an inverted pyrosol technique [4]. The solution was a mixture of SnCl$_2$.2H$_2$O in a solvent, containing 5% DI water and 95% ethanol with different amount of NH$_4$F (F/Sn = 0.00, 0.06, 0.09, 0.12, 0.75 and 2.5) as a fluorine dopant agent. The substrate temperature was set at 450°C.

The thickness and the composition of the thin SnO$_2$ films were investigated by RBS technique and scanning electron microscopy (SEM). A collimated 2.13 MeV $^4$He$^{2+}$ ion beam with a diameter of 1 mm and with a divergence of less than 0.05°, from 1.7-MV tandem accelerator, was used for the RBS analysis. The backscattered beam was detected at scattering angle of 170° by a standard SSB detector. A schematic representation of RBS measurement system at Chiang Mai University was drawn in Figure 1. The sample holder was electrically isolated from the RBS chamber and acts as a Faraday cup. During measurement the RBS chamber was under a vacuum pressure of 5 x 10$^{-6}$ mbar. The thickness and the composition of SnO$_2$ films were calculated by way of making a comparison between the collected spectra and the simulated spectra from SIMNRA code [5]. Direct result of thickness from the code is normally expressed in the unit of atoms per unit area. The conversion into the unit of length was executed...
by assuming a SnO$_2$ atomic density of $8.3916 \times 10^{22}$ atoms/cm$^3$ (bulk density) [6].

**Fig. 1.** RBS setup at the Chiang Mai Ion Beam Technology Center at the Department of Physics, Chiang Mai University.

### 3. Results and discussion

The RBS spectrum of a F-doped SnO$_2$ is illustrated in Figure 2. From the experimental RBS data, SIMNRA was used to fit the data and calculate the film thickness and composition. The film thickness and stoichiometric with O/Sn ratio of the F-doped SnO$_2$ films are shown in Table 1.

The SEM images of a F-doped SnO$_2$ film are shown in Figure 3 a) and b).

**Fig. 2.** Energy spectrum of 2.13 MeV $^4$He$^{2+}$ ions backscattered from F-doped SnO$_2$ film deposited on silicon with F/Sn molar ratio of the solution = 0.06.

**Fig. 3.** SEM image of a F-doped SnO$_2$ film with F/Sn molar ratio of the solution = 0.06 a) cross section b) morphology

<table>
<thead>
<tr>
<th>F-doped SnO$_2$ film with F/Sn molar ratio of the solution</th>
<th>Thickness by RBS (nm)</th>
<th>O/Sn ratio</th>
<th>Thickness by SEM (nm) ± 20 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>762</td>
<td>2.17</td>
<td>750</td>
</tr>
<tr>
<td>0.06</td>
<td>405</td>
<td>2.16</td>
<td>340</td>
</tr>
<tr>
<td>0.09</td>
<td>274</td>
<td>2.16</td>
<td>220</td>
</tr>
<tr>
<td>0.12</td>
<td>536</td>
<td>2.18</td>
<td>480</td>
</tr>
<tr>
<td>0.75</td>
<td>351</td>
<td>2.06</td>
<td>325</td>
</tr>
<tr>
<td>2.50</td>
<td>387</td>
<td>2.03</td>
<td>300</td>
</tr>
</tbody>
</table>
As seen in Table 1, film thicknesses obtained from RBS spectra are slightly higher than those by SEM. To verify the data, both by RBS and SEM, a standard sample is necessary. However, a good thing of RBS is that it is a non-destructive method, sample preparation for thickness determination like SEM is not required. In SEM, EDS is a typical tool for composition analysis. However, oxygen data from EDS may not be accurate due to its low atomic number. On the other hand, RBS is not limited by this problem. The O/Sn ratio of the F-doped SnO$_2$ films can be obtained by RBS. Normally for SnO$_2$, O/Sn = 2. However, for the F-doped SnO$_2$ films on Si wafer, O/Sn ratio was between 2.03-2.18, the higher F/Sn molar ratio in the solution, the lower ratio of O/Sn. For O/Sn > 2, it may attribute to the native SiO$_2$ layer underneath the F-doped SnO$_2$ film. For fluorine, unfortunately, it has atomic number next to oxygen. Therefore fluorine peak in RBS spectrum was dominated by much stronger intensity of oxygen peak.

4. Conclusion

F-doped SnO$_2$ films on silicon wafers were characterized by Rutherford Backscattering spectrometry (RBS) in order to determine film thickness and composition. The film thickness by RBS was slightly higher than those by SEM. The O/Sn ratios of all films were more than 2 which attribute to oxygen in the underneath native SiO$_2$ layer on silicon.

References
