

Atomic layer deposition for spacecraft applications

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The deposition of thin films by atomic layer deposition is a natural technological fit for manufacturing spacecraft components where weight, conformality, processing temperature, and material selection are all at a premium. Applications include optical, thermal control, electric charge dissipation, and protective coatings for the surprisingly reactive environment of low Earth orbit.

Indium oxide (IO) and indium tin oxide (ITO) are widely used in optoelectronics applications as a high quality transparent conducting oxide layer [1]. These coatings also are useful for enhancing the electrical properties of spacecraft thermal radiator coatings, where dissipating built-up static charge is crucial. In this work, we present the thickness-dependent electrical and optical properties of IO thin-films synthesized by ALD with the aim of finding the optimum condition for coating radiator pigments [2]. Trimethylindium and ozone were used as precursors for IO, while a tetrakis(dimethylamino)tin(IV) source was used for Sn doping to produce ITO. As-deposited IO films prepared at 140°C resulted in a growth per cycle of 0.46 Å/cycle and relatively low film resistivity.

For the case of ITO thin-films, an ALD process supercycle consisting of 1 Sn + 19 In cycles was shown to provide the optimum level of Sn doping corresponding to the 10 wt.% widely reported in the literature. These ITO films were deposited onto a nanoparticle pigment that was then combined with a binder to form the finished thermal control coating. Samples of our coating are currently on-board the International Space Station as part of the one-year MISSE-10 materials test mission.

Metal halide coatings provide protection from atmospheric oxidation of Al mirror components prior to flight. Their excellent transparency to short-wavelength light make them particularly suitable for orbiting UV astronomy platforms [3]. We have developed an ALD process for AlF₃ films using a TiF₄ and TMA precursor system. Film spatial nonuniformity, particle formation, and other challenges will be described in this talk along with our hypothesized reaction mechanism.

As a third spacecraft application, we will discuss the ALD of Ni and NiO films using a nickelocene and ozone precursor system [4]. Because of the grazing-incidence focusing mechanism of X-rays [5], these focusing elements consist of very high aspect-ratio microchannels. ALD is a perfect means of conformally coating these glass elements with a thin film of reflective (Ni) material. Reduction of NiO to Ni and the wettability of the glass substrate during the nucleation phase will be discussed.

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Amorphous chromium carbide coatings: low temperature DLI-MOCVD growth and characterization.

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Deposition processes are undergoing intensive development due to growing needs for increasingly demanding and diverse application properties. The process/coating couple, whether they are protective coatings of structural parts or functional thin films, is intimately linked to satisfy the expected performances. This observation applies to all deposition processes but the difficulties to overcome for their development mainly concern either their industrial viability or the intrinsic properties of the deposited material.

It is well known that one of the major constraints of CVD processes is the high deposition temperature. If it is too high the process cannot be applied to thermally fragile substrates because they could undergo dimensional deformations, residual stress variations, phase transformations or catastrophic degradation of their prior architecture as in microelectronics. For targeted applications, solutions have been found by activating the process for example by plasma, photons, or hot filament, and as a result, CVD processes often become hybrids.

The use of metalorganic precursors is another route to lower the deposition temperatures. The MOCVD processes found their industrial development mainly in microelectronics in the 1970s with the III-V semiconductors, now supplemented by multifunctional oxides. Although carbides, nitrides, borides and other compounds may be deposited by CVD, the applications of the CVD coatings of these families are limited because of too high deposition temperatures. For the most part, they can also be deposited by MOCVD with effective properties for applications as protective metallurgical coatings. In that case, depending on the precursors used, the lowering of temperature can be very important.

For example, chromium carbide coatings are in production at 1323 K by the halide CVD process (pack cementation) [1] whereas similar coatings in composition, structure and growth rate can be elaborated at 823 K by MOCVD [2]. This gain raises the temperature constraint but as it is due to the great reactivity of the vapor phase used, it is the scale up of the process which becomes complicated and which hinders its industrial development. In addition, in a wide range of growth temperatures, a diversity of composition, microstructure and therefore properties can be obtained, and it is crucial to know them.

With the aim of developing a large-scale MOCVD process for the deposition of CrC_x for the protection of various metal parts in extreme environments, a direct liquid injection system delivering high vapor flow rates was connected to a horizontal hot-wall reactor 1 m long [2]. The coatings obtained with this DLI-MOCVD process at 723 K have a composition close to Cr_7C_3 and a high nanohardness (23 GPa) [2]. Furthermore they exhibit a very good resistance to high temperature oxidation in air and steam [3]. As a result, they were used as internal protective coating of fuel cladding segments (Figure 1) [4]. A joint approach between kinetics experimental studies and numerical modeling has led to thick coatings with sufficient uniform thickness inside 1 m long fuel cladding tube (ID 8 mm) [5]. Optimized reactor parameters consist in a combination low pressure (~600 Pa) and temperatures as low as ~600 K. For this geometry in long and narrow tubes, the low temperature is essential. This paper reports the influence of low deposition temperatures on the main features of these CrC_x protective hard coatings (Table I).

