Low-Temperature MOCVD Deposition of Crystalline Ga₂O₃ Nanowires using *t*-Bu₃Ga**

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ABSTRACT. Crystalline Ga₂O₃ nanowires were synthesized via a Au-catalyzed as well as a self catalyzed growth by low-temperature MOCVD process using *t*-Bu₃Ga as novel Ga source. Morphology, elemental composition and crystallinity of the resulting nanowires were analyzed by SEM, (HR)TEM, EDX, XRD, and SAED. Photoluminescence spectra of the Ga₂O₃ nanowires show efficient defect-luminescence in the visible and UV range with blue and green emission peaks at 430 nm and 512 nm at room-temperature.

KEYWORDS. Ga₂O₃, MOCVD, Nanowire Growth, Photoluminescence.

1. Introduction

β -Ga₂O₃ is a wide band gap semiconductor material ($E_g = 4.9$ eV),^[1] which has long been known to exhibit both conduction and luminescence properties.^[2,3] It is therefore of widespread interest in technological applications such as gas sensors,^[4] and dielectric layers for GaAs,^[5] GaN,^[6] or SiGe,^[7] as well as in catalysis and photo catalysis.^[8,9] Moreover, β -Ga₂O₃, which can exhibit up to three different emissions, UV, blue and green depending on the dopant and preparation of the sample, and amorphous Ga₂O₃ have received attention as phosphor host materials for thin film electroluminescent (TFEL) devices.^[10]

One-dimensional (1D) structures are of potential interest for future nanodevices and several processes have been developed for the synthesis of Ga₂O₃ nanowires such as arc discharge,^[11] laser ablation,^[12] carbothermal reduction,^[13] thermal oxidation and other methods.^[14,15] In addition, metal-catalyzed *vapor-liquid-solid* (VLS) processes as well as *vapour-solid* (VS) mechanism were successfully used for the growth of Ga₂O₃ nanowires.^[16,17] Unfortunately, all these processes require high process temperatures, typically exceeding 800 °C. In contrast, deposition temperatures as low as 550 °C were achieved using Ga(acac)₃ in a chemical vapor deposition (CVD) processes as well as by use of self-catalyzed VS processes.^[15a,18,19] Moreover, Ga₂O₃ films were deposited at 550 °C in an *atmospheric pressure chemical vapor deposition* process (APCVD) using GaCl₃ and methanol,^[20] by reaction of Ga(NMe₂)₃ with donor-stabilized alcohols,^[21] and by use of (donor-stabilized) gallium alkoxides. However, the latter approach only yielded amorphous, oxygen-deficient Ga₂O₃ films.^[22]

In order to further lower the reaction temperature, the *metal organic chemical vapor deposition* (MOCVD) process was expected as a suitable process due to the generally reduced thermal stability of metal organic precursors. However, to the best of our knowledge, only Kim et al. recently reported on the synthesis of β -Ga₂O₃ nanowires on Au-coated Si(100) substrates using Me₃Ga and molecular oxygen between 600 and 700 °C.^[23] In addition, the synthesis of Ga₂O₃ nanoparticles in a low-pressure flame reactor using a premixed H₂/O₂/Ar flame with dilute concentrations of Me₃Ga was reported.^[24]

Herein, we report on the low-temperature (400–550 °C) synthesis of β -Ga₂O₃ nanowires by using both a self-catalyzed and a Au-catalyzed growth with *t*-Bu₃Ga and molecular oxygen on Si(100) substrates.

2. Results and Discussion

The decomposition temperature of metal alkyl complexes typically decreases with increasing chain length and branching due to a decreasing metal-carbon bond energy.^[25] As a consequence, *t*-Bu₃Ga **1** was expected to have a lower decomposition temperature than Me₃Ga ($T_{\text{onset}} = 222$ °C), Et₃Ga ($T_{\text{onset}} = 212$ °C) or *i*-Pr₃Ga ($T_{\text{onset}} = 184$ °C), respectively. The decomposition pathways of gallium alkyl complexes R₃Ga (R = Me, Et, *i*-Pr, *t*-Bu) have been studied in detail, showing both homolytic and heterolytic bond breakage reactions.^[26] According to a DSC study, the onset temperature of exothermic decomposition of **1** is about 159 °C and the decomposition is finished at almost 230 °C (Fig. 1).

Fig. 1. DSC curve of *t*-Bu₃Ga as-obtained in a sealed Al crucible under argon atmosphere.

