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Atomic layer deposition: a key technology for the controlled growth of oxide thin films for advanced applications

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Abstract. Atomic layer deposition (ALD), also referred to as atomic layer epitaxy (ALE), was originally developed to process thin film electroluminescent structures for flat panel displays, which include as core components also insulating oxide layers such as alumina and aluminium titanium oxide. Another early application of oxide ALD was the processing of overlayers for catalysts. More recently, a major breakthrough for the ALD technology is the use of oxide thin films in microelectronics as gate and dynamic random access memory capacitor dielectrics. Besides giving a brief introduction to the ALD/ALE technology, the paper will address the present status of depositing binary and more complex (i.e. perovskite-type) metal oxides emphasizing precursor chemistry.

Key words: atomic layer deposition, atomic layer epitaxy, metal oxides, microelectronics, thin films, volatile precursors.

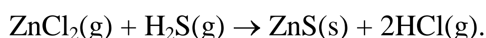
1. INTRODUCTION

A novel innovation called atomic layer epitaxy (ALE) for the processing of thin films for large-area flat panel electroluminescent (EL) displays was developed by T. Suntola and his co-workers in the 1970s [1,2]. The ALE technique, nowadays more often referred to as atomic layer deposition (ALD), and its applications have frequently been reviewed by several authors including us [3–6], Suntola [7–9], George et al. [10], and most recently in a comprehensive way by Ritala and Leskelä [11]. These reviews give a good insight into the development and special features of ALD, emphasizing its high application potential in materials technology. The present paper is of limited scope with focus on oxide ALD, because processing of oxide materials has been one of the most successful areas of the ALD technology where also the prospects for the future remain very bright, especially in microelectronics [12].

2. THE DEVELOPMENT OF ALD/ALE

The first ALE reactor was just a simple vacuum chamber designed for the deposition of zinc sulphide thin films directly from the elements [2]. By the use of a rotating substrate holder (Fig. 1), the substrates were exposed to alternating streams of gaseous precursors (Zn and S) provided by the zinc and sulphur furnaces, respectively. In this case no complicated chemistry was involved, but a direct reaction between the elements on the substrate surface produced the desired ZnS thin film.

With the advent of travelling wave or gas-flow type reactor introduced in 1978 (Fig. 2) more complex precursor chemistries could be exploited. Thus, zinc sulphide was prepared, also on a commercial scale, from zinc chloride and hydrogen sulphide:



$$ZnCl_2(g) + H_2S(g) \rightarrow ZnS(s) + 2HCl(g).$$

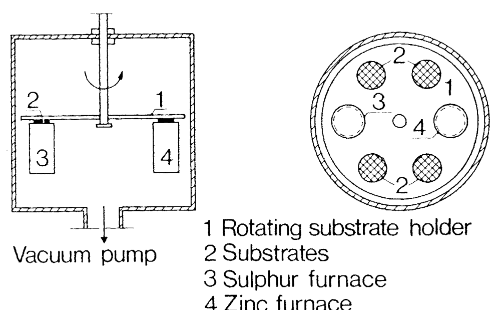


Fig. 1. ALD reactor based on rotating substrate holder to produce ZnS thin films directly from the elements [2].

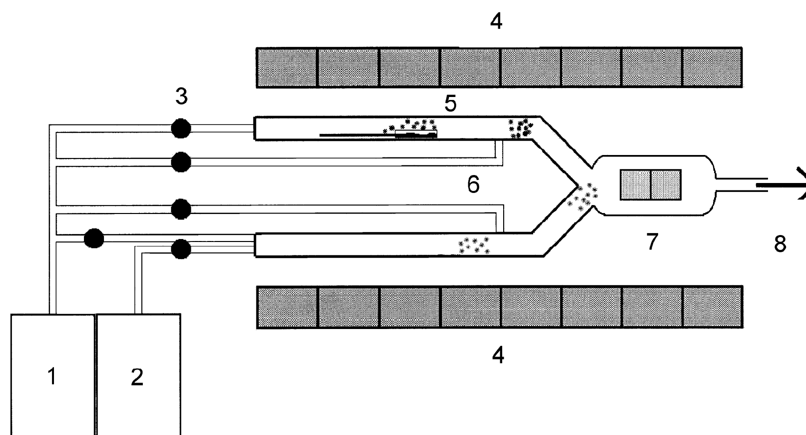


Fig. 2. Schematic view of an ALE reactor of travelling wave type. (1) N_2 generator, (2) source for gaseous precursor, (3) pulsing valves, (4) heating elements, (5) source boat for solid precursor, (6) inlets for purge gas, (7) substrates, (8) outlet to vacuum pump [5].

