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Title	Crystallinity of inorganic films grown by atomic layer deposition: Overview and general trends
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Citation	Journal of Applied Physics. Vol. 113 (2013) No: 2, 101 p.
Date	2013
URL	http://dx.doi.org/10.1063/1.4757907
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Crystallinity of inorganic films grown by atomic layer deposition: Overview and general trends

Ville Miikkulainen, Markku Leskelä, Mikko Ritala, and Riikka L. Puurunen

Citation: J. Appl. Phys. **113**, 021301 (2013); doi: 10.1063/1.4757907
View online: http://dx.doi.org/10.1063/1.4757907
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APPLIED PHYSICS REVIEWS

Crystallinity of inorganic films grown by atomic layer deposition: Overview and general trends

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(Received 10 November 2011; accepted 29 August 2012; published online 8 January 2013)

Atomic layer deposition (ALD) is gaining attention as a thin film deposition method, uniquely suitable for depositing uniform and conformal films on complex three-dimensional topographies. The deposition of a film of a given material by ALD relies on the successive, separated, and self-terminating gas–solid reactions of typically two gaseous reactants. Hundreds of ALD chemistries have been found for depositing a variety of materials during the past decades, mostly for inorganic materials but lately also for organic and inorganic–organic hybrid compounds. One factor that often dictates the properties of ALD films in actual applications is the crystallinity of the grown film: Is the material amorphous or, if it is crystalline, which phase(s) is (are) present. In this thematic review, we first describe the basics of ALD, summarize the two-reactant ALD processes to grow inorganic materials developed to-date, updating the information of an earlier review on ALD [R. L. Puurunen, J. Appl. Phys. **97**, 121301 (2005)], and give an overview of the status of processing ternary compounds by ALD. We then proceed to analyze the published experimental data for information on the crystallinity and phase of inorganic materials deposited by ALD from different reactants at different temperatures. The data are collected for films in their as-deposited state and tabulated for easy reference. Case studies are presented to illustrate the effect of different process parameters on crystallinity for representative materials: aluminium oxide, zirconium oxide, zinc oxide, titanium nitride, zinc zulfide, and ruthenium. Finally, we discuss the general trends in the development of film crystallinity as function of ALD process parameters. The authors hope that this review will help newcomers to ALD to familiarize themselves with the complex world of crystalline ALD films and, at the same time, serve for the expert as a handbook-type reference source on ALD processes and film crystallinity. © *2013 American Institute of Physics*.
[http://dx.doi.org/10.1063/1.4757907]

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I. INTRODUCTION

Atomic layer deposition (ALD) is gaining attention as a thin film deposition method, uniquely suitable for depositing uniform and conformal films on complex three-dimensional topographies. The indifference of ALD to substrate shape facilitates the use of ALD in highly different fields of technology, e.g., dynamic random access memory (DRAM) and metal–oxide–semiconductor field-effect transistor (MOSFET) production in microelectronics, thin-film electroluminescent (EL) displays (the oldest industrial application of ALD), catalysis, solar energy, microelectromechanical systems (MEMS), coating of fibers—in fact almost any area of nanotechnology research. The rising number of scientific publications on ALD per year, shown in Figure 1, testifies to the increasing world-wide interest towards ALD.

The deposition of a film of a given material by ALD relies on the successive, separated, and self-terminating gas–solid reactions of typically two gaseous reactants. Hundreds of ALD chemistries have been found for depositing a variety of materials during the past decades, mostly for inorganic materials but lately also for organic and inorganic–organic hybrid compounds. ALD processes are at different levels of technology maturity: Some have been in continuous industrial use for more than two decades (e.g., the process to deposit Al_2O_3 from chloride reactants),[1] some have been shown to work in small laboratory-scale reactors with no commercially obtainable reactants yet, while most ALD processes are somewhere in between.

One factor that often dictates the properties of ALD films in actual applications is the crystallinity of the grown film: Is the material amorphous or, if it is crystalline, which phase(s) are present. Amorphous films are desired, for example, as diffusion barriers, since grain boundaries between individual crystals would provide a leakage path for the diffusion of ions and lead to device failure. Crystalline films

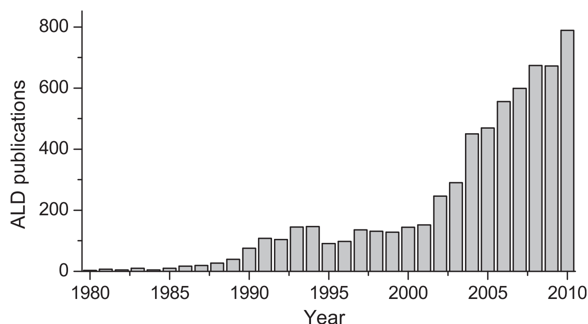

FIG. 1. The number of scientific publications on ALD published per year during 1980 to 2010, analysed from the Web of Science.

with a specific phase, in turn, are often desired for their chemical or electrical properties. It is not trivial to know in advance, whether a given ALD process will result in an amorphous or crystalline film, and in the case of a crystalline film, what phase, orientation, crystal size, etc. are present, but these need to be experimentally determined.

The main goal of this review is to collect experimental data on the crystallinity of ALD films in their as-deposited state, to analyze the data superficially for all ALD processes and in more detail for selected cases, and finally to report general trends in the evolution of crystallinity of ALD films as function of important growth parameters. Collecting the experimental data has been a major effort and has included analyzing a major part of the published ALD literature. While working to achieve the main goal, we have also reviewed the ALD process development that has occurred during the past years, and as a secondary goal it has been to update the information of two-reactant ALD processes published in an earlier ALD review by one of the authors.[2] The earlier review,[2] whose main target was to review the $AlMe_3 - H_2O$ ALD process in detail, covered ALD publications until early 2005, while the current work includes articles published by the end of 2010. Please note that two of the current authors have earlier reviewed many general aspects of ALD, which are not covered in this work.[3,4]

Since the ALD reviews were listed in the earlier review,[2] several reviews have been published on various specific aspects of ALD, which the readers may find useful. Putkonen *et al.*[5] reviewed the analysis of ALD films by ion-beam techniques; Putkonen and Niinistö[6] the use of organometallic reactants (precursors) for ALD; Elers *et al.*[7] the film uniformity in ALD; Schumacher *et al.*[8] ALD and "AVD" (atomic vapour deposition) for next-generation microelectronic devices; Jones *et al.*[9] ALD (and MOCVD) of high-κ oxides; Knez *et al.*[10] the fabrication of nanostructures by ALD; Zaera[11] the surface chemistry of ALD for electronic device manufacturing; Sherman[12] general aspects of ALD; Ritala and Niinistö[13] ALD in general; Niinistö *et al.*[14] high-κ oxides of Group 4 metals for memory applications; Kim *et al.*[15] applications of ALD to nanofabrication and emerging nanodevices; Clavel *et al.*[16] non-aqueous sol–gel routes to ALD of oxides; George[17] ALD in general; Puurunen *et al.*[18] the use of ALD for MEMS; Bakke *et al.*[19] the nanoengineering and interfacial engineering of photovoltaics (PV) by ALD; Profijt *et al.*[20] the basics, opportunities, and challenges of plasma-assisted ALD; Detavernier *et al.*[21] the tailoring of nanoporous materials by ALD; George *et al.*[22] metalcone molecular layer deposition (MLD); Marin *et al.*[23] ALD in corrosion protection; Parsons *et al.*[24] the progress in ALD chemistry; Leskelä *et al.*[25] novel materials by ALD and MLD; Bae *et al.*[26] ALD for 3D nanostructure fabrication; Elam *et al.*[27] ALD for clean energy conversion, utilization, and storage; Kessels and Putkonen[28] advanced ALD process technologies (e.g., PEALD, roll-to-roll); Peng *et al.*[29] ALD for electrochemical energy generation and storage; Im *et al.*[30] ALD for plasmonics and nanobiotechnology; Knoops *et al.*[31] ALD for nanostructured Li-ion batteries; Zaera[32] the surface chemistry of ALD of solid films; Elliott[33] the atomic-scale simulation of ALD chemistry; Knez[34] the

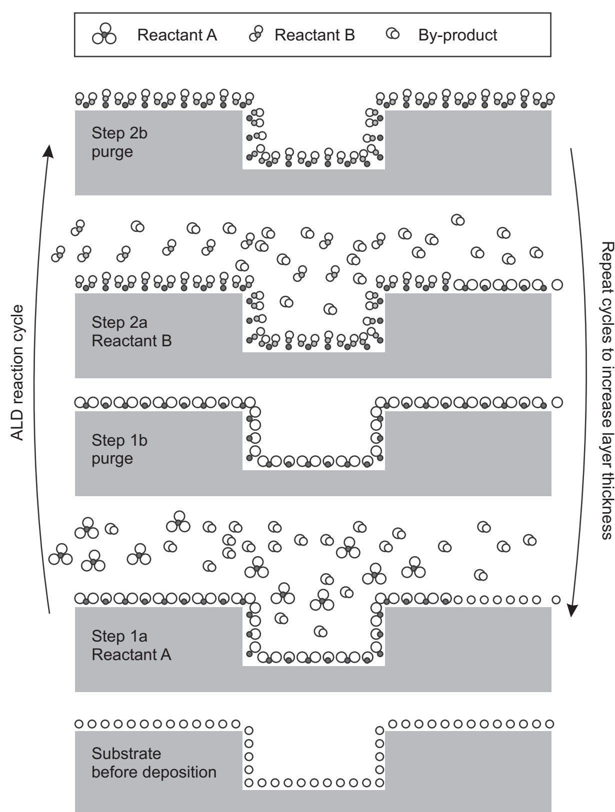

FIG. 2. Schematic illustration of one ALD reaction cycle.

diffusion phenomena in ALD; van Delft *et al.*[35] ALD for photovoltaics; and Wiemer *et al.*[36] ALD rare-earth-based binary and ternary oxides for microelectronic applications. Nilsen and co-workers published a valuable series of articles on the modeling of the growth of crystalline ALD layers from seed objects.[37–40]

The structure of this review is as follows. We first describe the basics of ALD, summarize the two-reactant ALD processes to grow inorganic materials developed to-date, updating the information of an earlier review on ALD,[2] and give an overview of ternary materials made by ALD. We then proceed to analyze the published experimental data for information on the crystallinity and phase of inorganic materials deposited by ALD from different reactants at different temperatures. The data are tabulated for easy reference. Case studies are presented to illustrate the effect of different process parameters on crystallinity for representative materials: aluminium oxide, zirconium oxide, zinc oxide, titanium nitride, zinc sulfide, and ruthenium. Finally, we discuss the trends in the development of ALD film crystallinity as function of ALD process parameters.