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Table I. List of analytical techniques used to characterize the CrC_x coatings grown by DLI-MOCVD as a function of the deposition temperature whose results will be described and commented in this paper.

Temperature (K)	SEM-FEG	XRD	TEM	EPMA	Raman	RBS	TGA
773	yes	yes	N/A	yes	N/A	yes	N/A
723	yes	yes	yes	yes	yes	yes	yes
673	yes	yes	yes	yes	yes	N/A	N/A
648	yes	yes	N/A	N/A	N/A	N/A	N/A
623	yes	yes	N/A	yes	yes	yes	yes
598	yes	yes	TBD	yes	yes	yes	TBD

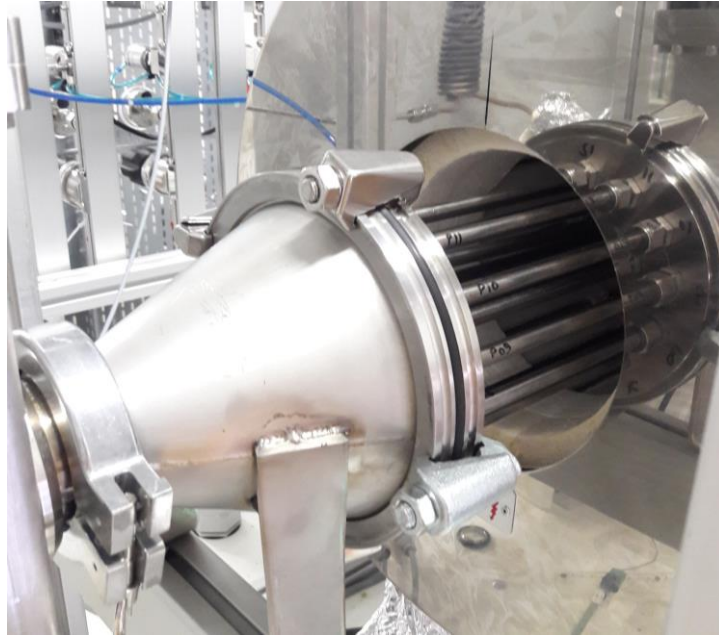


Figure 1. Picture showing the entrance of the DLI-MOCVD pilot used for the CrC_x deposition inside 16-fuel cladding tubes 1 m long in a single batch.

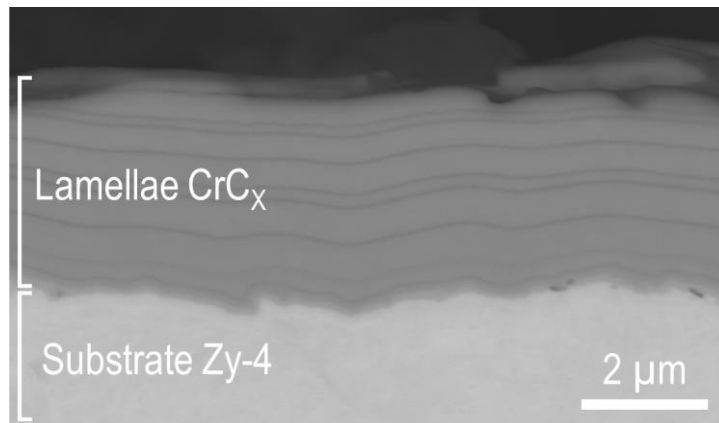


Figure 2. SEM-FEG cross section of a CrC_x amorphous coating grown at 598 K by DLI-MOCVD on the inner-wall of fuel cladding tube (Zr-4 alloy) exhibiting a self-organization as lamellar structure induced by in situ variations in the deposition temperatures around the set-point temperature of the furnace revealing the high sensitivity of the process in this low temperature kinetics regime.

