Formation of ITO Nanowires Using Conventional Magnetron Sputtering

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ITO nanowires could be grown in oxygen-free Ar sputtering gas directly after the formation of ITO films with thicknesses of 10–50 nm using conventional magnetron sputtering. Growth of nanorods occurred at substrate temperatures of about 100 °C and higher, whereas nanowires with lengths of 1–10 μm and diameters of roughly 20–200 nm were formed at about 175 °C and above. The diameter, length and density of the nanowires could be controlled by varying the sputtering time, substrate temperature, and SnO2 content in the ITO sputtering target.

Influence of deposition temperature.— Figure 1 shows the change in the surface morphology with substrate temperature for samples deposited in Ar without O2 addition using a sputtering target with 7.0 wt% SnO2. It can be seen that for temperatures of 100 °C, ITO nanorods (NRs) were produced. On the other hand, for temperatures of 175 °C and above, the growth of NWs became apparent, and their density was much higher than that of the NRs. The temperature of 175 °C is close to the melting point of indium (156.6 °C). In addition, the NWs were composed of trunks and bulb-shaped heads. These results suggest that the NWs were formed by a VLS mechanism involving indium or indium-tin droplets.

Influence of SnO2 content in sputtering target.— Figure 3a compares SEM images of NWs grown using sputtering targets with SnO2 contents of 5–30 wt%. The substrate temperature was 300 °C, and the deposition time was 600 s. Figures 3b–3e show the diameter, length and density of NWs as a function of the SnO2 content in the sputtering target. The NW dimensions were determined using the built-in FE-SEM measurement function. Each data point in Fig. 3 is the average of 10–15 nanowires in each sample. The density values in Fig. 3e were determined by counting the number of NWs in a 25 μm2 region. As seen in Fig. 3b, the head diameter decreased with increasing SnO2 content, and a similar dependence was found for the trunk diameter, as shown in Fig. 3c. The results shown in Figs. 3b–3d indicate that NWs could be grown using an ITO target with a restricted SnO2 content range of about 5–30 wt%. As shown in Fig. 3e, the NW density increased with SnO2 content of up to about 30 wt%. The In, Sn and O content at different points in the NWs was analyzed using energy-dispersive X-ray spectroscopy in the FE-SEM system, and the composition of the head and trunk sections was found to be almost the same.

The sputtering power and gas pressures were also investigated as factors influencing the formation of NRs and NWs. The NRs or NWs were grown by sputtering with the wide ranges of 50 W–600 W power and 0.07 Pa – 4.0 Pa Ar pressure applied in this work. The length of

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Figure 1. Effect of substrate temperature on the growth of ITO NRs and NWs. The deposition time was 600 s.

Figure 2. Influence of deposition time and the presence of O₂ in the Ar sputtering gas on NR or NW growth. The substrate temperature was 300 °C.

Figure 3. Effect of SnO₂ content in ITO sputtering target. (a) SEM images showing change in surface morphology. (b)-(d) effect on ITO NW dimensions. (e) Effect on ITO NW density. The sputtering conditions for deposition using a 100 wt% SnO₂ target were 300 °C, 150 W (RF), 0.666 Pa and 5000 s.
NWs reached to about 10 μm by RF sputtering-processing (300° C, 150 W, 0.666 Pa, 14400 s deposition time, ITO target with 12.0 wt% SnO2).

**X-ray diffraction analysis of ITO and NWs.**— The crystal structure and lattice spacing for the ITO NWs were analyzed using X-ray diffraction (XRD; SmartLab, Rigaku). Figure 4a shows an XRD pattern and an SEM image for a sample deposited at 300° C using a sputtering target with 12 wt% SnO2. It can be seen that the intensity of the (400) peak exceeds that of the (222) peak, which indicates that the crystal grains in the sample are oriented in the (400) direction. This was confirmed by comparison with the (400)/(222) intensity ratio for a standard sample of ITO powder (JCPDS file No. 01-089-4598).

Figure 4b shows that the diffraction angles for the (222) and (400) peaks decrease slightly with increasing SnO2 content, which indicates an increase in the lattice spacing. As shown in Fig. 4d, the (400)/(222) intensity ratio increases with SnO2 content. This increase is similar to that observed for the NW density in Fig. 3. Therefore, it is considered that NW growth proceeds along the ITO (400) direction, normal to the substrate.

More detailed elemental and chemical analyzes using electron energy loss spectroscopy and X-ray photoelectron spectroscopy are currently in process. In addition, a crystal structure analysis is being carried out using electron beam nano-diffraction. The results of these analyzes will be described in an upcoming publication.

In summary, a technique was developed for the formation of ITO NWs using a conventional magnetron sputtering system. The key factor is to deposit ITO in Ar sputtering gas in the absence of O2.

ITO NRs were grown at substrate temperatures higher than about 100° C by successive sputtering after the formation of an ITO thin layer with a thickness of 10–50 nm during the initial deposition stage. The growth of NWs became conspicuous at a substrate temperature of about 175° C or higher. It was found that the diameter, length and density of the NWs could be controlled by varying the sputtering time, substrate temperature, and SnO2 content in the ITO target. The successful growth of ITO NRs or NWs required a SnO2 content of about 5–30 wt%. The NWs consisted of trunks and ball-shaped heads, and the XRD analysis results suggested that they grew in the (400) direction.

**References**

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