II. ALD PROCESSES: BASICS

This section describes the basics of ALD, summarizes the two-reactant ALD processes to grow inorganic materials developed to-date, updating the information of an earlier review on ALD,[2] and gives an overview of ternary materials made by ALD.

A. Principles of ALD

The principles of ALD are here described in sufficient detail that the readers can grasp an understanding of what ALD is, and to support the discussion on the crystallinity of ALD films from theoretical and practical viewpoint. Those looking for a more thorough description are referred to other sources, e.g., Refs. 2 and 3.

ALD is a chemical vapor deposition (CVD) technique that relies on saturating and irreversible, separated gas–solid reactions of typically at least two compounds, which are repeated in a cyclic manner. One ALD cycle consists typically of four steps, as schematically illustrated in Figure 2: first gas–solid reaction, i.e., chemisorption reaction, of the first reactant (Reactant A), typically of a metal reactant (Step 1a); purge or evacuation to remove the unreacted precursor and gaseous by-products (Step 1b); second gas–solid reaction, i.e., chemisorption reaction, of the second reactant (Reactant B), typically of a non-metal reactant (Step 2a); and again purge or evacuation to remove the unreacted precursor and gaseous by-products (Step 2b). (Please note that the compounds used to deposit film by ALD are often called precursors or reactants. Sometimes, a distinction is made to call the metal compound "precursor" and the non-metal compound "reactant." In this work, such distinction is not made but the two terms are used interchangeably.)

Essential characteristics of ALD processes, required to have a uniform thin film on large-area substrates and complex 3D shapes, are the *irreversible* and *saturating* reactions. Figure 3 compares the saturating and irreversible gas–solid reactions (often called self-terminating, self-limiting, etc.) to other types of adsorption. It is clear that only irreversible and saturating reactions, which are allowed to saturate, automatically lead to the same amount of material adsorbed on different parts of large and/or complex-shaped substrates, irrespective of how much reactant was available and of the exposure and purge times. As discussed elsewhere, reactant partial pressures also do not influence the amount of material adsorbed in saturating, irreversible reactions.[2] This automatic control of the amount of material deposited is a key feature of ALD.

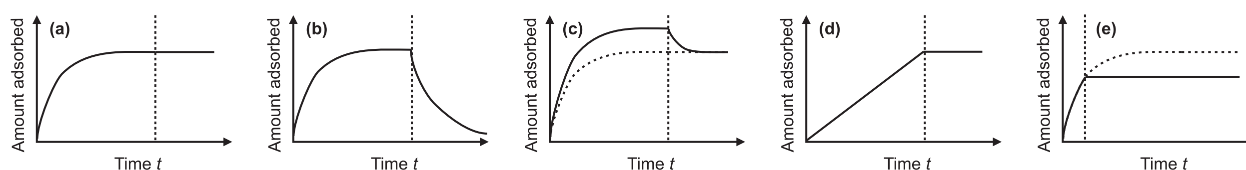

FIG. 3. Schematic illustration of different types of adsorption: (a) irreversible saturating adsorption (as required for ALD), (b) reversible saturating adsorption, (c) combined irreversible and reversible saturating adsorption, (d) irreversible non-saturating adsorption (i.e., deposition), and (e) irreversible saturating adsorption not allowed to saturate. The dashed vertical line denotes the end of reactant pulse (Step 1a or 2a) and the beginning of purge sequence (Step 1b or 2b), as described in the text. Reprinted with permission from J. Appl. Phys. **97**, 121301 (2005). Copyright 2005 American Institute of Physics.

Another key feature of ALD processes is their weak temperature dependency. CVD processes in general are strongly temperature dependent, especially when they are in the surface-reaction-limited regime where the growth rate follows an Arrhenius-type relationship *increasing exponentially* with deposition temperature. In the special case of ALD, the amount deposited per cycle depends on the conditions where the saturating, irreversible reactions are carried out (reactants, substrate, temperature). Several trends how temperature may affect the amount deposited per cycle, referred to here as *growth per cycle* (GPC) and often in ALD literature as *growth rate* (denoting growth in a cycle, not per time unit), have been observed in the literature and are schematically summarized in Figure 4. The temperature dependencies are typically weak: For example in the case of Al_2O_3 deposition from $AlMe_3$ and H_2O, collection of data from many sources leads to the conclusion that in the temperature range $180 - 300\,^{\circ}C$ an $100\,^{\circ}C$ increase in the deposition temperature *decreases linearly* the GPC by about 20%, as opposed to the expected exponentially increasing trend for CVD in general. The explanation behind the decreasing trend is the decrease of the –OH group concentration of the Al_2O_3 surface, as explored in detail elsewhere.[2,41]

Good ALD processes follow the above description closely. When reactants that completely fulfill the ALD criteria are used in reactors optimized to operate in the ALD regime—with Steps 1a–2b tuned so that the reactions are allowed to saturate and the purges are sufficient to prevent Reactants A and B from mixing in the gas phase—the ALD growth should result in the same amount of material deposited per cycle in all reactors, irrespective the process parameter details and the reactor manufacturer. Comprehensive reviews on whether this is really the case are scarce, but, for example, for the $AlMe_3 - H_2O$ process to deposit Al_2O_3 this seems to be valid.[2] In cases where the "correct" ALD behavior is known from the same results obtained by many groups with different reactors, obtaining results that deviate from the known trend points to problems in process optimization. In reality, however, many ALD processes deviate from the general requirements by having some non-ideal features not inherently typical for ALD. For example, the reactants may react partly reversibly, or some irreversible decomposition may take place, or reaction by-products may not be completely inert but they may interact with and adsorb on the surface, blocking further growth. Also the process conditions may be optimized closely, but not completely, to the ALD condition, generally to save time: Saturation of the surface by, e.g., 95% may be accepted, and the unreacted reactant may be purged to, e.g., 0.01% of the original concentration. Elers *et al.*[7] have devoted a complete review article for examining these and other non-ideal factors contributing to film non-uniformity in ALD, and more in-depth discussion on ALD non-idealities is also presented in Ref. 13.

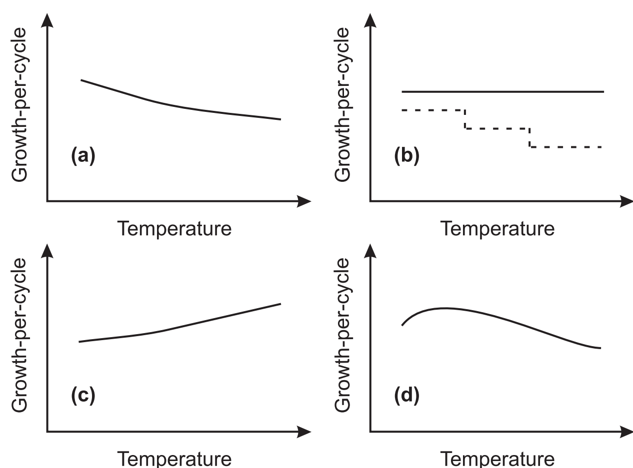

FIG. 4. Schematic illustration how the GPC can vary with the ALD temperature within the so-called *ALD window* (i.e., the temperature range where the process fulfills the criteria of ALD growth): (a) GPC decreases with temperature, (b) GPC is constant with temperature, optionally with different constant values at different temperatures, (c) GPC increases with temperature, and (d) GPC goes through a maximum with temperature. Reprinted with permission from J. Appl. Phys. **97**, 121301 (2005). Copyright 2005 American Institute of Physics.

B. ALD reactants

Reactants to be used in ALD have many requirements. They must be either gaseous or vaporizable at a temperature lower than the ALD reaction temperature to enable transportation through the gas phase. They must react, preferably fast and aggressively, in a saturating, irreversible manner with sites on the surface of the growth substrate. When gaseous reaction by-products are formed, they should be inert and not interfere with the ALD growth. The reactants should not dissolve in the film. The reactants must not decompose thermally, neither during the storage, at the vaporization temperature nor at the ALD growth temperature. They should be safe and easy to handle, preferably non-toxic and non-corrosive towards the substrate and the reactor materials. For industrial use, price is also an issue. High purity is needed for some applications. While it in principle does not matter whether the reactants are gases, liquids, or solids at room temperature, the phase affects the reactor construction: Solids and low-vapor-pressure liquids require special source designs. The list of preferred properties is long, and it is evident that not many reactants in actual use fulfill all the criteria. Compromises often need to be made regarding, for example, the reactivity and corrosiveness of the by-products.