Rational design of new NHC-stabilized copper(I) amide complexes for the deposition of copper containing nanostructures

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MOCVD and ALD techniques rely heavily on the precursors employed for the deposition of copper containing nanostructured materials that are highly relevant for microelectronics, sensors and photocatalysis. Especially the design of new copper precursors by systematic and logical variation of the organic ligand sphere remains a rare occurrence in recent literature. Herein, we report the synthesis of a series of new N-heterocyclic carbene (NHC) stabilized copper(I) amide complexes, that were subsequently evaluated for their potential application as precursors in MOCVD processes. While the influence of the NHC backbone with different substitution patterns on the thermal stability of copper(I) complexes was previously evaluated,^[1,2] the effect of the anionic backbone on thermal complex stability has not been investigated so far. Thus, in this study the substitution pattern of the neutral NHC backbone as well as the anionic organosilicon amide backbone were systematically varied. This results not only in spectroscopic similarities and differences as analyzed by ¹H-NMR, ¹³C-NMR, EI-MS and SC-XRD, but also significantly influences the thermal properties as derived by thermogravimetric analysis (Figure 1). It was found that the sterically less hindered *N*-*iso*-propyl substituted NHC backbones in complexes **3** and **4** was not able to impart the necessary thermal stability in combination with their highly reactive anionic organosilicon counterparts. However, the more sterically hindered *N*-*tert*-butyl substituted NHC backbones in complexes **1** and **2** were able to enhance the thermal stability with a difference of up to 60 °C. To demonstrate the application of this class of precursors, MOCVD experiments were conducted taking complex **2** as a representative Cu precursor together with oxygen for the deposition of nanostructured copper oxide thin films in the temperature range from 400 – 600 °C on Si(100) surfaces. The composition of the nanostructured copper-containing was analyzed by RBS/NRA, while crystallinity and morphology were evaluated *via* XRD (Figure 2a) and SEM (Figure 2b), respectively. Randomly oriented nanostructured Cu₂O whiskers were obtained at a deposition temperature of 500 °C in a vapor-liquid-solid (VLS) type growth mode. The systematic approach of tuning the precursor characteristics clearly showed that only small changes in the ligand sphere of the potential NHC-stabilized copper amide precursors can lead to a significant alteration of thermal properties, which influences the vapor phase deposition processes like CVD and ALD.

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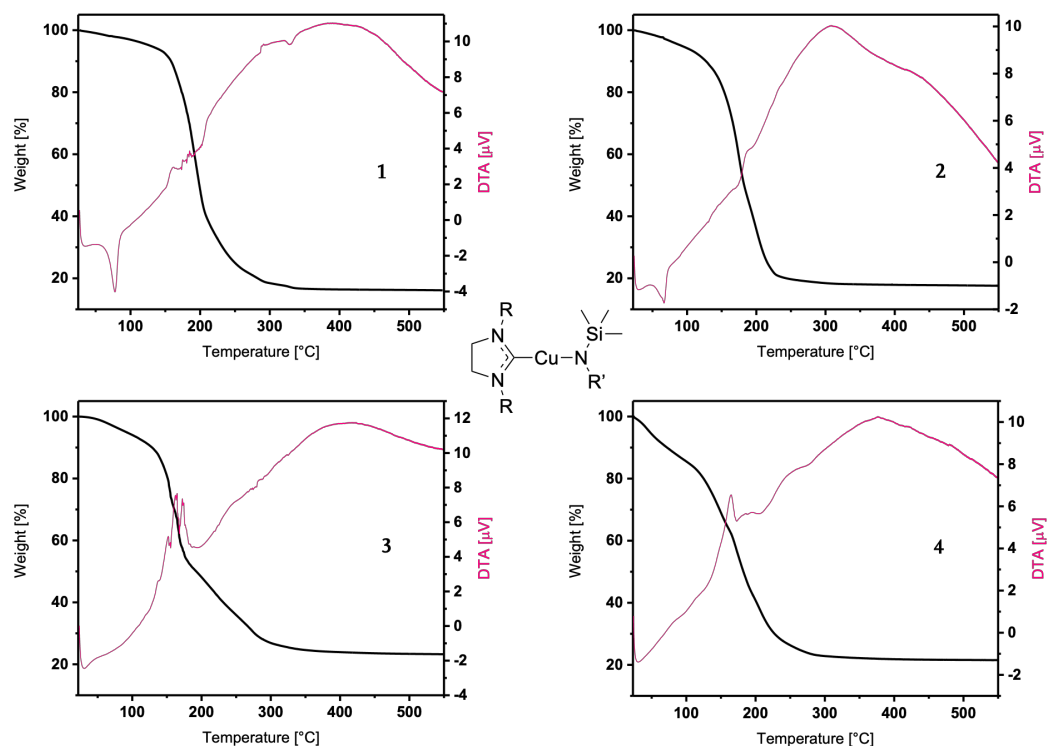


Figure 1. Thermogravimetric (black) and differential thermal (red) analysis of complexes 1 – 4.

