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Temperature-dependent Photoconductance and Optical Properties of In_2O_3 Thin Films Prepared by Autowave Oxidation

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The influences of ultraviolet (UV) irradiation and temperature on the electrical and optical properties in In_2O_3 films obtained by autowave oxidation were measured experimentally. The film resistance changed slightly for temperatures from 300 to 95 K, and more noticeably when the temperature was further decreased, measured in the dark. Under UV irradiation, the resistivity of the films at room temperature decreased sharply by ∼25 % and from 300 to 95 K, and continued to decrease by ∼38 % with a further decreasing temperature. When the UV source was turned off, the resistivity relaxed at a rate of 15 Ω/s for the first 30 seconds and 7 Ω/s for the remaining time. The transmittance decreased by 3.1 % at a wavelength of 6.3 μm after the irradiation ceased. The velocity of the relaxation transmittance was 0.006 %/s. The relaxation of the electrical resistance and transmittance after UV irradiation termination were similar. It was assumed that the dominant mechanism responsible for the change in the conductivity in the indium oxide films during UV irradiation was photoreduction.

Keywords: In_2O_3 thin films, photoconductance, autowave oxidation.
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Introduction

Transparent conducting oxide (TCO) layers are characteristically described as thin films that exhibit high visible wavelength transparency and electrical conductivity, simultaneously [1]. Two

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important widely used TCO materials are In_2O_3, and Sn doped In_2O_3 (commonly referred to as ITO) [2–4]. In_2O_3 thin films have been used in gas sensors, transparent thin-film transistors, flat panel displays, electrochromic devices and solar cells [3, 5–10]. The physical-chemical properties of indium oxide thin films strongly depend on the preparation methods [3, 11–17]. Currently, low temperatures and simple methods are being developed to deposit In_2O_3 films [8, 11, 13, 18, 19]. Among these methods, a process of metal oxide film deposition using the combustion mode has been proposed for temperatures below 200°C [11]. Recently, we investigated In_2O_3 thin films where the indium in the films was produced by autowave oxidation with a low initiation temperature (180°C) [20]. The autowave oxidation nature in the thin films was similar to the self-propagating high-temperature synthesis (SHS) method for powders, that is now widely used for the production of new materials [21]. SHS products are high-quality compounds and are believed to contain fewer impurities than the original mixtures [21]. SHS can be performed in a wide range of materials including fine powders, liquids, gases and thin TCO films [11, 22, 23].

In several papers, ultraviolet (UV) irradiation has been shown to affect the resistance of indium oxide films [24–28]. As a result of UV irradiation, the indium oxide films showed a sharp decrease in resistance. After UV irradiation, the resistance was slowly restored. The quantitative change in the resistance in the In2O3 films irradiated with UV light was strongly dependent on film's structure and morphology [24,26,28]. This effect could be used to improve the sensitivity of gas sensors based on indium oxide thin films [25, 28–30]. The effect of reducing the Casimir force in ITO films under UV has been reported [31]. One particular interest for practical application is to study the effect of temperature and UV irradiation on the electro-optical properties of TCO films [32, 33] obtained using different methods.

In this article, we present the influence of temperature and UV irradiation on the electronic and optical properties of indium oxide films produced by autowave oxidation. The relaxation of the electrical resistance and transmittance in the wavelength range of 5–20 μm after UV irradiation was also studied.

1. Experimental details

Indium oxide films were prepared by the autowave oxidation reaction [20]. The initial In+In_2O_3 films were obtained by thermal evaporation of pure indium (99.999 %) at a pressure of 1.5 Torr. The films were deposited on cover glass, quartz, MgO and KBr substrates at room temperature. Prior to deposition, the substrates were cleaned in an ultrasonic bath with acetone and then methanol. Autowave oxidation reactions were carried out by heating the initial In+In_2O_3 films with speed of > 1 K/s, to a temperature of 200°C at a pressure of 0.5 Torr. After reaching the initiation temperature, $T_0 \approx 180$°C, nucleation of the In_2O_3 phase began, spreading over the surface in a self-sustaining manner [20].

The film thickness was measured with a Hitachi S5500 scanning electron microscope cross-section and was approximately 300 nm. The cross-section was prepared by cleaving sample from the substrate side. A Hg discharge lamp was used as the UV source and a 3.1–5.0 eV bandpass filter was applied to obtain the UV wavelengths from the lamp's emission spectrum. The lamp emission power was about 0.1 W at the film.