This promising thermal behavior prompted our interest toward a controlled, Au-catalyzed growth of Ga₂O₃ nanowires using *t*-Bu₃Ga and O₂ diluted in Ar as carrier gas as Ga and O sources. Detailed investigations on the role of the precursor flow rates on the growth of Ga₂O₃ nanowires showed, that the best results were obtained with small O₂ flow rates (0.3 sccm) but higher *t*-Bu₃Ga flow rates (4 sccm). Moreover, the nanowire growth strongly depends on the substrate temperature, which was varied from 400 to 550 °C (Fig. 2).

Fig. 2. SEM micrographs of Ga₂O₃ nanowires grown by an Au-catalyzed MOCVD process between 450 and 525 °C on Si(100).

Ga₂O₃ nanowire growth was found to start at 450 °C. The density of the nanowires on the substrates steadily increases with increasing substrate temperature as is typical for a kinetically-controlled growth process. However, starting at 500 °C, the nanowires also show an increasing

tendency toward kinking. In contrast, dense arrangements of almost linear Ga_2O_3 nanowires, whose lengths range from 650 - 750 nm, were grown at 475 °C. Fig. 3 shows TEM micrographs of the Ga_2O_3 nanowires. Diffraction patterns and power spectra of HRTEM images can be indexed and simulated using data of $\beta\text{-Ga}_2\text{O}_3$ clearly revealing the crystalline nature of the nanowires as was also confirmed by X-ray diffraction (Fig 4).^[27] The reflections correspond very well with literature values of monoclinic $\beta\text{-Ga}_2\text{O}_3$ (JCPDS 41-1103).

Fig. 3. TEM micrographs of Ga_2O_3 nanowires grown by an Au-catalyzed MOCVD process at 475 °C on Si(100).

Fig. 4. XRD pattern of Ga_2O_3 nanowires grown by a Au-catalyzed MOCVD process at 500 °C on Si(100). Peaks labeled with an asterisk correspond to elemental Si.

The nanowires have small crystals of material appearing with a dark contrast on their tips, which has no sharp or planar interface with the nanowire. The lattice fringes of the tip material can't be assigned to d-values of pure Au and EDS-analysis indicate the presence of gold and gallium. The tip material is covered by a seam of Ga_2O_3 as revealed by EDS that is amorphous or has the same lattice fringes as the nanowire (Figs. 5 and 6).

Fig. 5. TEM micrograph of a Ga_2O_3 nanowire with catalyst at the tip (a); HRTEM image of the tip area of the nanowire (b) and indexed FT of the nanowire close to the tip (c).

Based on the Au-Ga phase diagram we assume that Ga dissolves in the Au catalyst droplets leading to a liquid Au/Ga alloy at 450 °C with at least 20% of Ga.^[28] Ga_2O_3 is formed on the surface of the Au-Ga alloy catalyst droplet and is transported via the surface to feed the growing nanowire. Thus, the nanowires are not grown via the classical VLS-mechanism as known for the Au-catalyzed growth of Si.^[29] However, this specific growth mechanism as well as the low decomposition temperature of *t*-Bu₃Ga might be responsible for the growth of highly crystalline Ga_2O_3 nanowires at low temperatures.

Fig. 6. TEM micrograph of a thin Ga_2O_3 nanowire with catalyst at the tip lying on amorphous carbon (a); indexed FT of the indicated area of the nanowire (b) and Bragg-filtered magnification of the indicated area the distance of fringes of 0.282 nm fits very well with $d_{(002)}$ of $\beta\text{-Ga}_2\text{O}_3$ (c).

The growing direction of the nanowires is not uniform and not parallel to the approximately cubic closed packed oxygen planes in $\beta\text{-Ga}_2\text{O}_3$. The most frequent observed directions are along $\langle \bar{2} 0 1 \rangle$ (Fig. 5) or along $\langle 0 0 2 \rangle$ of $\beta\text{-Ga}_2\text{O}_3$ (Fig. 6). Comparable findings were reported by Yang et al. for Ga_2O_3 nanowires obtained from a Ga-droplet mediated growth process.^[30]

Thereafter we investigated the possibility of a self-catalyzed growth of Ga_2O_3 nanowires. Ga droplets have been shown in the past to initiate nanowire growth of a variety of materials such as Si, Ge,^[31] and GaSb.^[32] Small Ga droplets, which were formed by decomposition of $t\text{-Bu}_3\text{Ga}$ at 500 °C, were found to catalyze the growth of Ga_2O_3 nanowires. Experiments were performed at 500 °C according to the same procedure as applied for the Au-catalyzed growth (flow rates: 0.3 sccm O_2 , 10 sccm Ar).