Many classes of compounds are used as metal reactants in ALD. Figure 5 summarizes the most typical ones (please note that this is an introductory view—a more complete treatment can be found in the Table I and Figure 7). The metal reactants used in ALD can be roughly divided into two groups, inorganic and metal organic, and these can be further categorized into elements, halides, alkyls, cyclopentadienyls, alkoxides, β-diketonates, amides and imides, phosphines, silyls, and amidinates. Occasionally, other types of reactants, e.g., heterocyclic compounds, have been used (generally referred to as other compounds in Figure 5), and active reactant development continues to take place. Each type of reactant has its benefits and drawbacks regarding reactivity, stability, gaseous by-products, and impurities left in the films. For example, alkyls, which are organometallic reactants containing a direct metal–carbon bond, are generally very reactive, but stable alkyls are not available for

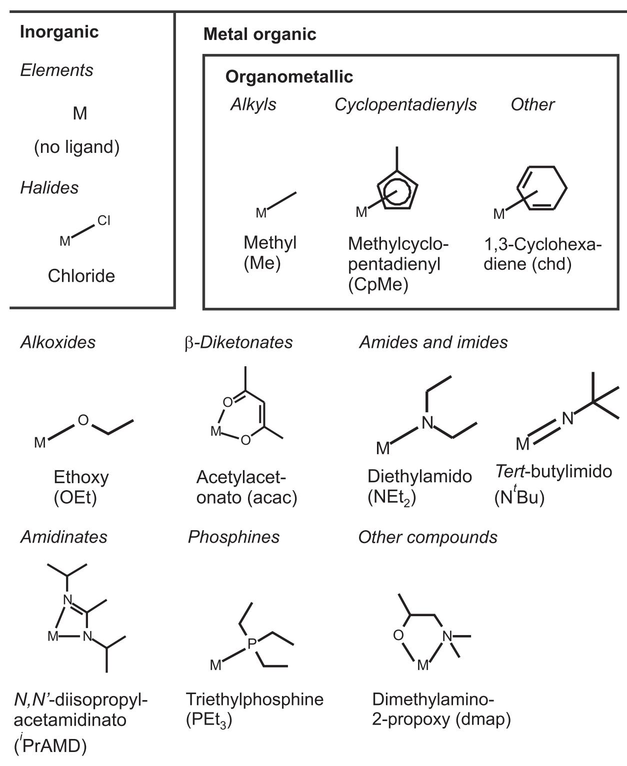

FIG. 5. Overview of the classes of metal reactants used in ALD.

many metals and the deposition temperatures are limited because of the decomposition of the reactants. Chlorides, which belong to the general class of halides, are reactive, stable at a broad temperature range and available for many metals, but the deposited films may suffer from chlorine residues and film thickness gradients in the direction of flow, formed by secondary reactions of the HCl released in the reactions.

The variety in non-metal reactants is somewhat less than in metal reactants. The most typically used non-metal reactants are shown in Figure 6. (Again, a more complete list can be found in Table I.) The most commonly used types of non-metal reactants in ALD are the hydrides of the non-metal elements: water (H_2O), ammonia (NH_3), and hydrogen sulphide (H_2S), etc., which are used to grow oxides, nitrides, and sulphides, respectively. The advantage of these types of reactants is their generally high stability and reactivity in a broad temperature range, including high temperatures. Ozone is often used for deposition of oxides especially from compounds having bulky ligands that are not reactive with water. The drawback of ozone is that it can oxidize also the surface of the underlying substrate. Ozone is also unstable, and its decomposition may be catalysed by the film itself. In such cases limitations arise for uniformity across large wafers and batches, and for conformality in high aspect ratio 3D structures. With plasma-enhanced ALD (PEALD), molecular elements which otherwise would not be sufficiently

TABLE I. Overview of ALD processes based on two reactants (source: ISI Web of Science, status end December 2010).

Z	Material	Reactant A[a]	Reactant B	References
3	Lithium			
	Li_2O (LiOH)	Li(O^{t}Bu)	H_2O	44
	Li_2CO_3	Li(thd)	O_3	45
5	Boron			
	B_2O_3	BBr_3	H_2O	46 and 47
	BN	BCl_3	NH_3	48
		BBr_3	NH_3	49 and 50
		BBr_3	NH_3[n]	50
	$B_xP_yO_z$	$B(OMe)_3$	$POCl_3$	51 and 52
6	Carbon			
	C[b]	CF_x	H[c]	53
		CCl_3	H[c]	54
12	Magnesium			
	MgO	$MgCp_2$	H_2O	55–62
		$Mg(CpEt)_2$	H_2O	63
		$Mg(thd)_2$	H_2O_2	64
		$Mg(thd)_2$	O_3	65
	MgF_2	$Mg(thd)_2$	TiF_4	66 and 67
		$Mg(thd)_2$	TaF_5	68
	MgTe	Mg	Te	69 and 70
13	Aluminum			
	Al	$AlMe_3$	H_2[e]	71 and 72
	Al_2O_3	$AlCl_3$	H_2O	73–98
		$AlCl_3$	H_2O + cat.	99
		$AlCl_3$	O_2	100 and 101
		$AlCl_3$	ROH[d]	80, 88, and 102

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$AlCl_3$	$Al(OEt)_3$	103
		$AlCl_3$	$Al(O^iPr)_3$	103 and 104
		$AlBr_3$	H_2O	105
		$AlMe_3$	H_2O	41, 59, 86, 94, 95, 98, and 106–396
		$AlMe_3$	H_2O[n]	397 and 398
		$AlMe_3$	H_2O_2	325 and 399–407
		$AlMe_3$	O_3	152, 160, 161, 186, 199, 216, 236, 267, 270, 333, 380, 381, and 408–428
		$AlMe_3$	O_2[e]	199, 242, 310, 336, 354, 357, 372, 375, 426, and 429–456
		$AlMe_3$	$O_2 + H_2O + H_2O_2$	457
		$AlMe_3$	CO_2[e]	458
		$AlMe_3$	N_2O	402
		$AlMe_3$	NO_2	110
		$AlMe_3$	N_2O_4	459
		$AlMe_3$	^iPrOH	460 and 461
		$AlMe_3$	^nPrOH	281
		$AlMe_3$	$Al(O^iPr)_3$	103
		$AlMe_2Cl$	H_2O	462 and 463
		$AlMe_2O^iPr$	H_2O	464 and 465
		$AlMe_2O^iPr$	O_2[e]	435
		$AlEt_3$	H_2O	56
		$Al(OEt)_3$	H_2O	80
		$Al(OEt)_3$	O_2	80
		$Al(O^nPr)_3$	H_2O	80 and 88
		$Al(O^nPr)_3$	O_2	80
		$Al(mmp)_3$	H_2O	466
		$AlH_3N:(C_5H_11)$	O_2[e]	467
		$Al(NEt_2)_3$	H_2O	468–471
		$Al(NEt_2)_3$	O_3	472–475
		$Al(N^iPr_2)_3$	H_2O	470
		$Al(^iPrAMD)Et_2$	H_2O	476
	AlN	$AlCl_3$	NH_3	477 and 478
		$AlCl_3$	$NH_3 + H_2$[e]	479 and 480
		$AlMe_3$	NH_3	125, 132, 338, 478, and 481–490
		$AlMe_3$	NH_3[n]	491
		$AlMe_3$	$NH_3 + N_2$[e]	492
		$AlMe_3$	$NH_3 + H_2$[e]	493
		$AlMe_3$	$N_2 + H_2$[e]	494
		$AlEt_3$	NH_3	495 and 496
		$Me_3N:AlH_3$	NH_3	497
		$Me_2EtN:AlH_3$	NH_3	498–500
		$Al(NMe_2)_3$	NH_3	501
	AlP	$AlMe_2H$	PH_3	502–504
		$Me_2EtN:AlH_3$	PH_3	505
	AlAs	$AlCl_3$	AsH_3	506 and 507
		$AlMe_3$	AsH_3	508–518
		$AlMe_2H$	AsH_3	519–523
		$AlEt_3$	AsH_3	512 and 514
		Al^iBu_3	AsH_3	524
		$Me_3N:AlH_3$	AsH_3	525
		$Me_3N:AlH_3$	$As(NMe_2)_3$	526 and 527
		$Me_2EtN:AlH_3$	AsH_3	523 and 528–533
	AlSb	$AlCl_3$	$(Et_3Si)_3Sb$	534
	$Al_xSi_yO_z$	$AlCl_3$	$Si(OEt)_4$	535
	Al_2O_3/ SiO_2[f]	$AlMe_3$	$(^tBuO)_3SiOH$	43, 536, and 537
	$Al_xTi_yO_z$	$AlCl_3$	$Ti(OEt)_4$	103
		$AlCl_3$	$Ti(O^iPr)_4$	103
		$AlMe_3$	$Ti(O^iPr)_4$	103
	$Al_xCr_yO_z$	$AlMe_3$	CrO_2Cl_2	538