The temperature dependence measurements for the resistance of the In_2O_3 samples were performed using a Quantum Design PPMS-9. Changes in the electrical resistance and resistance relaxations in the films during and after UV irradiation were measured using a standard four-probe method. To measure the resistance changes in the films during UV irradiation and cooling

a Linkam THMS600 was used.

A Bruker Vertex 80 V spectrometer was used to determine the transmittance relaxation of the indium oxide films that were deposited on KBr substrates in the wavelength range of 5–20 μm, after UV irradiation. The transmittance was determined by subtracting the spectra recorded before UV irradiation. Measurements were carried out under vacuum at a residual pressure of 3×10^{-4} Torr.

2. Experimental, results and discussion

Fig. 1 shows the dependence of the indium oxide film resistance (R) versus temperature in the range 3–300 K, measured in the dark.

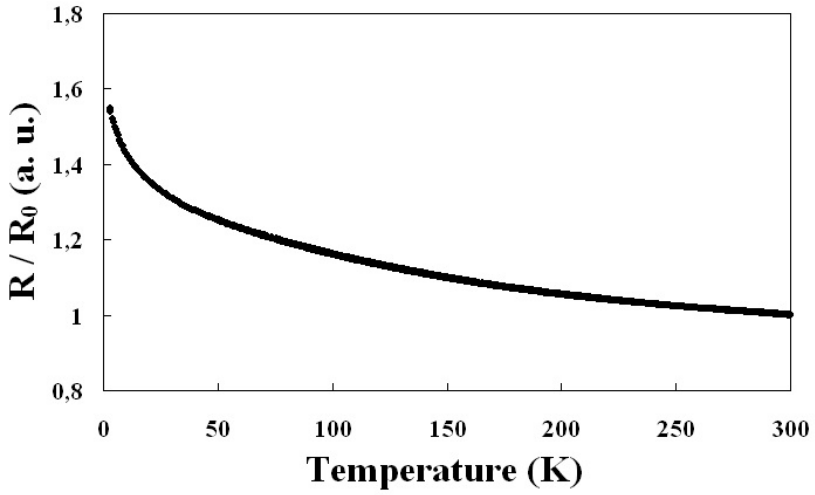

Fig. 1. Variation of normalized electrical resistivity as a function of temperature for an In2O3 film, measured in the dark

The resistance of the films slightly changed for temperatures from 300 to 95 K, with a more noticeable change for further decreasing temperatures. This dependence clearly indicated that the film conductivity was semiconducting. The dependence of the indium oxide film's resistivity on time, with the resistance relaxation, when the UV light was turned on and off is plotted in Fig. 2. The resistivity of the films reduced sharply when the UV light source was turned on. This change in resistance was approximately 25 % at room temperature, presumably because of the generation of free charge carriers and/or an increase the electron mobility, caused by desorption of oxygen ions from the grain boundaries [29]. Similar resistance behavior in In_2O_3 films under UV irradiation was observed in another study [34].

After the UV irradiation was terminated, the resistivity began to relax at a speed of 15 Ω/s for the first 30 seconds and 7 Ω/s for the remaining time. The inset in Fig. 2 shows how the resistivity changed as the temperature was decreased under the UV irradiation conditions. Fig. 1 suggests that the resistivity increased when the temperature was reduced. The resistivity decreased by $\sim$38 % with a decreasing temperature. The most likely explanation for this phenomenon is that the mobility of the charge carriers increased with a decreasing temperature. Several studies have shown that, when the temperature was decreased in the dark, the concentration and mobility of

