Single Source Precursor-Based Deposition of Sb_2Te_3 Films by MOCVD-Process**

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The low-temperature MOCVD deposition of Sb_2Te_3 films is achieved by use of the *single source precursor* $(\text{Et}_2\text{Sb})_2\text{Te}$ **1**. The role of the substrate temperature on the deposition rate and the composition of the resulting material film is described. The resulting Sb_2Te_3 films were characterized by XRD, SEM, EDX and Auger spectroscopy. Moreover, the Seebeck coefficient of the Sb_2Te_3 films deposited at 200 °C were determined and values of up to 160 $\mu\text{V/K}$ demonstrate the high quality and low carrier density of the Sb_2Te_3 films.

Keywords: Single-Source-Precursor, Sb_2Te_3 , Thin Film, Seebeck Coefficient, Thermoelectric

1. Introduction

Group V/VI materials of the type V_2VI_3 such as antimony and bismuth telluride (Sb_2Te_3 , Bi_2Te_3) are narrow band-gap layered semiconductors with tetradymite structure, which exhibit low bandgaps (0.1 – 0.3 eV) and superior thermoelectric characteristics near room temperature.^[1,2] In fact, they were considered as the best thermoelectric materials for applications at near room temperature since p-type $\text{Bi}_2\text{Te}_3/\text{Sb}_2\text{Te}_3$ superlattices were found to possess the highest thermoelectric figure of merit (ZT) of about 2.4 at 300 K. Bulk Sb_2Te_3 finds widespread technical applications in minipower-generation systems and micro coolers,

charge coupled device (CCD) technology, and infrared detectors.^[3-6] Since nanostructuring is expected to further enhance the figure of merit^[7] due to reduction of the thermal conductivity,^[8-13] Sb₂Te₃ films and nanostructures with well defined morphologies have been fabricated both via solution phase and gas phase processes such as solvothermal processes,^[14-24] microwave heating,^[25,26] vapor transport growth,^[27] vapor-liquid-solid growth,^[28,29] electrochemical atomic layer epitaxy,^[30-33] electrochemical deposition,^[34-38] DC and RF sputtering,^[39] and molecular beam epitaxy (MBE) processes.^[40] In addition, (metal organic) chemical vapor deposition processes (MOCVD) using trialkylstibanes SbR₃ (R = Me, Et) and dialkyltellanes TeR'₂ (R = Et, *i*-Pr) have been successfully employed.^[41-44]

The electrical and optical properties of nanostructured semiconductors critically depend on the nature and the purity of the precursor. The molecular structure of the metal organic precursor and its specific decomposition mechanism seriously influences the incorporation of impurities such as carbon into the layer and hence the semiconductor properties of the resulting materials. Unfortunately, organometallic precursors typically require high pyrolysis temperatures, hence the MOCVD growth has to be performed at rather high substrate temperatures, which can cause the incorporation of impurities such as carbon due to uncontrolled decomposition of the precursor as well as interlayer diffusion in superlattices such as Sb₂Te₃/Bi₂Te₃ superlattices. As a consequence, alternate processes such as photoinduced dissociation or thermal pre-cracking of the precursors as well as alternate reactor designs have been applied. Venkatasubramanian et al. for instance reported on the growth of Sb₂Te₃ thin films at substrate temperatures as low as 225 °C on GaAs substrates using a specific MOCVD-reactor.^[45] In addition, these thermal problems might be overcome by use of alternate (thermolabile) precursors, which decompose at much lower temperatures compared to conventional metal alkyls. Therefore, there is a strong demand for new, tailor-made metal organic precursors.

Single-source precursors often have numerous advantages compared to simple metal alkyl complexes conventionally used in MOCVD processes. They are less air and moisture sensitive and very often show lower growth temperatures. Moreover, pre-reaction in the gas are suppressed due to the presence of only one precursor molecule and, even more important, the ligand design to some extent allows control over the decomposition mechanism and therefore the level of impurities such as carbon incorporated into the resulting material films. Finally, the alignment of the V/VI molar ratio is not necessary since the single-source precursor already contains the correct elemental composition of the desired material film within the single molecule. Unfortunately, the high molecular weights of these single-source

precursors typically lower their volatility, which is a major drawback in conventional atmospheric pressure CVD processes.^[46] As a consequence, the number of suitable single-source precursors is limited and to the best of our knowledge, the deposition of Sb₂Te₃ films by use of such a *single source precursor* was only once reported.^[47] Sb[(TePi-Pr₂)₂N]₃, a solid precursor that decomposes at 160 °C, was introduced as solution in toluene in an aerosol-assisted (AA)CVD process. Pure Sb₂Te₃ films were deposited in the temperature range from 375–475 °C. The films consist of hexagonal Sb₂Te₃ nanoplates, whose size range from 100 to 200 nm. The 20 nm thick nanoplates are randomly oriented with a preferred orientation along the (003) plane. Four-point probe resistivity measurements gave conductivity values as high as 180 Ω⁻¹ cm⁻¹.