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
	$Al_xZr_yO_z$	$Al(OEt)_3$	$ZrCl_4$	103
	$Al_xHf_yO_z$	$Al(OEt)_3$	$HfCl_4$	103
	$AlLaO_3$	$LaAl(O^iPr)_6(^iPrOH)]_2$	H_2O	539
	$AlPr_xO_y$	$AlPr(O^iPr)_6(Pr^iOH)]_2$	H_2O	540
	$AlNd_xO_y$	$AlNd(O^iPr)_6(Pr^iOH)]_2$	H_2O	540
14	Silicon			
	Si	$SiCl_4$	Si_2H_6	541
		$SiCl_2H_2$	H_2	542 and 543
		$SiCl_2H_2$	H[c]	544–551
		$SiCl_2H_2$	…[j]	552–554
		$SiEt_2H_2$	…[k]	555
		SiH_4	…[k]	556–558
		Si_2Cl_6	$Si_2 H_6$	547, 548, 559, and 560
		Si_2Cl_6	H[c]	561
		Si_2H_6	…[k]	562–570
		Si_2H_6	…[j]	571–575
		Si_2H_6	…[k]	570 and 576
		Si_2H_6	…[l]	577
		Si_3H_8	…[k]	546 and 578
	SiO_2	$SiCl_4$	H_2O	74, 111, 130, and 579–587
		$SiCl_4$	H_2O + cat.[g]	99, 584, and 588–594
		Si_2Cl_6	O_3	595
		$SiCl_3H$	H_2O	596–598
		$SiCl_2H_2$	O_3	599 and 600
		SiH_4	N_2O[e]	601
		$Si(OMe)_4$	H_2O	602
		$Si(OMe)_4$	$H_2O + NH_3$ cat.	603
		$Si(OEt)_4$	H_2O + cat.	604 and 605
		$Si(OEt)_4$	O_2[e]	433, 606, and 607
		$Si(OEt)_3((CH_2)_3NH_2)$	$H_2O + O_3$	362 and 608–612
		$Si(O^tPe)_3OH$	$AlMe_3$	613 and 614
		HMDS[h]	O_2	615
		$SiH_2(N(CH_3)_2)_2$	O_3	616 and 617
		$SiH_2(NH\ ^tBu)_2$	O_2[e]	618
		$SiH_2(NEt_2)_2$	O_2[e]	619
		$SiH_2(NEt_2)_2$	O_3	389
		$SiH(N(CH_3)_2)_3$	O_3	616, 617, and 620–622
		$SiH(N(CH_3)_2)_3$	H_2O	623
		$SiH(N(CH_3)_2)_3$	H_2O_2	623 and 624
		$Si(NCO)_4$	H_2O	625
		$Si(NCO)_4$	NEt_3	626
		$MeOSi(NCO)_3$	H_2O_2	627 and 628
	Si_3N_4	$SiCl_4$	NH_3	629–633
		$SiCl_2H_2$	NH_3	632 and 634
		$SiCl_2H_2$	NH_3[e]	635
		$SiCl_2H_2$	NH_3 + cat.	636 and 637
		Si_2Cl_6	$N_2 H_4$	638
		Si_2Cl_6	NH_3	639
	SiC	$SiCl_2H_2$	$C_2 H_4$	640 and 641
		$SiEt_2H_2$	…[i]	642
		Si_2H_6	$C_2 H_4$	643
		Si_2H_6	$C_2 H_2$	644–647
	$Si_xAl_yO_z$	$Si(OEt)_4$	$AlCl_3$	535
	SiO_2/ Al_2O_3[f]	$(^tBuO)_3SiOH$	$AlMe_3$	43, 536, and 537
	$Si_xTi_yO_z$	$Si(OEt)_4$	$TiCl_4$	51
		$Si(O^nBu)_4$	$Ti(NEt_{2)4}$	648 and 649
	$Si_xZr_yO_z$	$SiCl_4$	$Zr(O^tBu)_4$	650
		$Si(OEt)_4$	$ZrCl_4$	103
		$Si(O^nBu)_4$	$ZrCl_4$	103 and 651

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$Si(O^nBu)_4$	$Zr(NEt_2)_4$	652
	$Si_xHf_yO_z$	$Si(OEt)_4$	$HfCl_4$	653
		$Si(OEt)_4$	HfI_4	653
		$(^tBuO)_3SiOH$	$Hf(NMe_2)_4$	654
		$Si(O^nBu)_4$	$Hf(NEt_2)_4$	655–659
		$Si(O^tPe)_3OH$	$Hf(NEt_2)_4$	660
		$Si(O^nBu)_4$	$Hf(NEtMe)_4$	661
	$Si_xBi_yO_z$	$Bi(CH_2SiMe_3)_3$	O_3	662 and 663
15	Phosphorus			
	PO_x	PCl_3	H_2O	664–668
		$POCl_3$	H_2O	581, 669, and 670
	$P_xB_yO_z$	$POCl_3$	$B(OMe)_3$	51 and 52
20	Calcium			
	CaO ($CaCO_3$)	$Ca(thd)_2$	H_2O	671
		$Ca(thd)_2$	O_3	672 and 673
		$Ca(^iPr_3Cp)_2$	H_2O	674
	CaS	$Ca(thd)_2$	H_2S	671 and 675–679
	CaF_2	$Ca(thd)_2$	HF	680
		$Ca(thd)_2$	TiF_4	67 and 681
21	Scandium			
	Sc_2O_3	$ScCp_3$	H_2O	682
		$Sc(thd)_3$	O_3	682
		$Sc(thd)_3$	$O_3 + H_2O_2$	682
		$Sc(^iPrAMD)_3$	H_2O	683
		$Sc(emd)_3$	O_2	684
22	Titanium			
	Ti	$TiCl_4$	H_2[e]	685 and 686
	TiO_2	TiF_4	H_2O	687
		$TiCl_4$	H_2O	44, 92, 94, 123, 128, 153, 176, 179, 198, 207, 218, 228, 238, 305, 365, 378, 379, 390, 581, 585, 664, 665, and 688–814
		$TiCl_4$	H_2O_2	301, 334, 403, 405, 490, 815, and 816
		$TiCl_4$	MeOH	817
		$TiCl_4$	O_2[e]	818 and 819
		TiI_4	H_2O	820–822
		TiI_4	H_2O_2	823–825
		TiI_4	O_2	826 and 827
		$Ti(CpMe_5)(OMe)_3$	O_3	828 and 829
		$Ti(CpMe_5)(OMe)_3$	O_2[e]	375
		$Ti(CpMe)(O^iPr)_3$	O_2[e]	375
		$Ti(OMe)_4$	H_2O	95 and 830–838
		$Ti(OEt)_4$	H_2O	122, 128, 760, 773, 782, 837, and 839–845
		$Ti(OEt)_4$	H_2O_2	96, 760, 773, and 782
		$Ti(O^iPr)_4$	H_2O	128, 212, 236, 273, 280, 285, 325, 328, 350, 359, 362, 396, 764, 788, 791, and 846–881
		$Ti(O^iPr)_4$	H_2O[e]	864 and 878
		$Ti(O^iPr)_4$	H_2O_2	325, 334, 406, 407, 848, 882, and 883
		$Ti(O^iPr)_4$	O_2	736 and 884
		$Ti(O^iPr)_4$	O_2[e]	375, 439, 442, 448, 607, 854, 864, 878, and 885–895
		$Ti(O^iPr)_4$	$O_2 + NH_3$	896
		$Ti(O^iPr)_4$	NH_3	897 and 898
		$Ti(O^iPr)_4$	O_3	236, 890, and 899–906
		$Ti(O^iPr)_4$	N_2O[e]	890 and 907

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$Ti(O^iPr)_4$	HCOOH	908
		$Ti(O^iPr)_4$	CH_3 COOH	908–911
		$Ti(O^iPr)_2(dmae)_2$	H_2O	912
		$Ti(O^iPr)_2(thd)_2$	H_2O	913–916
		$Ti(trhd)_2(O(CMe_2Et)_2$	H_2O	913
		$Ti(OBu)_4$	H_2O	917
		$Ti(NMe_2)_4$	H_2O	356, 377, 864, 914, and 918–924
		$Ti(NMe_2)_4$	O_3	418 and 925
		$Ti(NMe_2)_4$	H_2O[e]	864
		$Ti(NMe_2)_4$	O_2[e]	864 and 926–930
		$Ti(NMe_2)_4$	[q]	931
		$TiCp_2((^iPrN)_2C(NH^iPr))$	[q]	932
	TiN	$TiCl_4$	NH_3	766 and 933–956
		$TiCl_4$	NH_3 + cat.	121, 934–936, 957
		$TiCl_4$	N_2, H_2[e]	429, 449, 453, and 958–965
		$TiCl_4$	Me_2NNH_2	966
		$TiCl_4$	$^tBuNH_2$	967
		$TiCl_4$	$AyNH_2$	967
		TiI_4	NH_3	936 and 968
		TiI_4	$^tBuNH_2$	967
		TiI_4	$AyNH_2$	967
		$Ti(NMe_2)_4$	NH_3	319 and 969–987
		$Ti(NMe_2)_4$	NH_3[e]	986, 988, and 989
		$Ti(NMe_2)_4$	NH_3, H_2[e]	493, 979, and 990
		$Ti(NMe_2)_4$	N_2[e]	986, 989, and 991–994
		$Ti(NMe_2)_4$	H_2[e]	989, 992, 993, and 995
		$Ti(NMe_2)_4$	[q]	931
		$Ti(NEt_2)_4$	NH_3	977
		$Ti(NEtMe)_4$	NH_3	996 and 997
		$Ti(NEtMe)_4$	[q]	998
		$TiCp_2((^iPrN)_2C(NH^iPr))$	[q]	932
	TiS_2	$TiCl_4$	H_2S	999
	$Ti_xAl_yO_z$	$Ti(OEt)_4$	$AlCl_3$	103
		$Ti(O^iPr)_4$	$AlCl_3$	103
		$Ti(O^iPr)_4$	$AlMe_3$	103
	$Ti_xSi_yO_z$	$TiCl_4$	$Si(OEt)_4$	51
		$Ti(NEt_2)_4$	$Si(O^nBu)_4$	648 and 649
	$Ti_xZr_yO_z$	$Ti(O^iPr)_4$	$ZrCl_4$	103, 1000, and 1001
	$Ti_xHf_yO_z$	$Ti(O^iPr)_4$	$HfCl_4$	103
23	Vanadium			
	VO_x	$VOCl_3$	H_2O	581, 668, 698, 705, and 1002–1005
		$VO(O^iPr)_3$	H_2O	1006–1010
		$VO(O^iPr)_3$	H_2O[e]	1010
		$VO(O^iPr)_3$	H_2O_2	1011
		$VO(O^iPr)_3$	O_2	1012–1014
		$VO(O^iPr)_3$	O_2[e]	1010
		$VO(O\ ^nPr)_3$	CH_3 COOH	910, 1015, and 1016
		$VO(acac)_2$	O_2	1017–1019
24	Chromium			
	CrO_x	CrO_2Cl_2	H_2O	581, 668, 670, 698, 705, and 1020–1028
		CrO_2Cl_2	H_2O_2	1026
		CrO_2Cl_2	MeOH	538, 817, and 1028–1031
		$Cr(acac)_3$	O_2	736 and 1032–1036
	$Cr_xAl_yO_z$	CrO_2 Cl_2	$AlMe_3$	538