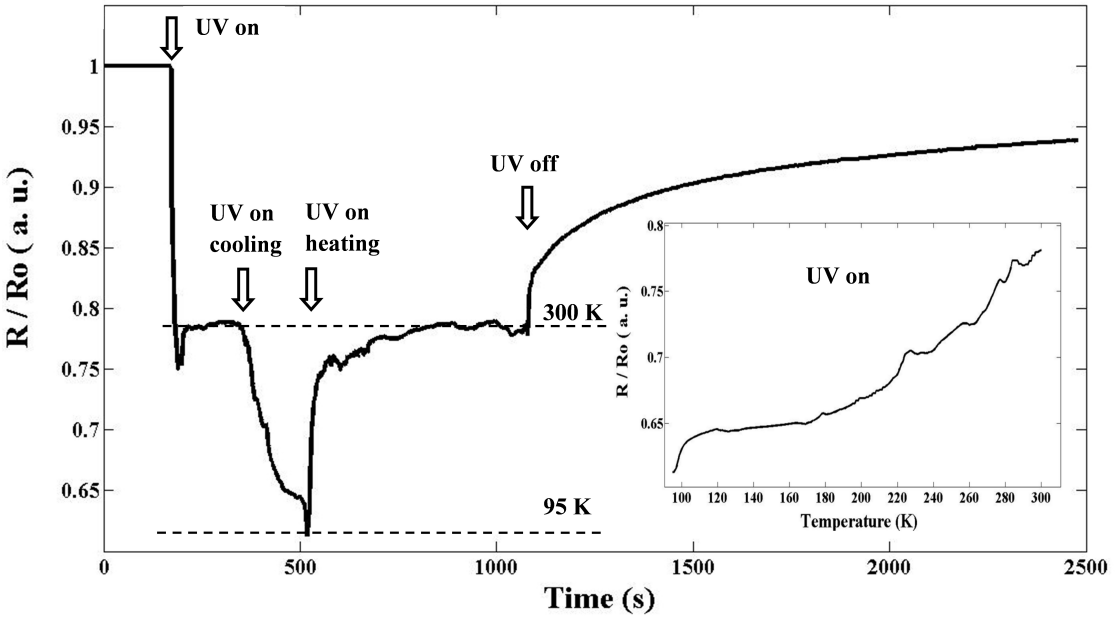

Fig. 2. Variations in the normalized electrical resistivities of an indium oxide thin film as function of time with the UV turned on and off while the sample was cool. The inset shows the normalized resistivity versus temperature under UV irradiation

the charge carrier's in the In_2O_3 films decreased and increased, respectively [35–37].

The temperature dependences of the resistivity in the In_2O_3 samples [35, 36] were in agreement with the measurements taken in the dark. When the temperature was decreased to ∼100 K, the mobility increased. This was most likely caused by a reduction in the electron dispersion of the optical phonons in the In_2O_3 films. This hypothesis is in agreement with recent studies [38]. Upon UV irradiation, there were additional charge carriers. When the temperature was reduced, the mobility of the charge carriers increased. Finally, the resistance decreased dramatically when the films were under UV irradiation and low temperatures. Our suggestion was also confirmed by the fast recovery of the resistance (see Fig. 2) when the temperature of the In_2O_3 samples was quickly increased to room temperature, indicating a non-time delay process.

One method to determine the free charge carrier concentration in semiconducting nanofilms is infrared (IR) spectroscopy [39]. In the framework of the Drude model for free electrons, the equation relating the charge carrier concentration to the plasma absorption frequency ω_p, is given by [40–42]:

$$\omega_p = \sqrt{\frac{Ne^2}{m^*\varepsilon_0}}, \tag{1}$$

where N is the free charge carrier concentration, e is the charge of an electron (1.6×10^{-19} C), m^* is the effective mass of an electron and is equal to 0.36 m_e [43] (m_e is the mass of an electron 9.1×10^{-31} kg) and ε_0 is the permittivity of free space (8.85×10^{-12} F/m). It was assumed that N was $10^{19} cm^{-3}$, corresponding to a resistivity of $\sim 2\times10^{-2}\Omega\cdot cm$ [44]. By substituting these data into equation (1), the plasma frequency for our In_2O_3 samples was $\omega_p \sim 3\times10^{14} s^{-1} (\sim 6.3\ \mu m)$.

We assumed that, when there was generation of free charge carriers by UV irradiation, there

was additional absorption near the plasma frequency. To test these hypotheses, we measured the transmittance of the In_2O_3 films on KBr substrates in the infrared region (Fig. 3), after UV irradiation. The transmittance measurements were carried out after the irradiation had been terminated for 60 seconds. The delay in the measurement was associated with the time taken to achieve operation of an 80 V Bruker Vertex vacuum spectrometer.