We report herein on the MOCVD deposition of Sb₂Te₃ films at temperatures as low as 200 °C using the *single source precursor* (Et₂Sb)₂Te **1**. **1** is a stable liquid at ambient temperature with a reasonably good volatility. The distinctive role of the substrate temperature on the growth of the antimony telluride films and their chemical composition was investigated in detail. The composition and crystallinity of the resulting films was analyzed by EDX, XRD and their surface morphology was studied by SEM. The Seebeck coefficient of these films was determined by a Seebeck microprobe system.

2. Results and Discussion

(Et₂Sb)₂Te **1** was prepared in almost quantitative yield by reaction of elemental tellurium with tetraethyldistibine Et₄Sb₂.^[49–51] This is a well-known reaction for the synthesis of compounds of the general type (R₂M)₂E (M = Sb, Bi; E = S, Se, Te; R = alkyl, aryl). Compounds of this type typically decompose upon heating with formation of elemental antimony or bismuth chalcogenide M₂E₃ (M = Sb, Bi; E = S, Se, Te) and the corresponding trialkylstibine and – bismuthine R₃E, respectively (Scheme 1). The thermal properties of **1** were recently described by us.^[48] **1** decomposes between 160 – 200 °C with subsequent formation of SbEt₃, which is thermally stable up to 250 °C.

Scheme 1. Synthesis of **1** and its thermal decomposition.

This specific decomposition reaction, which often occurs at low-temperature, can be used for the size- and shape-selective synthesis of nearly monodisperse Sb₂Te₃ nanoplates at 170 °C in a solution-based process as we demonstrated recently.^[48] In contrast, to the best of our knowledge, this decomposition reaction has never successfully applied for the growth of M₂E₃

films by MOCVD process. Only Dickson et al. reported on the use of **1** in a hot wall MOCVD process with H₂ as carrier gas between 400 and 450 °C, but only decomposition of the precursor and formation of elemental Sb was observed.^[52] Keeping the thermal behavior of **1** into mind, these findings are not surprising. We therefore used **1** in orienting studies in a home-made MOCVD reactor at a working pressure of 10⁻¹ mbar. Argon was used as carrier gas and the mass flow was regulated to 15 sccm by use of a mass flow controller.

Sb₂Te₃ films were deposited on Si(100) substrates with a native oxide layer at substrate temperatures of 200, 250 and 300 °C using a home-made MOCVD cold-wall reactor. The growth rate at 200 °C, which was determined to approximately 600 nm/h, was found to increase with increasing substrate temperature as is typical for a kinetically-controlled growth process. Uher et al. recently reported that a key parameter for the growth of high quality Sb₂Te₃ films by MBE technique on (0001) sapphire substrates is the substrate temperature,^[52] which strongly affects the reaction rate and the kinetics of the species, hence influencing the composition and quality of the resulting Sb₂Te₃ film. High-quality films were grown near 330 °C, which was explained by the different sticking coefficient of Sb and Te. The higher sticking coefficient of Te at low substrate temperatures results in the formation of a Sb-deficient film, whereas the sticking coefficient of Te decreases at higher substrate temperatures, hence yielding a stoichiometric Sb₂Te₃ film.^[53] Recently, Periano et al. reported on the growth of stoichiometric Sb₂Te₃ films by MBE process at 250 °C.^[54]

Fig. 1. EDX analysis of a Sb₂Te₃ film deposited at 200 °C; the Si peak results from the substrate.

Table 1. Substrate temperature and chemical composition (%) of the resulting material film according to EDX studies.