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
25	Manganese			
	MnO_x	$Mn(CpEt)_2$	H_2O	1037
		$Mn(thd)_3$	O_3	1038–1041
	MnS	$MnCl_2$	H_2S	77 and 78
	MnTe	Mn	Te	70 and 1042–1051
	MnAs	$Mn(CpMe)_2$	$As(NMe_2)_3$	1052
26	Iron			
	Fe	$Fe(^tBuAMD)_2$	H_2	1053
	FeO_x	$FeCl_3$	H_2O	1054
		$Fe(acac)_3$	O_2	1055
		$Fe(acac)_3$	O_3	1056
		$Fe(thd)_3$	O_3	1057–1061
		$Fe(Cp)_2$	O_2	821, 1062, and 1063
		$Fe(Cp)_2$	O_3	609, 1064, and 1065
		$Fe(^tBuAMD)_2$	H_2O	1053
		$Fe_2(^tBuO)_6$	H_2O	1064–1066
27	Cobalt			
	Co	$Co(^iPrAMD)_2$	H_2	1053, 1067, and 1068
		$Co(^iPrAMD)_2$	NH_3	1068
		$Co(^iPrAMD)_2$	NH_3[e]	1069
		$CoCp(^iPrAMD)$	NH_3[e]	1070
		$CoCp_2$	NH_3[e]	1071 and 1072
		$CoCp(CO)_2$	NH_3[e]	1073 and 1074
		$CoCp(CO)_2$	H_2[e]	1074 and 1075
		$CoCp(CO)_2$	N_2[e]	1074 and 1075
		$Co_2(CO)_8$	H_2[e]	1075–1077
		$Co_2(CO)_8$	N_2[e]	1075
	CoO_x	CoI_2	O_2	1078
		$Co(acac)_2$	O_2	1079–1081
		$Co(acac)_3$	O_2	1079–1087
		$Co(thd)_2$	O_3	1060 and 1088–1090
		$Co(^iPrAMD)_2$	H_2O	1053
	$CoSi_2$	$CoCp_2$	NH_3, SiH_4[e]	1091
28	Nickel			
	Ni	$Ni(acac)_2$	H_2	1092
		$Ni(^iPrAMD)_2$	H_2	1053
		$Ni(dmamb)_2$	H_2[e]	1093
		$Ni(dmamb)_2$	NH_3[e]	1072 and 1093
		$Ni(hfip)_2$	H_2	1094 and 1095
	NiO	$NiCp_2$	H_2O	1096
		$NiCp_2$	O_3	1097–1100
		$Ni(CpEt)_2$	O_3	1097 and 1100
		$Ni(dmamb)_2$	H_2O	1101
		$Ni(dmamp)_2$	H_2O	1102 and 1103
		$Ni(acac)_2$	O_2	1104–1106
		$Ni(acac)_2$	O_3	1092 and 1107
		$Ni(thd)_2$	H_2O	1108–1110
		$Ni(thd)_2$	O_3	1111
		$Ni(apo)_2$	O_3	1107
		$Ni(dmg)_2$	O_3	1107
29	Copper			
	Cu	CuCl	H_2	1112 and 1113
		CuCl	$H_2 + H_2O$	1113 and 1114
		CuCl	Zn	1115
		$Cu(acac)_2$	H_2	1092
		$Cu(acac)_2$	H_2[e]	1116–1118

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$Cu(thd)_2$	H_2	1119–1122
		$Cu(thd)_2$	H_2[e]	1123
		$Cu(hfac)_2$	ROH[d]	1124
		$Cu(hfac)_2$	HCHO[m]	1124
		$Cu(hfac)_2$	H_2 + cat.	1125
		Cu(hfac)(vtmos)	H_2[e]	1126
		$Cu(^iPrAMD)$	H_2	1053 and 1067
		$Cu(^sBuAMD)$	H_2	1127–1131
		$Cu(dmap)_2$	$ZnEt_2$	1132
		Cu(dki)(vtms)	SiH_2Et_2	1133
	CuO_x	$Cu(acac)_2$	O_2	1134
		$Cu(thd)_2$	O_2	1135–1137
		$Cu(hfac)_2$	H_2O	1138
		$Cu(P^nBu_3)_2(acac)$	H_2O, O_2	1139
	Cu_3N	$Cu(^sBuAMD)$	NH_3	1128 and 1140
		$Cu(hfac)_2$	$NH_3 + H_2O$	1141
	Cu_xS	$Cu(thd)_2$	H_2S	1142–1146
		$Cu(^sBuAMD)_2$	H_2S	1147
	Cu_xSe	$Cu(O_2C\ ^tBu)_2$	$(Et_3\ Si)_2Se$	1148
		CuCl	$(Et_3Si)_2Se$	1148
30	Zinc			
	Zn	$ZnEt_2$	H_2O	1149
	ZnO	Zn	O_2	1150
		Zn	H_2O	1151
		$ZnCl_2$	H_2O	1152
		$ZnCl_2$	O_2	1153–1155
		$ZnMe_2$	H_2O	1156–1162
		$ZnMe_2$	O_2[e]	1163 and 1164
		$ZnMe(O^iPr)$	H_2O	1165
		$ZnEt_2$	H_2O	60, 117, 145, 147, 204, 240, 264, 282, 291, 297, 300, 305, 306, 318, 323, 346, 350, 352, 368, 374, 812, 1011, 1149, 1157, and 1159–1265
		$ZnEt_2$	H_2O[e]	1266
		$ZnEt_2$	H_2O[n]	1179
		$ZnEt_2$	$H_2O + NH_3$	292 and 1267
		$ZnEt_2$	$H_2O + NH_3 + O_2$	457
		$ZnEt_2$	H_2O_2	1223, 1268, and 1269
		$ZnEt_2$	O_2	1186
		$ZnEt_2$	O_2[e]	1270–1275
		$ZnEt_2$	O_3	1193
		$ZnEt_2$	N_2O	1276–1278
		$ZnEt_2$	N_2O[e]	458
		$Zn(OAc)_2$	H_2O	1160 and 1279–1285
	ZnS	Zn	S	76 and 1286–1288
		Zn	H_2S	1289
		$ZnCl_2$	H_2S	77, 78, 84, and 1290–1305
		ZnI_2	H_2S	1305
		$ZnMe_2$	H_2S	1156 and 1306–1314
		$ZnEt_2$	H_2S	206, 1177, 1194, 1215, and 1315–1320
		$ZnEt_2$	Et_2S_2	1321
		$Zn(CH_3COO)_2$	H_2S	1304
		$Zn(OAc)_2$	H_2S	680, 1279, 1292, 1293, 1295–1297, 1304, and 1322
	ZnF	$Zn(OAc)_2$	HF	680
	ZnSe	Zn	Se	1150, 1287, and 1323–1339
		Zn	H_2Se	1340
		Zn	Et_2Se	1341