The reaction mechanism of ZnS formation, including the surface species involved, has been investigated in detail by Pakkanen et al. [13] using quantum chemical calculations. ZnS thin films are commercially exploited in EL displays, which emit bright yellow light when doped with manganese. This was the first major industrial application of the ALD technology.

Other ALD applications explored include epitaxial compound semiconductors, optical and protective coatings, catalysts, solar cells, and gas sensors. However, since the 1990s major interest in ALD, as mentioned above, stems from the semiconductor industry.

3. OXIDE ALD

The ZnS : Mn EL-emitting layer requires on its both sides insulating layers. This was the beginning of oxide ALD on an industrial scale because the dielectric–semiconductor–dielectric stack of thin films can be effectively and economically processed in one pump-down by ALD. The insulating oxide materials used in commercial displays are alumina and aluminium titanium oxide (Fig. 3; [14]). It should be noted here that Aleskovskii and others ([15] and references therein) have also described an ALD-like process. The method was called molecular layering and applied to the growth of silica overlayers on silicon from $SiCl_4$ and H_2O precursors.

Another early application of ALE was the adsorption-controlled preparation of solid catalysts [16]. In this technique, silica and alumina catalyst supports with a high surface area are coated from the gas phase with volatile metal-containing precursors, which then after adsorption can be converted to catalytically active oxides. Thus, for instance ZrO_2 and CrO_x have been deposited using the ALD reactions $ZrCl_4$(ad.) + H_2O and Cr(acac)$_3$(ad.) + O_2, respectively [17,18].

A further example of the surface-controlled nature of ALD and its exploitation is the coating of atomic force microscopy tips by ALD-deposited thin SnO_2 layers [19]. The original complex shape of the silicon tip is strictly preserved even when the coating is 470 nm thick (Fig. 4).

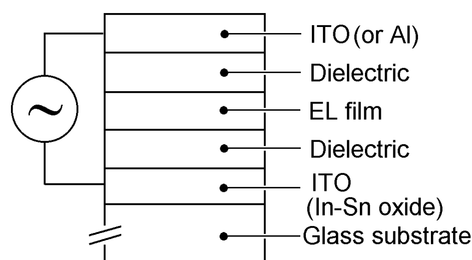


Fig. 3. Schematic structure of a thin film electroluminescent device.

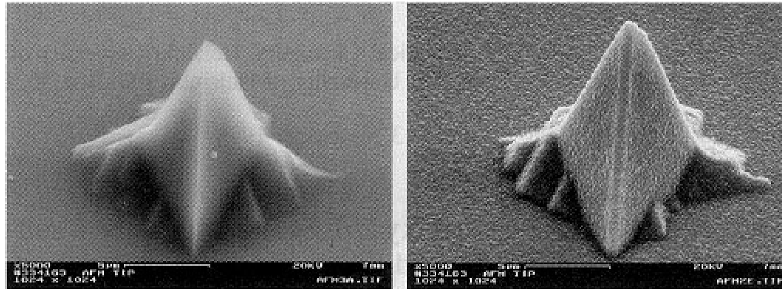


Fig. 4. SEM images of the atomic force microscopy tips when coated with 30 nm (left) and 470 nm (right) thick layers of ALD-grown SnO_2 [[19]].

Oxide ALD has also been successfully used to deposit tin dioxide overlayers for gas sensors [[20,21]]. As demonstrated by Dücsô et al. [[20]], the inherent surface-controlled properties of an ALD process can be exploited to an extreme when pores with a high aspect ratio (140 : 1) in porous silicon are conformally coated. This was corroborated by cross-sectional transmission electron microscopy and secondary ion mass spectrometry studies.