Fig. 7. SEM micrographs of Ga_2O_3 nanowires grown by a self-catalyzed MOCVD process at 500 °C on $\text{Si}(100)$ with a $t\text{-Bu}_3\text{Ga}$ flow rate of 14 sccm.

Dense films with Ga_2O_3 nanowires up to 3 to 4 μm in length were obtained at 500 °C (Fig. 7). The nanowire density strongly depends on the $t\text{-Bu}_3\text{Ga}$ flow rate as was shown in deposition studies using 14, 10 and 5 sccm $t\text{-Bu}_3\text{Ga}$.

XRD and TEM studies (Fig. 8) revealed the crystalline nature of the Ga_2O_3 nanowires. The crystal quality of the nanowires grown by self-catalyzed MOCVD is worse compared to the Au-catalyzed grown material, also indicating that the specific growth mechanism as observed in the Au-catalyzed nanowire growth might be responsible for the growth of crystalline Ga_2O_3 nanowires. Amorphous regions that disturb the lattice fringes, which are indicated by diffuse scattered electrons in the power spectra (Fig. 9b) and also by the shape and intensity of the Bragg-peaks in XRD, are clearly visible. Indexing of the Fourier transforms succeeds with the parameters for $\beta\text{-Ga}_2\text{O}_3$ and the observed types of growing directions are the same as for the Au-catalyzed nanowires (Fig. 9).

Fig. 8. TEM micrographs of Ga_2O_3 nanowires grown by a self-catalyzed MOCVD process at 475 °C on Si(100).

Fig. 9. HRTEM micrograph of a Ga_2O_3 nanowire grown by self-catalyzed MOCVD (a); FT (b) of the indicated area with indices for the strongest maxima and detail of the nanowire with fringes of 0.29 nm that are in accordance with $d_{(110)}$ of $\beta\text{-Ga}_2\text{O}_3$ (c).

Spectrally and time resolved photoluminescence (PL) spectroscopy was used in order to investigate the optical properties of Au-catalyzed Ga_2O_3 nanowires.

Fig. 10. PL spectra of Au-catalyzed Ga_2O_3 nanowires deposited at 500 °C, obtained at 7 K and 300 K and an excitation density of 170 mW/cm².

Fig. 10 shows time integrated PL-spectra of the nanowire array obtained at 7 K and 300 K using an excitation density of 170 mW/cm². At low temperature, the nanowires exhibit a spectrally wide PL-emission within the whole visible spectral range and extending to the UV-range with two clearly separated peaks in the blue and the green spectral range at around 430 nm and 505 nm, respectively. At room-temperature the UV-emission has disappeared while the overall intensity is reduced by a factor of 2 – 3 due to enhanced thermally activated non-radiative losses

via surface or defect states. Increasing the temperature to 300 K leads to an only slight red-shift of the green emission peak (about 7 nm), while no significant shift of blue emission peak is obtained. Green, blue and UV luminescence was also previously obtained for Ga_2O_3 and is discussed controversially in the literature.^[33]

In general it is attributed to originate from recombination processes via donor and acceptor states located inside the bandgap of Ga_2O_3 . Oxygen vacancies are supposed to form a donor band while gallium vacancies or oxygen-gallium vacancy pairs are supposed to form trapped acceptor states. The blue and UV-emission of Ga_2O_3 is described in an often cited model by L. Binet and D. Gourier.^[33b] Here, an electron in the donor band is captured by a hole on an acceptor via a tunnel transfer. The trapped exciton can subsequently recombine and generates a blue photon. The UV emission in contrary is attributed to stem from the recombination of thermally de-trapped electrons and holes, which can migrate and form a self- trapped exciton. The green emission is mostly assumed to be related to recombination processes via impurities, which form additional donor and/or acceptor states.^[33a] Nevertheless the observed PL-spectrum is to our knowledge the first PL-emission of Ga_2O_3 showing clearly separated blue and green emission peaks. This fact reflects a crystalline structure of high quality and opens the possibility to investigate the recombination dynamics of the blue and green emission separately.

Fig. 11. PL-decay curves of the UV, blue and green emission peaks of Au-catalyzed Ga₂O₃ nanowires detected at 7 K and 300 K, respectively, using an excitation density of 170 mW/cm².

Fig. 11 shows PL- decay curves of the obtained UV, blue and green emission at 7 K and 300 K, respectively. A multi-exponential decay of the PL signal is observed both at room temperature and at low temperature. In general, a fast initial decay (few ps) – which we relate to non-radiative losses via surface or defect states in our nanowires - is followed by decay characteristics with time constants on the order of several hundreds of ps or a few ns. Increasing the temperature from 7 K to 300 K, this fast initial decay becomes more dominant, in agreement with the observed loss in PL intensity. The long decay constant systematically decreases with decreasing wavelength and increasing temperature. At low temperature, we obtain $\tau = 800$ ps, $\tau = 1.4$ ns and $\tau = 2.5$ ns for the UV, the blue and the green emission peak, respectively.