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$ZnCl_2$	H_2Se	1342–1344
		$ZnCl_2$	$(Et_3Si)_2Se$	1148
		$ZnMe_2$	H_2Se	1312–1314 and 1345–1355
		$ZnEt_2$	H_2Se	1356
		$ZnEt_2$	Et_2Se_2	1321
		$Zn[N(SiMe_3)_2]_2$	H_2Se	1356
	ZnTe	Zn	Te	1044, 1323, 1326–1328, 1330, and 1357–1366
		$ZnCl_2$	$(Et_3Si)_2Te$	1148
		$ZnMe_2$	Et_2Te	1367
		$ZnMe_2$	MeAyTe	1367
31	Gallium			
	Ga_2O_3	$Ga(acac)_3$	O_3	1368 and 1369
		$Ga(acac)_3$	H_2O	1368
		$[GaMe_2NH_2]_3$	O_2[e]	1370–1372
		$Ga_2(NMe_2)_6$	H_2O	1373
	GaN	Ga	N_2	1374 and 1375
		GaCl	NH_3	1376 and 1377
		$GaCl_3$	NH_3	1378 and 1379
		$GaMe_3$	NH_3	485 and 1380–1384
		$GaEt_3$	NH_3	496, 1385, and 1386
		$GaEt_3$	NH_3[o]	1387
	GaP	Ga	P	1286
		GaCl	PH_3	1388
		$GaMe_3$	PH_3	502, 516, and 1389–1394
		$GaMe_3$	$P(NMe_2)_3$	1395
		$GaEt_3$	PH_3[n]	1396
	GaAs	GaCl	AsH_3	1388, 1394, and 1397–1410
		$GaCl_3$	AsH_3	506, 507, and 1411–1415
		$GaCl_3$	As	1414
		GaBr	AsH_3	1416
		GaI	AsH_3	1416
		$GaMe_3$	AsH_3	508–511, 514, 516, 518, 521, 524, 1390, 1393, and 1417–1467
		$GaMe_3$	$EtAsH_2$	1468
		$GaMe_3$	$^tBuAsH_2$	1469–1476
		$GaEt_3$	AsH_3	512, 514, 1390, 1424, 1433, 1438, and 1477–1485
		$GaEt_3$	$^tBuAsH_2$	1475 and 1486
		$GaEt_3$	$(Me_2N)_3As$	527, 1487, and 1488
		$GaEt_2Cl$	As	1489
		$GaEt_2Cl$	AsH_3	1450 and 1490
		$GaEt_2Me$	AsH_3	1390
		Ga^iBu_3	AsH_3	1480
		$GaNp_3$	$^tBuAsH_2$	1475 and 1491
	GaSb	$GaCl_3$	$(Et_3Si)_3Sb$	534
32	Germanium			
	Ge	$GeCl_4$	H[c]	1492 and 1493
		$GeMe_2H_2$	H[c]	1494–1496
		$GeEt_2H_2$	…[k]	1497 and 1498
		GeH_4	…[k]	558 and 1499
		Ge_2H_6	…[k]	1500 and 1501
	GeO_2	$GeCl_4$	H_2O	689 and 1502
		Ge(dpp-BIAN)	O_3	1503
	GeSb	$GeCl_2 \cdot (C_4H_8O_2)$	$(Et_3Si)_3Sb$	534
	GeTe	$GeCl_2 \cdot (C_4H_8O_2)$	$(Et_3Si)_2Te$	1148 and 1504

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
38	Strontium			
	SrO ($SrCO_3$)	$Sr(Cp^iPr_3)_2$	H_2O	1505 and 1506
		$Sr(Cp^nPrMe_3)_2$	H_2O	1507
		$Sr(Cp^nPrMe_3)_2$	O_2	1507
		$Sr(Cp^tBu_3)_2$	H_2O	838
		$Sr(thd)_2$	O_3	1508
		$Sr(methd)_2$	O_2[e]	885
	SrS	$Sr(CpMe_5)_2$	H_2S	1509
		$Sr(Cp^iPr_3)_2$	H_2S	1509 and 1510
		$Sr(thd)_2$	H_2S	675, 677, and 1511–1513
	SrF_2	$Sr(thd)_2$	HF	680
	$SrTa_2O_6$	$Sr[Ta(OEt)_5(me)]_2$	O_2[e]	1514
39	Yttrium			
	Y_2O_3	YCp_3	H_2O	1515 and 1516
		$Y(CpMe)_3$	H_2O	1515 and 1517–1520
		$Y(CpMe)_3$	O_3	1521
		$Y(CpEt)_3$	H_2O	1522 and 1523
		$Y(thd)_3$	O_2	1524
		$Y(thd)_3$	O_3	135, 153, and 1524–1526
		$Y(thd)_3$	O_2[e]	1527–1532
		$Y(^iPrAMD)_3$	H_2O	1533
	YF_3	$Y(thd)_3$	TiF_4	1534
	Y_2O_2S	$Y(thd)_3$	H_2S	1535
40	Zirconium			
	ZrO_2	$ZrCl_4$	H_2O	98, 128, 135, 138, 146, 162, 221, 736, 1516, and 1536–1563
		$ZrCl_4$	H_2O_2	1544 and 1545
		$ZrCl_4$	O_2	1564 and 1565
		ZrI_4	H_2O	1566
		ZrI_4	H_2O_2	1567–1570
		$ZrCp_2Cl_2$	O_3	1571–1573
		$ZrCp_2Me_2$	H_2O	1574 and 1575
		$ZrCp_2Me_2$	O_3	1571 and 1576
		$ZrCp_2Me(OMe)$	O_3	1577
		$ZrCp(NMe_2)_3$	O_3	1578
		$Zr(CpMe)_2Me_2$	O_3	1579–1581
		$Zr(CpMe)_2Me(OMe)$	H_2O	1582
		$Zr(CpMe)_2Me(OMe)$	O_3	1579–1584
		$Zr(CpMe)(NMe_2)_3$	O_3	1578
		$Zr(CpEt)(NMe_2)_3$	O_3	1578
		$Zr(Cp_2CMe_2)Me_2$	O_3	1585
		$Zr(Cp_2CMe_2)Me(OMe)$	O_3	1585
		$Zr(O^iPr)_4$	H_2O	1586
		$Zr(O^iPr)_2(dmae)_2$	H_2O	861 and 1587
		$Zr(O^tBu)_4$	H_2O	273, 850, 852, 853, 879, and 1588–1594
		$Zr(O^tBu)_4$	O_2	1592
		$Zr(O^tBu)_4$	O_2[e]	1595–1598
		$Zr(O^tBu)_4$	N_2O	1592
		$Zr(O^tBu)_4$	Ar[e]	1598
		$Zr(O^tBu)_2(dmae)_2$	H_2O	1587 and 1599
		$Zr(O^tBu)_2(dmae)_2$	H_2[e]	1600
		$Zr(dmae)_4$	H_2O	1587
		$Zr(thd)_4$	O_3[e]	1571
		$Zr(NMe_2)_4$	H_2O	350, 1517–1519, and 1601–1605
		$Zr(NEt_2)_4$	H_2O	295, 311, 315, 320, 1601, and 1606
		$Zr(NEt_2)_4$	O_2	1607
		$Zr(NEt_2)_4$	O_2[e]	1596 and 1608

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$Zr(NEtMe)_4$	H_2O	1601 and 1609
		$Zr(NEtMe)_4$	O_3	1610–1615
		$Zr(NEtMe)_4$	O_2[e]	1616–1619
		$Zr(NEtMe)_4$	N_2O[e]	1618
		$Zr(NEtMe)_4$	[q]	998
		$Zr[N(SiMe_3)_2]_2Cl_2$	H_2O	1620
		$Zr(MeAMD)_4$	H_2O	1609
	ZrN_x	$ZrCp_2(NMe_2)_2$	N_2[e]	1621
		$Zr(NMe_2)_4$	NH_3	1622
		$Zr(NEtMe)_4$	NH_3	1622
		$Zr(NEt_2)_4$	NH_3	1622
		$Zr(NEt_2)_4$	N_2[e]	1623
	$Zr_xAl_yO_z$	$ZrCl_4$	$Al(OEt)_3$	103
	$Zr_xSi_yO_z$	$ZrCl_4$	$Si(OEt)_4$	103 and 1624
		$ZrCl_4$	$Si(O^nBu)_4$	103 and 651
		$Zr(O^tBu)_4$	$SiCl_4$	650
		$Zr(NEt_2)_4$	$Si(O^nBu)_4$	652
	$Zr_xTi_yO_z$	$ZrCl_4$	$Ti(O^iPr)_4$	103, 1000, and 1001
	$Zr_xLa_yO_z$	$Zr(CpMe)_2Me(OMe)$	$La(^iPrCp)_3$	1625
41	Niobium			
	Nb_2O_5	$Nb(OEt)_5$	H_2O	91, 122, 391, 842, 1539, and 1626–1629
	NbN	$NbCl_5$	NH_3	933, 935, and 1630–1632
		$NbCl_5$	NH_3 + cat.	935 and 1630
		$NbCl_5$	Me_2NNH_2	966
		$Nb(N^tBu)(NEtMe)_3$	H_2[e]	1633
42	Molybdenum			
	Mo	$MoCl_5$	Zn	1634
	Mo_xN	$MoCl_5$	NH_3	933 and 1635
		$MoCl_5$	Me_2NNH_2	966
		$Mo(N^tBu)_2(NMe_2)_2$	NH_3	1636–1640
		$Mo(N^tBu)_2(NEt_2)_2$	NH_3	1638 and 1640
44	Ruthenium			
	Ru	Ru(chd)(ipmp)	O_2	1641
		$RuCp_2$	O_2	95, 98, and 1642–1651
		$RuCp_2$	NH_3[e]	1649
		$Ru(CpEt)_2$	O_2	1649 and 1652–1659
		$Ru(CpEt)_2$	NH_3[e]	196, 1649, 1656, and 1660–1670
		$Ru(CpEt)_2$	$N_2 + H_2$[e]	994 and 1671
		$Ru(CpEt)_2$	H_2[e] + O_2	900
		Ru(CpEt)(dmp)	O_2	906 and 1672–1677
		Ru(CpEt)(dmp)	[q]	1678
		Ru(CpEt)(pyr)	NH_3[e]	1679
		$RuCp(CpCH(Me)(NMe_2))$	O_2	1680
		$RuCp(CO)_2Et$	O_2	1681 and 1682
		$RuCp(CO)_2Et$	O_2[e]	1681
		$Ru(od)_3$	O_2	1683
		$Ru(thd)_3$	H_2	1684
		$Ru(thd)_3$	O_2	1644 and 1685–1687
		$Ru(^tBuAMD)_2(CO)_2$	O_2	1688
		$Ru(^tBuAMD)_2(CO)_2$	NH_3	1689 and 1690
		RuO_4	H_2	1691
	RuO_2	$Ru(CpEt)_2$	O_2	1652, 1654, 1687, and 1692
		$Ru(thd)_2(cod)$	O_2	1693
45	Rhodium			
	Rh	$Rh(acac)_3$	O_2	1653 and 1694