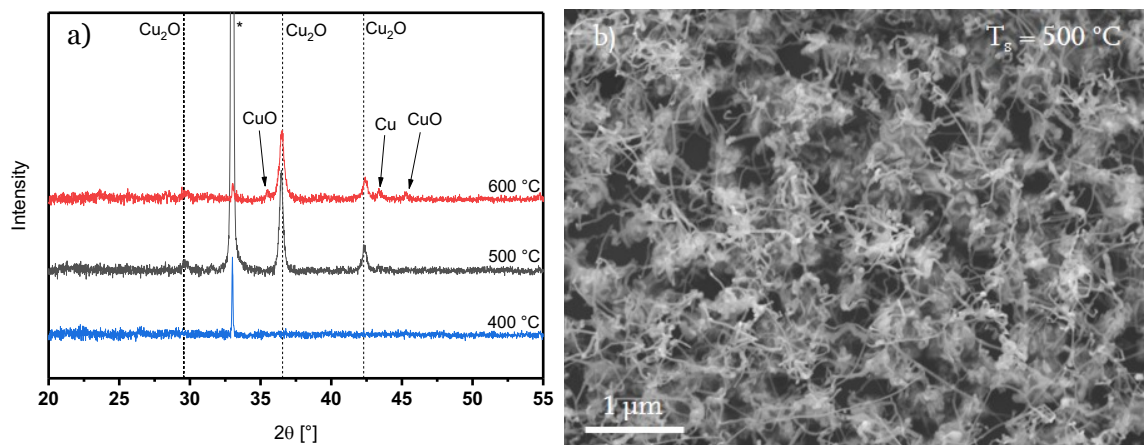


Figure 2. X-ray diffraction pattern of the copper containing nanostructured films on Si(100). b) SEM images of the randomly oriented whiskers deposited at 500 °C.

Highly uniform Al₂O₃ ultra-thin layers by seed-layer-free Atomic Layer Deposition onto monolayer epitaxial Graphene on 4H-SiC

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The Graphene (Gr) with its unique features, such as atomic thickness, excellent carrier mobility, and field-effect tunable ambipolar current transport, has attracted an increasing interest for the next generation of high frequency devices. Moreover, the deposition of thin high- κ layers on Gr is a crucial aspect to fully exploit its potential in microelectronic field. Atomic layer deposition (ALD) is the most promising method to obtain uniform films of high- κ dielectrics with atomic scale thickness control, at relatively low deposition temperature.

However, the lack of out-of-plane bonds in the planar sp² lattice of Gr is an obstacle to the uniform ALD nucleation and growth [1]. For this reason, several pre-functionalization or seeding layers on Gr have been reported as solutions to promote the ALD process. Nevertheless, these approaches adversely affect the Gr electrical properties or give rise to insulating films with increased equivalent oxide thickness and interface trapping [2]. To overcome these drawbacks, ALD growth of dielectrics on graphene, without pre-functionalization and seed-layers, is highly desirable.

In this work, a seed-layer-free thermal ALD approach at 250 °C, has been used to deposit ultrathin layers (~ 15 nm) of Al₂O₃ on Gr. In particular, highly homogeneous monolayer (1L) epitaxial graphene (EGr) (>98% 1L coverage) grown under optimized high temperature conditions on on-axis 4H-SiC(0001), has been employed as substrate. Uniform and pinhole-free Al₂O₃ film with smooth morphology can be observed, by atomic force microscopy (AFM) image, in fig.1a. The high uniformity of Al₂O₃ is not associated to the nature of SiC substrate, as demonstrated by the inhomogeneous Al₂O₃ coverage on 1L Gr transferred to SiC (fig.1b), but it is related to the peculiar buffer layer of EGr/SiC interface. Raman spectroscopy measurements, shown in fig.2, were carried out both on the virgin EG sample and after the Al₂O₃ deposition. No changes in the position and FWHM values of the characteristic G and 2D peaks of Gr can be observed after the Al₂O₃ deposition, indicating that the ALD process does not significantly affect the doping and strain of the EG. Ab-initio DFT calculations provide an enhanced adsorption energy for water molecules on highly n-type doped monolayer Gr, indicating the high doping of EGr induced by the underlying buffer layer as the origin of the excellent Al₂O₃ nucleation. Nanoscale resolution current mapping by conductive atomic force microscopy (C-AFM) showed highly uniform insulating properties of the Al₂O₃ thin films on 1L EG, with a breakdown field >8 MV/cm. These results will have important impact in epitaxial graphene device technology.