	200 °C	250 °C	300 °C

Sb	39.9	43.5	48.4
Te	60.1	56.5	51.6

Sb₂Te₃ films with the expected Sb:Te molar ratio of 40:60 were obtained between 200 °C and 220 °C, an increasing substrate temperature leads to the formation of Sb-rich films. The chemical composition of the material films (Table 1) strongly depends on the substrate temperature as was shown by EDX analysis (Fig. 1). Even though the vapor pressure of Te is higher than that of Sb, hence in general allowing the re-evaporation of Te as was recently demonstrated,^[55] both the low substrate temperatures and the moderate pressure conditions (10⁻¹ mbar) makes this explanation rather improbable. Instead, the increasing Sb concentration most likely points to a partial thermal decomposition of SbEt₃, which is formed as a by-product during decomposition of **1** (scheme 1). Pure Et₃Sb starts to decompose at about 240 °C according to DSC studies,^[48] which agrees very well with our experimental findings according to which the Sb concentration steadily increases with increasing substrate temperature. As a consequence, the substrate temperature should not exceed 220 °C when using **1** as *single source precursor*. EDX studies also revealed the presence of small amounts of carbon (5-6%), most likely due to impurities on the surface due to the transport of the films through the air or due to incompletely decomposed precursor residues.

Fig. 2. SEM picture and SAM/AES studies (mapping of Sb, Te, Si and C) of the Sb_2Te_3 film deposited at 200 °C; high element concentrations indicated by bright color.

The composition of the films was also investigated by scanning Auger electron microscopy studies (SAM/AES), showing high carbon and oxygen concentrations (Fig. 2). However, these contaminations are rather located on the substrate surface as was shown by elemental mapping. C, O as well as Si was mostly present in "holes" of the Sb_2Te_3 films on the Si substrate (Fig. 2d-f), whereas the materials film mainly consists of Sb and Te (Fig. 2b,c).

The powder diffractogram of the film deposited at 200 °C (Fig. 3) clearly proved the formation of pure, crystalline Sb_2Te_3 films. Reflection peaks with a significant intensity in the X-ray diffractogram can be indexed on the basis of the structure of rhombohedral Sb_2Te_3 . In addition, small reflections due to the Si(100) substrate are visible at 33° and 62° (not shown

here), respectively. The most intense peaks in the diffractogram correspond to (00l) planes, pointing to a preferred c-axis oriented growth as was previously reported for MBE^[55-58] and MOCVD^[43-45] grown Sb₂Te₃ films. In contrast, the diffractogram of the film deposited at 300 °C shows additional reflections due to the presence of crystalline elemental antimony (Fig. 4). Quantification of the Sb reflections shows an almost 1:1 ratio of Sb₂Te₃ and Sb as was expected from the EDX results.

Fig. 3. 2-θ XRD pattern of the film deposited at 200 °C; peaks of Sb₂Te₃ in black (JCPDS-ICDD card 71-0393).

Fig. 4. 2- θ XRD pattern of the film deposited at 300 °C; peaks of Sb_2Te_3 in black, those of elemental Sb in red (JCPDS-ICDD card 035-0732).

Fig. 5. SEM photographs of Sb_2Te_3 films deposited on Si(100) substrates at substrate temperatures of 200, 250 and 300 °C.

Fig. 6. SEM photograph of a) an isolated Sb_2Te_3 hexagonal plate and b) a cross-section view of the Sb_2Te_3 film deposited at 200 °C on Si(100) substrates.

The surface morphology of the resulting material films deposited at 200, 250 and 300 °C, respectively, was investigated by SEM (Fig. 5). The film obtained at 200 °C consists of hexagonal Sb_2Te_3 nanoplates with a diameter of more than 2 μm (Fig. 6), which are randomly oriented with respect to the substrate. This is in good agreement with the X-ray data. Their formation can be explained by the low substrate temperature, which is expected to reduce the mobility of the adatoms on the substrate surface during growth, hence leading to island nucleation and coalescence growth mode instead of a step bunching growth process. With increasing temperature, the films become more flat as was expected, but their composition becomes more Sb-rich as shown before.