The inherently slow deposition rate in silica ALD can be overcome by using the catalytic process recently proposed in [[22]]. This ALD-like process yields highly conformal silica and alumina nanolaminates at rates of 12 nm or 32 monolayers per cycle. The cycle time is considerable, however.

Until now, the three most often studied ALD processes are the formation of Al_2O_3 from trimethylaluminium and water, as well as the Zr and Hf chloride processes with water to produce ZrO_2 and HfO_2, respectively. These processes have gained interest as producing materials to replace SiO_2 as gate oxide in metal-oxide-semiconductor field-effect transistor to be discussed in more detail below.

3. ALD FOR SEMICONDUCTOR INDUSTRY

Currently, a major technological change is taking place in the semiconductor industry, where silicon dioxide having a dielectric constant of 3.9 will be replaced by so-called high-k materials [[23]]. This change to new materials is necessary as the packing densities increase (Moore's law) and SiO_2 may no longer serve as a gate dielectric due to its rather low k-value. Therefore materials with a k-value higher than that of SiO_2 have to be used as gate dielectrics.

Candidates for gate dielectrics include the oxides of aluminium ($k = 9$), yttrium ($k = 15$), lanthanum ($k = 30$), zirconium ($k = 25$), and hafnium ($k = 25$) [[23]]. Production of Al_2O_3, from trimethylaluminium and water forms an almost ideal ALD process, but the moderate k value of alumina is a major drawback. On the other hand, the oxides of Zr and Hf exhibit great potential, but the chloride process to produce them has its limitations. Achieving extremely low equivalent

oxide thickness values has proven to be difficult because the nucleation directly onto native oxide-free Si-substrate surface is poor leading to a nonuniform microstructure [[24]]. Also particle contamination and halide impurities may cause problems. Thus, other precursors as well as barrier layers in the Si-high-*k* interface need to be considered.

Being a chemical method, ALD offers a wide range of precursor combinations for the deposition of metal oxides. Thus, several precursor chemistries have been developed for the deposition of zirconia thin films. As a source for the Zr metal, volatile chlorides, iodides, alkoxides, alkylamides or β-diketonate-type precursors may be used, while oxygen is usually provided by water but more efficiently by ozone or hydrogen peroxide, for instance (cf. Fig. 5 in [[25]]). In order to improve the quality of ZrO_2 thin films and to widen the process window, Kim et al. [[26]] have recently employed plasma-enhanced ALD when processing ZrO_2 from Zr-t-butoxide and $Zr(NEt_2)_2$ precursors. The latter precursor and its Hf analogue together with water appear to be interesting precursor alternatives even without plasma as good quality and conformal zirconia and hafnia overlayers on hydroxylated surfaces have been produced [[27]]. The step coverage, including theoretical modelling, has been studied in the case of the $Hf(NMe_2)_4$ precursor [[28]].

As an example of the novel precursor choices available, the recently developed ALD process for ZrO_2 based on organometallics will be described below. The true Zr organometallics, viz. the cyclopentadienyl (Cp) compounds like $Cp_2Zr(CH_3)_2$, are more volatile and reactive than the chlorides and β-diketonates, thus usually yielding higher thin film growth rates at low deposition temperatures [[29,30]]. In contrast to the halide processes, the incorporation of chlorine can be totally avoided in Cp-based processes where in general the contents of other impurities are also low within the ALD-window, i.e. the temperature window for the surface-controlled growth. The ALD process for zirconia can also be used for the production of yttrium-stabilized zirconia when the ALD process for Y_2O_3 is incorporated