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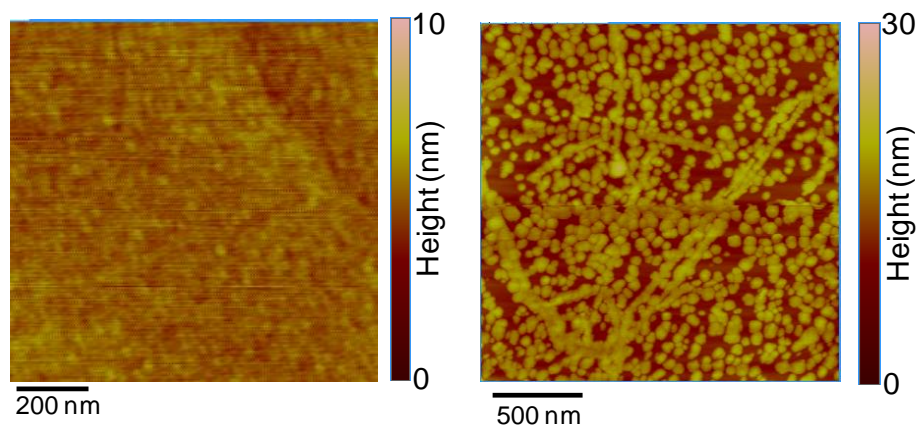


Figure 1. AFM morphology of Al_2O_3 coverage on 1L-epitaxial grown-Gr on 4H-SiC (a) and on transferred Gr on 4H-SiC (b).

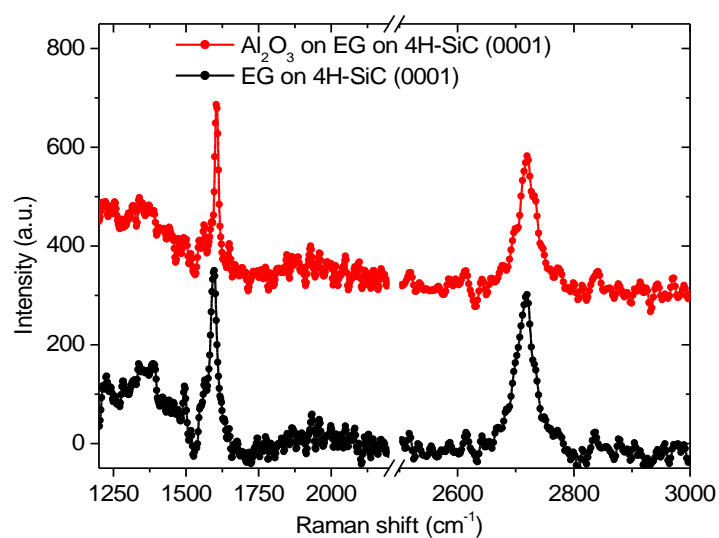


Figure 2. Raman spectra of virgin EG and after the Al_2O_3 deposition.

Van der Waals epitaxy of 2D materials using atomic layer deposition

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Van der Waals (vdW) epitaxy is a case of epitaxy where epitaxial registration of film to the substrate is enabled by van der Waals interactions at a film/substrate interface that consists of inert surfaces with no dangling bonds.[1,2] Whereas conventional epitaxy usually requires a close lattice match between the film and the substrate, the absence of covalent film-substrate bonding allows for vdW epitaxial growth of materials with very different lattice constants or even different crystal structures. A common case of vdW epitaxy is growth of a 2D material on either another 2D material or on a 3D material with a passivated surface. Therefore, vdW epitaxy also offers nearly limitless opportunities to grow various high quality 2D/2D or 2D/3D heterostructures. One of the main advantages of vdW epitaxy is reduction or even elimination of grain boundaries due to fixed rotational alignment of individual nuclei, which leads to improved material properties.

Initially, vdW epitaxy was mostly studied for different chalcogenides deposited under UHV conditions using MBE.[1,2] The recent boom of 2D materials, in particular transition metal dichalcogenides (TMDCs), has led to vdW epitaxy studies using CVD at high temperatures, typically 600–1000 °C. We have instead explored vdW epitaxy in conditions met in typical flow-type ALD reactors, i.e. mbar-level vacuum and temperatures below 500 °C. Our aim is to circumvent the typical difficulties in controlling the morphology as well as the grain size of 2D materials grown by ALD, two factors that have crucial effects on their properties.

Herein, we deposited a variety of 2D materials, including transition metal dichalcogenides HfS₂,[3] ReS₂,[4] SnS₂,[5] and ZrS₂[3] as well as PbI₂[6] at temperatures ranging from 75 to 400 °C. Apart from the triclinic 1T' structure of ReS₂, the materials crystallize in trigonal, 1T-type structure. We used two substrates commonly applied in vdW epitaxy, sapphire and muscovite mica. Epitaxial growth on mica was achieved for the 1T disulfides HfS₂, SnS₂, and ZrS₂ as studied by in-plane X-ray diffraction, which enables studies of ultrathin films only a few monolayers thick. These materials exhibited (001)MS₂ || (001)mica out-of-plane orientation. In-plane, preferred orientations of both [100] or [-100]MS₂ || [100]mica (0 or 60° domains, respectively) as well as domains rotated by 30°, i.e. [120] or [-1-20]MS₂ || [100]mica (30 or 90° domains) were observed in ϕ scans (Figure 1).