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
	Rh_2O_3	$Rh(acac)_3$	O_3	1695
46	Palladium			
	Pd	$Pd(thd)_2$	H_2	1684
		$Pd(thd)_2$	O_2	1136
		$Pd(hfac)_2$	H_2	1696–1698
		$Pd(hfac)_2$	H_2[e]	1699 and 1700
		$Pd(hfac)_2$	$H_2 + N_2$[e]	1701
		$Pd(hfac)_2$	HCOCOOH	1696
		$Pd(hfac)_2$	HCHO[m]	368, 396, 1011, and 1702–1704
47	Silver			
	Ag	$Ag(O_2C^tBu)(PEt_3)$	H_2[e]	1705
		Ag(hfac)(cod)	propanol	1706
48	Cadmium			
	CdS	Cd	S	1707 and 1708
		$CdCl_2$	H_2S	1300
		$CdMe_2$	H_2S	1315 and 1709–1714
	CdSe	Cd	Se	1287, 1708, and 1715–1722
		$CdMe_2$	H_2Se	1315 and 1346
	CdTe	Cd	Te	70, 1042–1047, 1049–1051, 1338, 1360, 1364, 1366, 1708, 1720, 1721, and 1723–1747
		$CdMe_2$	Et_2 Te	1367
		$CdMe_2$	MeAyTe	1367 and 1748–1750
		$CdMe_2$	$^iPr_2Te$	1751 and 1752
49	Indium			
	In_2O_3	$InCl_3$	H_2O	128 and 1753–1757
		$InCl_3$	H_2O_2	1757
		$InMe_3$	H_2O	1758
		InCp	O_3	787, 1759, and 1760
		$In(acac)_3$	H_2O	1761
		$In(acac)_3$	O_3	1761
	InN	In	N_2	1762
		$InEtMe_2$	NH_3	485, 1381, and 1382
	In_2S_3	$InCl_3$	H_2S	1763 and 1764
		$In(acac)_3$	H_2S	1177, 1178, and 1765–1772
	InP	InCl	PH_3	1388
		InCl	$^tBuPH_2$	1406
		$InMe_3$	PH_3	1436, 1449, and 1773–1781
		$InMe_3$	$^tBuPH_2$	1782–1784
		$InEt_3$	PH_3	1389 and 1391
		$InEt_3$	$^tBuPH_2$	1785
	In_2Se_3	$InCl_3$	$(Et_3Si)_2Se$	1148
	InAs	InCl	AsH_3	1398, 1399, and 1411
		$InMe_3$	AsH_3	1394, 1428, 1786, and 1787
		$InMe_3$	$^tBuAsH_2$	1476, 1775, 1777, 1788, and 1789
		$InClMe_2$	AsH_3	1790
		$InEt_3$	AsH_3	1421, 1791, and 1792
	InSb	In	Sb	1793 and 1794
50	Tin			
	SnO_2	Sn	O_2	1286
		$SnCl_4$	H_2O	128, 714, 747, 1369, and 1795–1812
		$SnCl_4$	H_2O_2	1804 and 1813–1818
		SnI_4	H_2O_2	1804
		SnI_4	O_2	1804, 1813, 1814, and 1819–1821
		$SnMe_4$	N_2O_4	459 and 883
		$SnEt_4$	N_2O_4	459

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$Sn^{n}Bu_2(CH_3COO)_2$	O_2[e]	1822–1825
		$Sn(NMe_2)_4$	H_2O_2	1760, 1826, and 1827
		Sn(tbba)	H_2O_2	1828
	SnS	$Sn(acac)_2$	H_2S	384
51	Antimony			
	Sb	$SbCl_5$	$(Et_3Si)_3Sb$	534
	Sb_2O_5	$SbCl_5$	H_2O	1797
		$Sb(NMe_2)_3$	O_3	1829
	Sb_2S_3	$Sb(NMe_2)_3$	H_2S	1226 and 1829
	Sb_2Te_3	$SbCl_3$	$(Et_3Si)_2Te$	534, 1148, and 1504
56	Barium			
	BaO ($Ba(OH)_2$)	$Ba(Cp^{t}Bu_3)_2$	H_2O	1830
	BaO	$Ba(Cp^{n}PrMe_4)_2$	H_2O	1519
	BaS	$Ba(CpMe_5)_2$	H_2S	1509 and 1510
		$Ba(thd)_2$	H_2S	675 and 1831
	BaB_2O_4	$Ba(Tp^{Et2})_2$	H_2O	1832
57	Lanthanum			
	La_2O_3	$La(thd)_3$	O_2	1135 and 1137
		$La(thd)_3$	O_3	44, 1088, 1111, 1577, and 1833–1835
		$La(thd)_3$	H_2O	237, 1836, and 1837
		$La[N(SiMe_3)_2]_3$	H_2O	654 and 1838–1842
		$La(^{i}PrAMD)_3$	H_2O	1053 and 1843
		$La(^{i}PrfAMD)_3$	H_2O	1844
		$La(^{i}PrfAMD)_3$	O_2	1607
		$La(^{i}PrfAMD)_3$	O_3	1844
		$La(Cp)_3$	H_2O	1835, 1845, and 1846
		$La(CpEt)_3$	O_2[e]	1847
		$La(CpEt)_3$	O_3[e]	1848
		$La(Cp^{i}Pr)_3$	H_2O	1849 and 1850
		$La(Cp^{i}Pr)_3$	O_3	1851 and 1852
		$La(Cp^{i}Pr)_3$	O_2[e]	1850 and 1853–1856
	La_2S_3	$La(thd)_3$	H_2S	1857
	LaF_3	$La(thd)_3$	TiF_4	67 and 1858
	$La_xSi_yO_x$	$La[N(SiMe_3)_2]_3$	$(^{t}BuO)_3Si$	654
	$LaAlO_3$	$LaAl(O^{i}Pr)_6(^{i}PrOH)]_2$	H_2O	539
	$La_xZr_yO_z$	$La(Cp^{i}Pr)_3$	$Zr(CpMe)_2$ Me(OMe)	1625
58	Cerium			
	CeO_2	$Ce(thd)_4$	O_2	1135 and 1137
		$Ce(thd)_4$	O_3	1859 and 1860
		$Ce(thd)_3phen$	O_3	1859
59	Praseodymium			
	PrO_x	$Pr[N(SiMe_3)_2]_3$	H_2O	1861 and 1862
		$Pr(^{i}PrAMD)_3$	H_2O	1863
		$Pr(Cp^{i}Pr)_3$	H_2O	1863
	$PrAl_xO_y$	$[PrAl(O^{i}Pr)_6(Pr^{i}OH)]_2$	H_2O	540
60	Neodymium			
	Nd_2O_3	$Nd(thd)_3$	O_3	1864 and 1865
	$NdAl_xO_y$	$NdAl(O^{i}Pr)_6(Pr^{i}OH)]_2$	H_2O	540
62	Samarium			
	Sm_2O_3	$Sm(thd)_3$	O_3	1864
63	Europium			
	Eu_2O_3	$Eu(thd)_3$	O_3	1864

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
64	Gadolinium			
	Gd_2O_3	$Gd(thd)_3$	O_3	1583, 1864, and 1866
		$Gd(CpMe)_3$	H_2O	1866
		$Gd(mmp)_3$-tetraglyme	H_2O	1867
		$Gd(dmb)_3$	H_2O	96 and 1868–1870
		$Gd((^iPrN)_2CNMe_2)_3$	H_2O	1871–1873
66	Dysprosium			
	Dy_2O_3	$Dy(thd)_3$	O_3	1864
		$Dy(emd)_3$	O_2	684
67	Holmium			
	Ho_2O_3	$Ho(thd)_3$	O_3	1864
68	Erbium			
	Er_2O_3	$Er(thd)_3$	O_3	1584, 1864, and 1874
		$Er(thd)_3$	O_2[e]	1527, 1528, and 1530–1532
		$Er(CpMe)_3$	H_2O	1875
		$Er(^tBuAMD)_3$	O_3	1876
69	Thulium			
	Tm_2O_3	$Tm(thd)_3$	O_3	1864
70	Ytterbium			
	Yb_2O_3	$Yb(thd)_3$	O_3	1877
71	Lutetium			
	Lu_2O_3	$Lu(O^iPr)_3$	H_2O	1878
		$Lu[Cp(SiMe_3)]_2Cl$	H_2O	1879 and 1880
	$LuSi_xO_y$	$Lu[N(SiMe_3)_2]_3$	H_2O	1881 and 1882
		$Lu[N(SiMe_3)_2]_3$	O_3	1881 and 1882
72	Hafnium			
	HfO_2	$HfCl_4$	H_2O	92, 135, 154, 160, 168, 169, 171–173, 175, 188–190, 199, 221, 259, 310, 587, 741, 761, 1520, 1541, 1551, 1554–1562, 1566, 1610, 1839, and 1883–1974
		$HfCl_4$	O_2	1975 and 1976
		$HfCl_4$	O_3	420, 423, 1897, 1917, 1933, 1951, 1954, 1977, and 1978
		$HfCl_4$	$Hf(mmp)_4$	1979
		$HfCl_2[N(SiMe_3)_2]_2$	H_2O	1980
		HfI_4	H_2O	1566, 1896, 1901, 1981, and 1982
		HfI_4	H_2O_2	1981
		HfI_4	O_2	1901 and 1983–1987
		HfI_4	O_2[q]	1988
		$HfCp_2Me_2$	H_2O	1989 and 1990
		$HfCp_2Me_2$	O_3	1990
		$HfCp_2Cl_2$	H_2O	1990
		$HfCp_2Cl_2$	O_3	1990
		$Hf(CpMe)_2Me_2$	H_2O	1579
		$Hf(CpMe)_2Me_2$	O_3	1991
		$Hf(CpMe)_2(OMe)Me$	H_2O	1579
		$Hf(CpMe)_2(OMe)Me$	O_3	1521, 1581, 1860, 1991, and 1992
		$Hf(CpMe)_2(O^iPr)Me$	H_2O	1993
		$Hf(CpMe)_2(mmp)Me$	H_2O	1993
		$Hf(Cp)(NMe_2)_3$	O_3	1994
		$Hf(CpMe)(NMe_2)_3$	O_3	1994
		$Hf(Cp_2CMe_2)Me_2$	O_3	1585