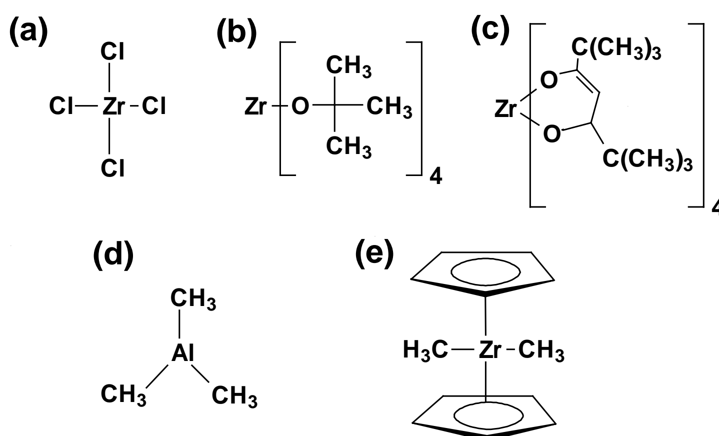


Fig. 5. Examples of precursors for oxide ALD. Volatile (a) halides, (b) alkoxides, (c) β-diketonates, (d) organometallics, and (e) organometallic cyclopentadienyl-type compounds have been exploited [[25]].

in the zirconia process as demonstrated recently in [31]. Other examples of the use of Cp compounds include Cp_2Mg and Cp_3Sc in the deposition of MgO [32] and Sc_2O_3 [33], respectively, where the growth rates were significantly enhanced by this precursor selection. Another advantage of the use of Cp compounds is that water is reactive enough as oxygen source, thus reducing the oxidation of the interface and the silicon substrate. As a rule, this takes place when ozone is used as oxygen source and may be detrimental to the functional properties.

4. SOME PROBLEMS IN ALD CHEMISTRY AND HOW TO SOLVE THEM

In the case of zinc sulphide processing of thin films from $ZnCl_2$ and H_2S it has been possible to resolve the reaction mechanism by a theoretical approach supported by experimental data [13], but this is no longer feasible when the complexity of the precursor molecules increases. However, some progress can be made by combining X-ray diffraction, spectroscopic, and thermoanalytical data. For instance, the unexpectedly high growth rate of ZnO from the zinc acetate precursor can be related to the formation in the gas phase and geometry of the tetrameric oxometallate complex $Zn_4O(Ac)_6$ [34,35].

Quartz crystal microbalance provides a rather simple but effective way for *in situ* probing of the growth process as shown by the Tartu group of Estonia [36] for TiO_2 growth, for example. Quadrupole mass spectrometric (QMS) studies of the vaporized precursors $Sc(thd)_3$ (thd = 2,2,6,6-tetramethylheptane-3,5-dionate [37], where thd is a β-diketonate-type ligand), have revealed that the precursor loses one of its thd-ligands forming the $Sc(thd)_2^+$ ion, which is responsible for the ALD growth of the Sc_2O_3 thin film. *In situ* QMS studies can also give important information of the growth mechanism as Rahtu [38] has described.

A major problem with the precursors for alkaline and rare earth elements is their affinity to the ambient (air or water vapour). This can be avoided by two innovative approaches as illustrated by examples from the rare earth and alkaline earth chemistry [6]. Rare earth and alkaline earth ions have large sizes, in other words, large ionic radii and thus they are in many of their compounds coordinatively unsaturated. With time this leads easily to reactions with the ambient and to oligomerization, which may reduce or destroy the volatility needed for the compound to function as a precursor in an ALD process.

The two approaches devised to overcome the problem are the following:

(i) adducting the precursor and this way saturating its coordination sphere and protecting the metal ion from reacting with the ambient [25], and

(ii) *in situ* synthesis of the precursor to provide a fresh and reactive supply of it onto the substrate [39].