No epitaxial growth was observed for the 1T TMDCs on sapphire, whereas PbI₂ grew mostly epitaxially on sapphire though a small number of polycrystalline domains were also present. In this case, the preferred out-of-plane and in-plane orientations were (001)PbI₂ || (001)sapphire and [-110] or [1-10]PbI₂ || [-110]sapphire, respectively, both determined from an in-plane pole figure (Figure 2). No signs of epitaxy were seen for ReS₂, which may be due to its less symmetric 1T' structure. Attempts to deposit the commonly studied TMDCs with 2H structure, namely MoS₂ and WS₂, are in progress. Furthermore, we are studying the effect of the film-substrate registration on the functional properties of the grown materials, in particular in photodetector applications.

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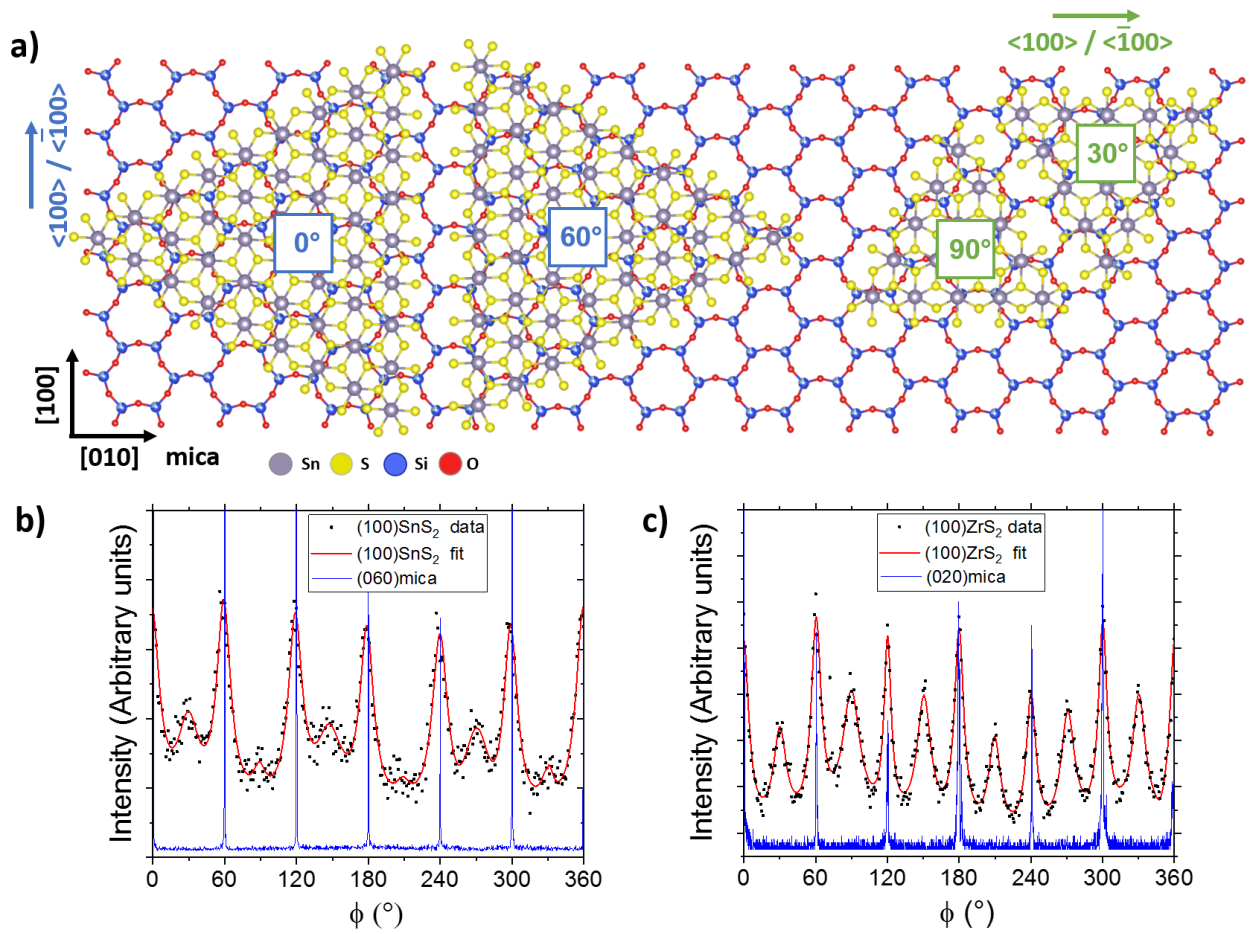


Figure 1. a) Schematic of the possible MS_2 (SnS_2) domains on muscovite mica. In-plane ϕ scans of b) SnS_2 and c) ZrS_2 films grown on muscovite mica showing the preferred in-plane orientations.