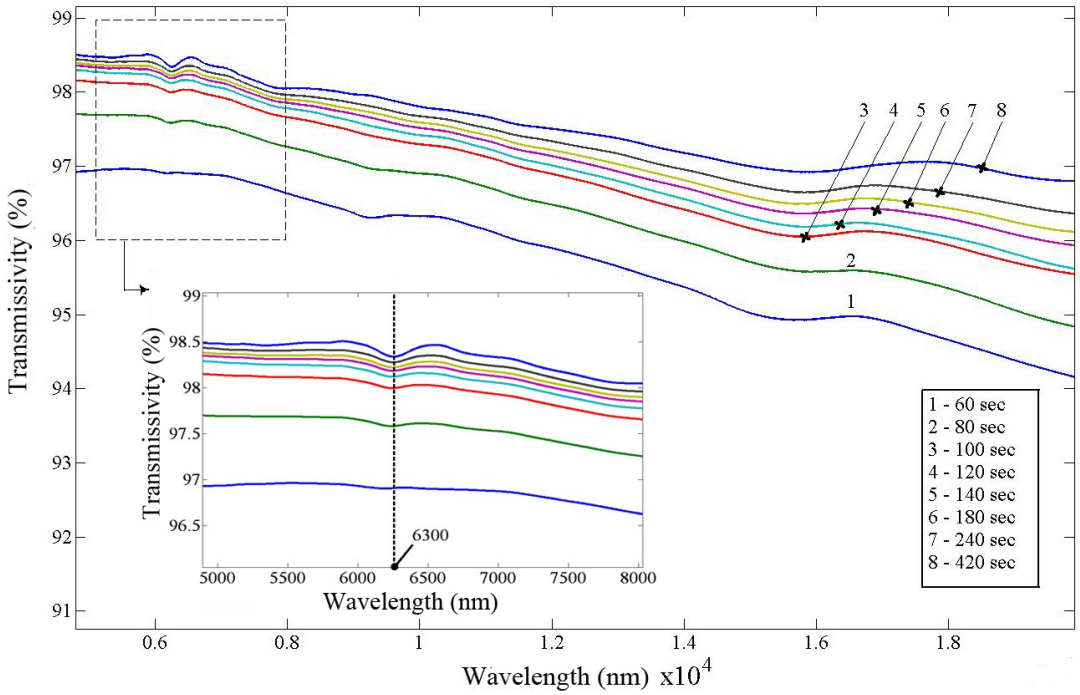

Fig. 3. The transmittance of an In2O3 thin film measured after UV irradiation was terminated, measured at room temperature

Fig. 3 showed that the transmittance was decreased by 3.1 % at a wavelength of 6.3 μm, after irradiation had been terminated. However, there was a relaxation process where there was a gradual increase in the film transparency. From the analysis of the relaxation transmittance at a wavelength of 6.3 μm, the velocity increased the transparency by 0.006 %/s.

For comparison, the relaxation of the electrical resistance and transmittance measurements are presented on the same graph (Fig. 4). These relaxation processes were associated with adsorption of oxygen ions at the film's surface, [30] caused by a residual pressure in the vacuum chamber. The adsorbed oxygen ions acted as trapping centers for the free charge carriers at the film's surface. This led to an increase in the resistivity and the transparency.

Currently, there are a few interpretations on how light effects the electrical properties of indium oxide [29]: the generation of electron/hole pairs, desorption of oxygen adsorbates and photoreduction. The band gap of the indium oxide films, depending on the method of preparation, can vary from 3.5 eV (355 nm) to 4 eV (310 nm) [5], which is consistent with our measurements [45].

For an electron to transit from the valence band to the conduction band, it is necessary to irradiate the indium oxide film with light that has a wavelength $\leqslant$ 355 nm. However, a large

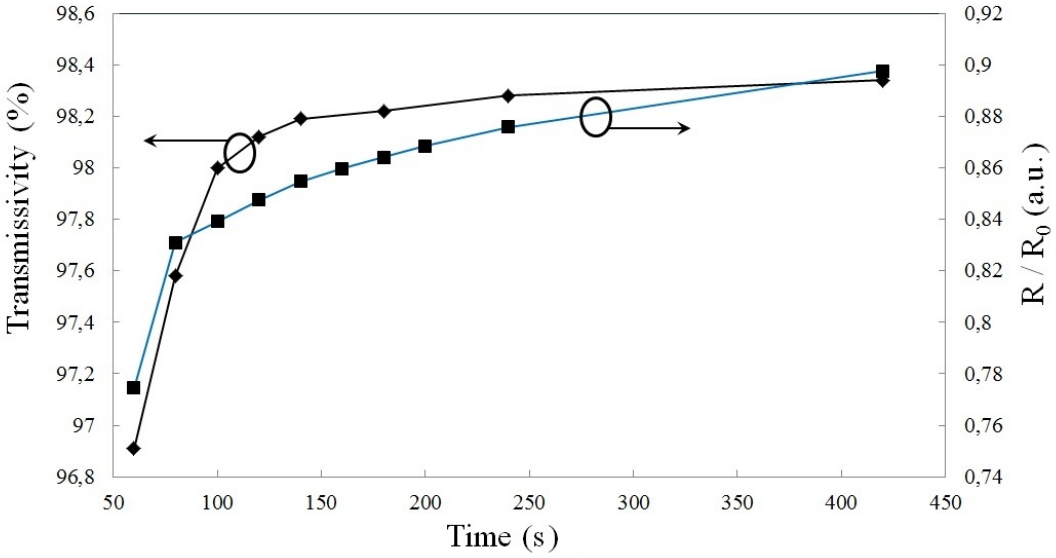

Fig. 4. Relaxation of the normalized resistance and transmittance after the UV irradiation had been terminated, as a function of time

number of surface defects and grain boundaries led to modifications of the indium oxide band structure. Hence, electron transfer is only possible when the energy of the light is smaller than the band gap energy [29, 30]. An electron/hole pair can be formed as a result of this transition.