The first approach is illustrated in the case of β-diketonate complexes of yttrium where the coordination number can be raised from the original six to a chemically more acceptable eight by adducting the $Y(thd)_3$ complex with nitrogen donor ligand (Fig. 6). The relatively weak metal–nitrogen bonds then break upon

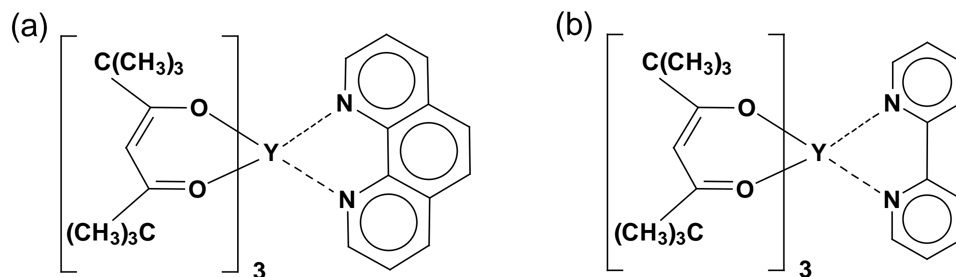


Fig. 6. Y(thd)$_3$ adducts with (a) 1,10-phenanthroline and (b) 2,2-bipyridyl [25].

volatilization, yielding an Y(thd)$_3$ precursor molecule to adsorb onto the substrate and to react during the next step with an oxygen donor.

The *in situ* process likewise aims at providing a fresh and reactive supply of the precursor onto the substrate surface to react there with the oxygen donor. The feasibility of the *in situ* approach has been demonstrated in depositing metal sulphide thin films [39] but it could be used in oxide ALD as well.

The choice of oxygen source is an important factor in oxide ALD. Usually water has been employed as oxygen source. But in the case of less reactive precursors, such as β-diketonates, ozone is needed. In particular, when producing rare-earth (RE) oxide thin films the combination of RE(thd)$_3$ with ozone has proven its usefulness [25]. As described above, however, ozone effectively oxidizes the Si-substrate and thus increases the equivalent oxide thickness in gate dielectric applications. On the other hand, ozone produces dense films with low impurity levels as carbon is burned off.

After the deposition, annealing may be used to crystallize the film and/or to induce chemical or structural changes. An example of the annealing effects is provided by Fig. 7, which shows that if La_2O_3 is processed from La(thd)$_3$ and

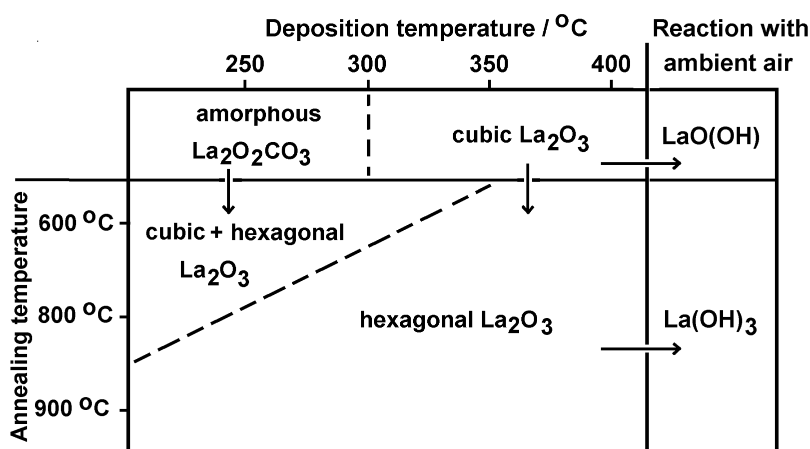


Fig. 7. Summary of the influence of deposition and annealing temperatures on La_2O_3 film composition. Also the effect of ambient air on film quality is shown [40].

ozone precursors around 300 °C, oxocarbonate is formed, which may be transformed to La_2O_3 by annealing at higher temperature [40,41]. Similarly, the annealing effects on $SrTiO_3$ thin films have been studied and found that as-deposited films contain $SrCO_3$ but subsequent annealing leads to stoichiometric $SrTiO_3$ [42].

5. PSEUDOBINARY AND TERNARY OXIDES

Most studies in our laboratory have been focused on yttrium-stabilized zirconia, but other doped oxides, for example Al_2O_3 : P and solid solutions (ceria-gadolinia [43]), have been considered as well. The most comprehensive ALD investigations, however, have been made into the processing of perovskite oxides, e.g. $LaAlO_3$ [44] and $LaGaO_3$ [45]. These materials have many potential applications in microelectronics as insulating and buffer layers.