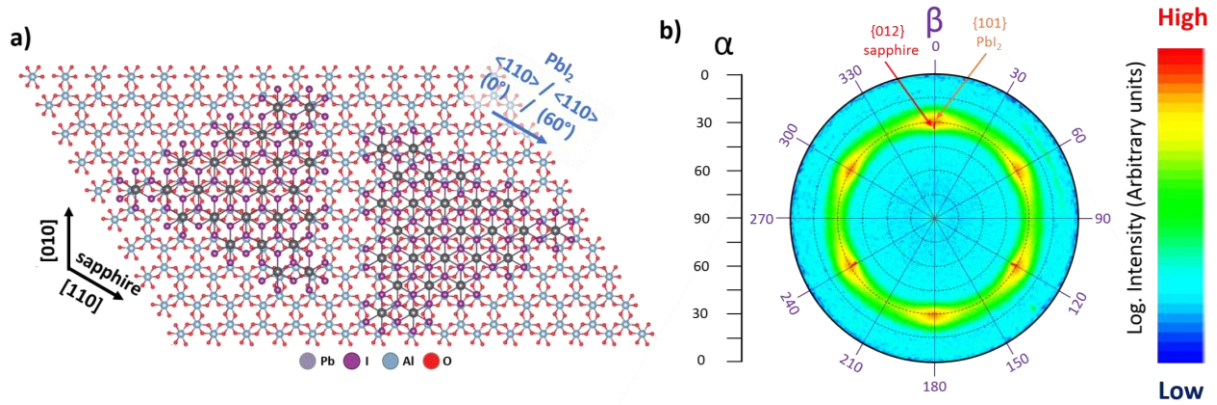


Figure 2. a) Schematic of the possible PbI_2 domains on sapphire. b) In-plane pole figure of a PbI_2 film grown on sapphire ($25.95^\circ 2\theta$), which was used to deduce both in-plane and out-of-plane registration between the film and the substrate.

Relationship Processing–Composition–Structure–Resistivity of LaNiO₃ Thin Films Grown by Chemical Vapor Deposition Methods

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In the perovskite-type ABO₃ family, Lanthanum Nickelate (LaNiO₃, or LNO) is a paramagnetic, metallic oxide with a bulk resistivity at room temperature around 2.5mΩ.cm [1]. As such, it is widely considered as an electrode in functional heterostructures. However, their integration can only be achieved if the relation binding the process parameters, the composition, the structure and the resistivity is clearly established.

In this work, LaNiO₃ thin films were deposited using three different Chemical Vapor Deposition supply systems, namely direct liquid injection, pulsed injection and aerosol generation [2]. One of the major parameters in a reliable CVD process is precursor evaporation. Hence, specific attention was given to the vaporization of both precursors: La(thd)₃ and Ni(thd)₂ (where thd stands for 2,2,6,6-tetramethyl-3,5-heptanedionato) and a link was established with the film's growth rate and composition. Additionally, a detailed explanation of the epitaxial growth of LaNiO₃ films on both (pseudo-) cubic (LaAlO₃, SrTiO₃) and trigonal (Al₂O₃) structures (Figure 1) is presented along with the impact of the rhombohedral distortion on the microstructural quality.

Highly epitaxial LaNiO₃/LaAlO₃ films with nearly stoichiometric composition exhibited a resistivity at room temperature comparable to that of the bulk (Figure 2). While it is generally assumed that oxygen deficiency is the main problem in highly resistive LNO films, films grown by CVD. In our films, grown with relatively high oxygen partial pressure, the resistivity was mainly dependent on the La/Ni composition and microstructural defects such as grain boundaries.