3. Conclusion

Ga₂O₃ nanowires with up to 750 nm in length were deposited on Si(100) substrates by either Au-catalyzed or Ga-catalyzed MOCVD processes at low-temperatures using *t*-Bu₃Ga as novel Ga source and characterized by SEM, XRD and EDX. The Au-catalyzed growth process somehow differs from the classical VLS growth model and the question, whether this specific growth mechanism is responsible for the growth of highly crystalline Ga₂O₃ nanowires, is currently under investigation. In addition, photoluminescence spectra of the Ga₂O₃ nanowires for the first time show efficient defect- luminescence in the visible and UV range with blue and green emission peaks at 430 nm and 512 nm at room-temperature, indicating their highly crystalline nature.

4. Experimental Section

Materials. *t*-Bu₃Ga was prepared according to literature method.^[34] Si(100) substrates were degreased with isopropanol, treated with hydrofluoric acid and heated to 500 °C at 10⁻⁶ mbar for 1 hour in the reactor in order to remove the native silicon dioxide layer.

MOCVD Reactor. MOCVD studies were performed in a cold-wall HV-MOCVD reactor (Fig. 14) equipped with a transparent glass plate which allows an optical control of the deposition process. The substrate heater can be heated up to 600 °C and the working pressure can be varied from atmospheric pressure down to HV-conditions up (10⁻⁶ mbar). The precursor mass flow is controlled by a mass flow controller. *t*-Bu₃Ga (0.1 g) was loaded into a bubbler attached to the MOCVD-reactor under inert conditions (Ar) and the bubbler temperature was set to 0 °C throughout the experiment.

MOCVD Deposition of Ga₂O₃ nanowires

1) Au-catalyzed growth. Small Au droplets of roughly 80-100 nm in diameter were obtained by sputter-deposition of a 2 nm thick Au-layer on Si(100) substrate followed by thermal annealing at 500 °C for 30 minutes. Thereafter, *t*-Bu₃Ga and oxygen, which were separated from each other due to the high reactivity of *t*-Bu₃Ga toward oxygen, were diluted with 10 sccm Ar and simultaneously introduced into the reactor. The mass flow rates (*t*-Bu₃Ga and O₂) were controlled by use of Ar as carrier gas (4 sccm *t*-Bu₃Ga; 0.3 sccm O₂). The working pressure was maintained at 10⁻² mbar during the deposition time (2 minutes) and the cooling time (30 minutes).

2) Self-catalyzed growth. *t*-Bu₃Ga was introduced into the reactor for 3 sec at a working pressure of 10⁻² mbar, yielding small Ga droplets of 60-80 nm in diameter.^[32] Thereafter, Ga₂O₃ nanowires were grown within 2 minutes as described for the Au-catalyzed growth at 500 °C and a working pressure of 10⁻² mbar. The oxygen flow rate was set to 0.3 sccm, while three different *t*-Bu₃Ga flow rates (4, 10, 14 sccm) were investigated.

Nanowire Analysis

SEM Analysis. SEM studies were carried out on an ESEM Quanta 400 FEG or a Jeol JSM 6510 equipped with an energy dispersive X-ray spectroscopy (EDX) device (Bruker Quantax 400).

TEM Analysis. Transmission electron microscopy studies (TEM) were performed on a Philips CM300UT-FEG operated at 300 keV and equipped with a germanium EDS-detector (ThermoScientific NSS) for EDS-analysis. The samples were prepared on perforated carbon foils without further grinding.

XRD Analysis. XRD patterns were obtained using a Bruker D8 Advance powder diffractometer with Cu K_α radiation (λ : 1.5418 Å) and a digitized Philips PW1050 with secondary monochromator with Co K_α radiation (λ : 1.7903 Å), respectively.

Photoluminescence spectroscopy. A frequency tripled Ti:sapphire laser ($\lambda_{\text{Laser}} = 266$ nm) with a repetition rate of 76 MHz and a pulse length of 100 fs was used for excitation. Time integrated PL spectra were obtained by a HORIBA Jobin Yvon monochromator (iHR320) with a focal length of 320 mm and a liquid nitrogen cooled CCD-camera (Symphony CCD-1024x256 open electrode). The time resolved PL was detected by a Hamamatsu streak camera system with an overall temporal resolution of 4 ps.

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Table of Content Entry

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