In general, the processing of a ternary compound, e.g. $LaAlO_3$, is more demanding than that of its component oxides, viz. La_2O_3 and Al_2O_3, because the ALD windows of both component oxides must have an overlapping temperature region where the ternary compound can be deposited (Fig. 8). In the case of $LaAlO_3$, stoichiometric thin films can be deposited at 325–375 °C [44]. When the deposition was performed onto (1 0 0) $SrTiO_3$, epitaxial growth after annealing was observed and the impurity levels of the film were low.

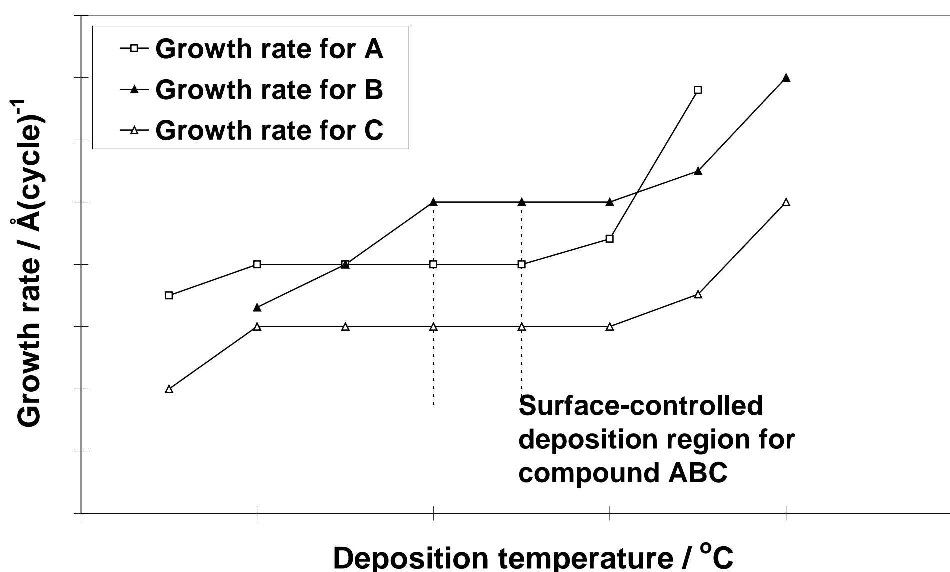


Fig. 8. Possible ALD temperature region of constant growth, viz. the ALD window, for an imaginary compound ABC as a function of the deposition temperatures of the binary compounds [25].

6. CONCLUSIONS

Atomic layer deposition is now a mature technique, which has established its position as a method-of-choice for the deposition of thin films for advanced applications. The wide range of volatile metal-containing precursors and various oxygen sources available enable the processing of thin films of desired characteristics. By properly choosing the precursor chemistry and the substrate, oxide thin films can be deposited with low impurity levels and also epitaxially, if needed. The innovative approaches developed, viz. *in situ* synthesis and the adducting of the precursor, assist in providing a fresh and reactive supply of the precursor for depositions. The compositional range of the oxides covers, besides the binary oxides, also the pseudobinary and ternary compounds, where especially the perovskites find many technical applications. Also doping by ALD, when needed, is straightforward.

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Aatomkihtsadestamine kui tänapäeval suurt rakenduslikku tähtsust omav õhukeste oksiidkilede kontrollitava kasvatamise menetlus

Lauri Niinistö

Aatomkihtsadestamine arendati kõigepealt välja oksiidkilesid sisaldavate elektroluminestsentspaneelide tootmiseks. Teine rakendusala puudutab oksiidkiledega aktiveeritud katalüsaatoreid. Hiljutiseks läbimurdeks tuleb pidada aatomkihtsadestamismenetluse haarde laiendamist mikroelektroonikas vajalikele oksiiddielektrikutele. Artiklis on antud menetluse üldiseloomustus ja peatutud selle staatusel binaarsete ja keerulisemate (perovskiit-) metalloksiidide kasvatamisel. Suuremat tähelepanu on pööratud prekursorkeemiale.