The spatial resolved Seebeck measurements were performed by a Potential Seebeck Microprobe (PSM).^[59] A heated tip scans the surface of the thin film, while the potential and the temperature difference between the edge of the sample and the tip is measured. The average Seebeck value of $153.35 \pm 6.68 \mu\text{V/K}$, which is higher than that of bulk Sb_2Te_3 crystals, was determined at room temperature (Fig. 7). This value corresponds very well to values recently reported by Yan et al. for Sb_2Te_3 films consisting of much smaller Sb_2Te_3 particles (100 nm diameter),^[27] whereas the authors reported significantly lower Seebeck values of 60–95 $\mu\text{V/K}$ for films containing larger Sb_2Te_3 particles.^[27] A similar Seebeck coefficient (65 $\mu\text{V/K}$) was found for Sb_2Te_3 films which were deposited by co-evaporation of elemental Sb and Te on glass substrates.^[60] Seebeck measurements of the 300 °C sample were not successful due to the low quality of the material. This sample was rather metallic due to the Te-deficiency, leading to a rather broad distribution of the thermoelectric voltage.

Sb_2Te_3 typically exhibits a strong tendency to form antisite defects and the stoichiometry strongly influences the Seebeck coefficient.^[61,62] The relative high Seebeck value, that is close

to optimize values of bulk materials,^[57] demonstrates a low defect density and low carrier densities in the order of 10^{19} to 10^{20} cm⁻³. Comparable values were recently reported by us for cold-pressed Sb₂Te₃ nanoplates as-obtained from a solution-based process.^[48] The average Seebeck value of the films deposited at 250 and 300 °C is significantly lower, most likely due to the increasing Sb-concentration as was determined by EDX.

Fig. 7. Distribution of Seebeck coefficients.

3. Conclusions. MOCVD deposition of thin Sb₂Te₃ films was achieved at substrate temperatures as low as 200 °C starting with the *single source precursor* (Et₂Sb)₂Te **1**. The substrate temperature was found to play a critical role on the chemical composition of the material film since Sb-rich material films were obtained at higher substrate temperatures. In order to achieve higher substrate temperatures, which most likely result in enhanced mobility of the adatoms and the formation of flat films, alternate *single source precursors* with enhanced thermal stabilities are currently under investigation. In addition, alternate substrate materials are investigated in order to improve the film morphology even at low substrate temperatures.

4. Experimental Section

Materials. Elemental tellurium was commercially available from Acros and used as received. (Et₂Sb)₂Te **1** was prepared according to a procedure as was previously reported.^[48]

MOCVD Reactor

MOCVD studies were performed in a cold-wall HV-MOCVD reactor (Fig. 8). The outer mantle of this home-made CVD reactor is made of glass, allowing 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 to 10^{-6} mbar. The distance between the substrate heater and the opening of the precursor feeding line can be varied from 1 to 8 cm (distance) and the orientation of the substrate heater to the precursor feeding line can be varied from 90° (orthogonal) to 180° (in line orientation). The precursor mass flow is controlled by a mass flow controller.

Fig. 8. Home-made MOCVD cold-wall reactor.

MOCVD Deposition. Sb₂Te₃ films were deposited on Si(100) substrates at a working pressure of 10^{-1} mbar within 90 minutes. The substrates were previously degreased with isopropanol, treated with hydrofluoric acid and heated to 500 °C at 10^{-3} mbar for 1 hour in the reactor in order to remove the native silicon dioxide layer. The precursor (0.1 g) was loaded into a bubbler attached to the MOCVD-reactor under inert conditions (Ar) and the bubbler temperature was set to 25 °C throughout the experiment. Argon was used as carrier gas and the precursor flow was controlled by use of a mass flow controller (MKS Instruments). After the film deposition was finished, the system was cooled to ambient temperature within 30 minutes under vacuum.

SEM Analysis. SEM studies were carried out on a Quanta 400 FEG ESEM or a Jeol JSM 6510 equipped with an energy dispersive X-ray spectroscopy (EDX) device (Bruker Quantax 400). A cross section sample of the Sb₂Te₃ film was prepared by use of a Jeol Cross-Section Polisher (IB-09010CP).

X-ray Analysis. XRD patterns were obtained using a Bruker D8 Advance powder diffractometer with Cu K α radiation (λ : 1.5418 Å).

Auger Spectroscopy. SAM/AES studies were carried out on a Perkin Elmer PHI 660 Scanning Auger Microprobe System.

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The low-temperature MOCVD deposition of Sb_2Te_3 films is achieved by use of the *single source precursor* $(\text{Et}_2\text{Sb})_2\text{Te}$ **1**. The Seebeck coefficient of the Sb_2Te_3 films deposited at 200 °C of up to 160 $\mu\text{V/K}$ demonstrates the high quality of the Sb_2Te_3 films.

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