Oxygen adsorbate desorption from the transparent conductive oxide film surface under UV irradiation can increase the mobility of the charge carriers, and hence increase the mobility of film [46]. In our case study this was possible, but not dominant. The infrared spectroscopy results of the samples (see Fig. 3), and the similarity of the relaxation nature of the electrical resistivity and transparency (see Fig. 4) showed that changes in the electron concentration were responsible for variations in conductivity of the indium oxide films. The IR spectroscopy results were constant for the transparency coefficients in the IR region and/or had a complete mismatch in the resistance relaxation and the transparency coefficient.

The strongest effect of UV illumination on the electronic conductance was caused by an indirect effect: photoreduction. For In_2O_3, this effect was caused by recombination of a hole (generated by optical excitation) with a binding electron from the InвЂ“O bond. As a result, the bonds broke and the oxygen atoms were able to diffuse into the crystal surface, where they were desorbed as O_2 (after recombination with another O atom). This left behind an oxygen vacancy, which is the source of the electrical conductivity in the indium oxide films [4, 47]. Additional oxygen vacancies could appear in the crystal structure. Therefore, we assumed that the dominant mechanism responsible for the changes in the indium oxide film conductivity under UV irradiation was photoreduction.

These results could be used to develop gas sensors based on indium oxide thin films, operating at room temperature. As a result of the additional carriers generation, given their lifetimes, these films may increase the sensitivity of sensors because gases adsorbed on the film surface will capture the generated carriers. The gas could then be identified from changes in the electrical resistance and the resistance relaxation rate [48, 49].

Conclusions

We investigated the effects of UV irradiation and temperature on the electrical and optical properties of In_2O_3 films obtained by autowave oxidation reactions. The temperature dependence of the In_2O_3 resistance in the dark clearly indicated that the film was semiconducting in nature. When the UV light source was turned on, the resistivity of the films was sharply decreased by approximately 25 %, at room temperature. Under UV irradiation, with a decreasing temperature from room temperature to 95 K, the resistivity decreased by ∼38%. Within experimental error, the optical band gap (∼3.5 eV) of the In_2O_3 films remained unchanged after UV irradiation. After the UV irradiation was terminated, the resistivity began to relax at a speed of 15 Ω/s for the first 30 seconds decreasing 7 Ω/s for the remaining time. The transmittance decreased by 3.1 % at a wavelength of 6.3 μm after UV irradiation. There was a relaxation process where there was a gradual increase in the transparency. The velocity of the relaxation transmittance was 0.006 %/s.

The relaxation nature of the electrical resistance and transmittance after UV irradiation were similar. It was assumed that the dominant mechanism responsible for the change in the conductivity of the indium oxide films during UV irradiation was photoreduction.

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Температурная зависимость фотопроводимости и оптические свойства тонких пленок In2O3, полученных методом автоволнового окисления

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Экспериментально исследовано влияние ультрафиолетового (УФ) излучения и температуры на электрические и оптические свойства пленок In_2O_3, полученных методом автоволнового окисления. При измерении в темноте сопротивление пленки менялось незначительно при температурах от 300 до 95 K и более заметно при дальнейшем уменьшении температуры. Под воздействием УФ–облучения удельное сопротивление пленок при комнатной температуре резко снизилось на ∼25 %, от 300 до 95 K, и продолжало снижаться до ∼38 % с дальнейшим понижением температуры. При отключении УФ–источника значение сопротивления релаксировало со скоростью 15 Ом/с в течение первых 30 секунд и 7 Ом/с в течение оставшегося времени. После прекращения облучения коэффициент пропускания снизился на 3,1 % при длине волны 6,3 мкм. Скорость релаксации коэффициента пропускания составила 0,006 %/с. Релаксации электрического сопротивления и коэффициента пропускания после прекращения УФ–облучения были одинаковыми. Предполагается, что доминирующим механизмом, ответственным за изменения проводимости в пленках оксида индия в процессе УФ-облучения, было фотовосстановление.

Ключевые слова: тонкие пленки In_2O_3, фотопроводимость, автоволновое окисление.