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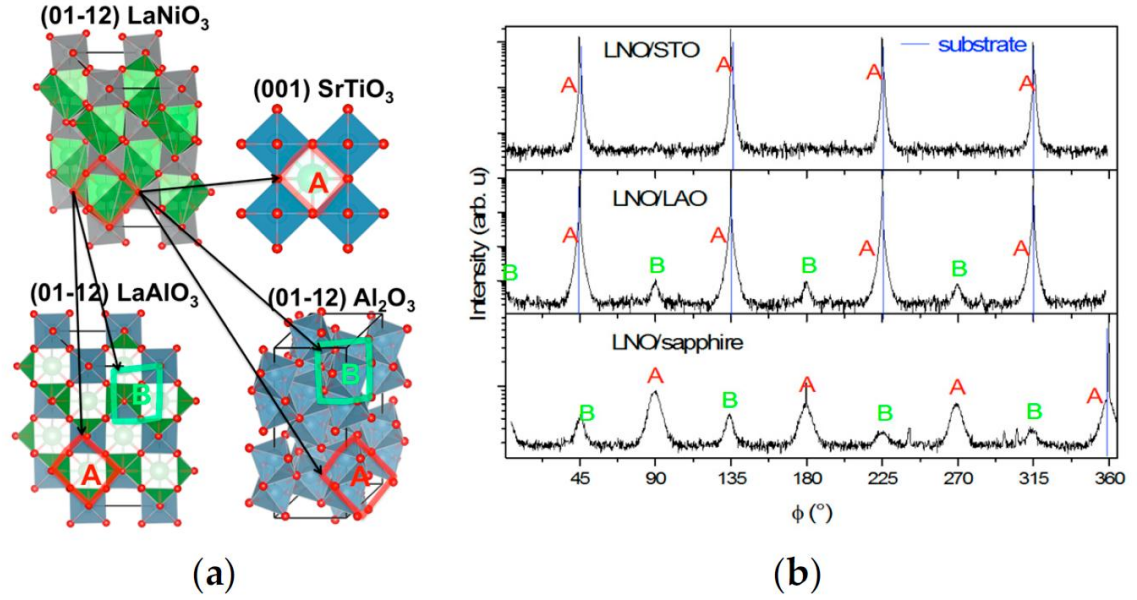


Figure 1. (a) Projections of polyhedral structure representations along the normal to the plane (001) of SrTiO_3 and to the (01 $\bar{1}2$) planes of LaNiO_3 , LaAlO_3 , and Al_2O_3 . Red balls represent oxygen anions, green balls-lanthanum or strontium cations, and blue balls-nickel, titanium, or aluminum cations. The LaNiO_3 cell matching on the surface of SrTiO_3 , LaAlO_3 , and Al_2O_3 are illustrated as well. The growth variants, rotated in the substrate plane by 45° with respect to each other, were named A and B. (b) XRD ϕ -scans of (0006)/{20 $\bar{2}2$ } planes of LaNiO_3 films on SrTiO_3 , LaAlO_3 , and Al_2O_3 substrates. [2]

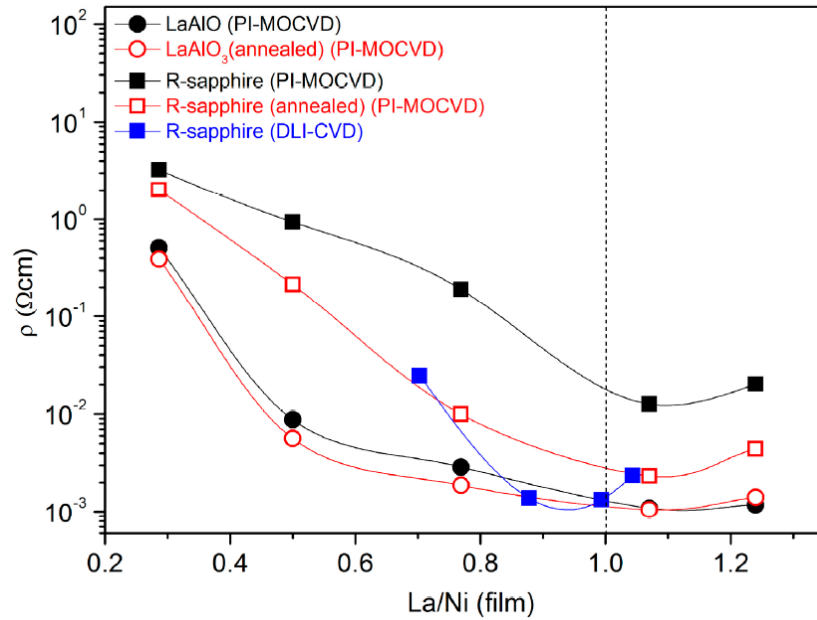


Figure 2. Composition dependence of resistivity of as-deposited and annealed (at 750 °C) LaNiO_3 films on LaAlO_3 and R-sapphire substrates grown by PI-MOCVD at 600 °C and by DLI-CVD at 750 °C [2]