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		$Hf(Cp_2CMe_2)Me(OMe)$	O_3	1585
		$Hf(O^iPr)_4$	O_2	1995
		$Hf(O^tBu)_4$	H_2O	274 and 1996
		$Hf(O^tBu)_4$	O_3	1997–1999
		$Hf(O^tBu)_4$	O_2	2000
		$Hf(O^tBu)_4$	O_2[e]	1597
		$Hf(O^tBu)_4$	HCOOH	908
		$Hf(O^tBu)_4$	CH_3 COOH	908–911
		$Hf(O^tBu)_2(mmp)_2$	H_2O	2001
		$Hf(O^tBu)_2(mmp)_2$	O_2	684
		$Hf(O^tBu)(NEtMe)_3$	O_3	2002 and 2003
		$Hf(mmp)_4$	H_2O	1867 and 2004
		$Hf(mp)_4$	H_2O	2005
		$Hf(mp)_4$	O_2[e]	2006
		$Hf(ONEt_2)_4$	H_2O	2007
		$Hf(NMe_2)_4$	H_2O	254, 312, 350, 358, 362, 654, 1601, 1602, 1605, 1660, 1960, and 2008–2033
		$Hf(NMe_2)_4$	O_3	2009 and 2034–2039
		$Hf(NMe_2)_4$	O_2	2040
		$Hf(NMe_2)_4$	O_2[e]	1667 and 2041–2044
		$Hf(NEt_2)_4$	H_2O	197, 468, 633, 1523, 1601, 1932, 1962, and 2045–2057
		$Hf(NEt_2)_4$	O_2	1522 and 2058
		$Hf(NEt_2)_4$	O_2[e]	440, 2006, and 2058–2069
		$Hf(NEt_2)_4$	O_3	472–475
		$Hf(NEt_2)_4$	N_2O	2070
		$Hf(NEt_2)_4$	N_2O[e]	2062 and 2070
		$Hf(NEtMe)_4$	H_2O	160, 247, 250, 253, 1601, 1842, 1849, 1912, 1967, 2023, and 2071–2095
		$Hf(NEtMe)_4$	O_3	428, 616, 620, 829, 1521, 1610, 1611, 1938, 1994, 1998, and 2095–2108
		$Hf(NEtMe)_4$	O_2[e]	438, 963, 1610, 1618, 1619, 1853, and 2109–2111
		$Hf(NEtMe)_4$	N_2O[e]	1618
		$Hf(NEtMe)_4$	[q]	998
		$Hf[N(SiMe_3)_2]_2Cl_2$	H_2O	2112
		$Hf(NO_3)_4$	H_2O	2113–2116
	Hf_3N_4	$Hf(NMe_2)_4$	NH_3	1622
		$Hf(NEt_2)_4$	NH_3	1622
		$Hf(NEtMe)_4$	NH_3	501 and 1622
	HfN	$Hf(NMe_2)_4$	H_2[e]	2042, 2117, and 2118
		$Hf(NMe_2)_4$	N_2[e]	2119
	$Hf_xAl_yO_z$	$HfCl_4$	$Al(OEt)_3$	103
	$Hf_xSi_yO_z$	$HfCl_4$	$Si(OEt)_4$	653 and 2120
		$HfCl_4$	$Si(O^nBu)_4$	2121 and 2122
		HfI_4	$Si(OEt)_4$	653
		$Hf(O^tBu)_4$	$Si(NEtMe)_4$	2123
		$Hf(NMe_2)_4$	$(^tBuO)_3SiOH$	654
		$Hf(NEtMe)_4$	$Si(O^nBu)_4$	661
		$Hf(NEt_2)_4$	$Si(O^nBu)_4$	655–659
		$Hf(NEt_2)_4$	$Si(O^tPe)_3OH$	660
		$Hf(NO_3)_4$	$(^tBuO)_3SiOH$	2116
	$Hf_xTi_yO_z$	$HfCl_4$	$Ti(O^iPr)_4$	103
73	Tantalum			
	Ta	$TaCl_5$	H_2[e]	685 and 2124
		TaF_5	H_2[e]	2125–2127
		TaF_5	Si_2H_6[p]	2128

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
	Ta_2O_5	$TaCl_5$	H_2O	75, 77, 78, 81, 90, 92, 128, 392, 1291, 1541, 1627, 1883, and 2129–2134
		$TaCl_5$	$Ta(OEt)_5$	103 and 2135
		TaF_5	H_2O	2136
		TaI_5	H_2O_2	2137
		TaI_5	O_2	2138
		$Ta(OEt)_5$	H_2O	85, 91, 122, 123, 128, 201, 803, 833, 842, 1539, 1563, 1627, 1885, and 2139–2146
		$Ta(OEt)_5$	O_2[e]	2143, 2147, and 2148
		$Ta(OEt)_5$	O_2[n]	2149
		$Ta(OEt)_4(dmae)$	O_3	2150
		$Ta(NMe_2)_5$	H_2O	843, 918, and 2151–2154
		$Ta(NMe_2)_5$	O_2[e]	375, 453, 918, 1650, 2152, and 2155
		$Ta(NEt_2)_5$	H_2O	2156
		$Ta(NEt)(NEt_2)_3$	H_2O	2156
		$Ta(N^tBu)(^tBu_2pz)_3$	O_3	2157
		$Ta(N^tBu)(^iPrAMD)_2(NMe_2)$	H_2O	2158
	TaO_xN_y	$Ta(N^iPr)(NEtMe)_2$	$H_2 + N_2 + NH_3$[e]	2159
	TaN_x	$TaCl_5$	NH_3	933 and 2160–2162
		$TaCl_5$	NH_3 + cat.	2160
		$TaCl_5$	Me_2NNH_2	966
		$TaCl_5$	$^tBuNH_2$	2163
		$TaCl_5$	$AyNH_2$	2163
		$TaCl_5$	N_2[e]	2164–2166
		$TaCl_5$	$H_2 + N_2$[e]	2167 and 2168
		$TaCl_5 \cdot SEt_2$	$NH_3 + AlMe_3$	2169
		$TaBr_5$	$^tBuNH_2$	2163
		TaF_5	$H_2 + N_2$[e]	2170
		TaF_5	H_2[e] $+ NH_3$	2126 and 2127
		TaF_5	H_2[e] $+ NH_3$[e]	2127
		$Ta(NMe_2)_5$	NH_3	2152 and 2171–2180
		$Ta(NMe_2)_5$	NH_3[e]	2181
		$Ta(NMe_2)_5$	H_2[e]	964, 2152, 2182, and 2183
		$Ta(NMe_2)_5$	N_2[e]	2181–2183
		$Ta(NMe_2)_5$	N_2, H_2[e]	453
		$Ta(NEtMe)_5$	NH_3	2184
		$Ta(N^iPr)(NEtMe)_3$	$H_2 + N_2$[e]	2185
		$Ta(N^tBu)(NEt_2)_3$	NH_3	2186–2190
		$Ta(N^tBu)(NEt_2)_3$	N_2H_4	2188
		$Ta(N^tBu)(NEt_2)_3$	H_2[e]	1669 and 2191–2193
		$Ta(N^tBu)(NEt_2)_3$	NH_3[e]	1668 and 2186
		$TaCp(N^tBu)(NEt_2)_2$	NH_3	2190
		$Ta(N^tAm)(NMe_2)_3$	H_2[e]	1671
	TaN_xC_y	$Ta(N^tBu)(NEt_2)_3$	H_2	2194
		$Ta(N^tBu)(NEt_2)_3$	H_2[e]	2194–2196
		$Ta(N^tBu)(NEt_2)_3$	H_2-CH_4[e]	2197
		$Ta(N^tBu)(NEt_2)_3$	NH_3	2198
		$Ta(N^tBu)(NEt_2)_3$	NH_3[e]	2198
		$Ta(N^tAm)[(NMe_2)]_3$	H_2[e]	2199
	$TaSi_x$	TaF_5	Si_2H_6	2200
	Ta_2SrO_6	$Sr[Ta(OEt)_5(Me)]_2$	O_2[e]	1514
74	Tungsten			
	W	WF_6	SiH_4	2201–2206
		WF_6	Si_2H_6	165, 184, 187, 193, 220, 249, 309, 314, 1183, and 2207–2219
		WF_6	B_2H_6	2202, 2205, and 2206
	WO_x	WF_6	H_2O	2220
		WF_6	[q]	2221

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
		WF_xO_y	H_2O	2220
		$WOCl_4$	…[q]	2222
		$W_2(NMe_2)_6$	H_2O	2223
	W_xN	WF_6	NH_3	944, 945, 2207, and 2224–2226
		WF_6	NH_3[e]	2226–2229
		WF_6	$NH_3 + B_2H_6$	2230
		$W(N^tBu)_2(NMe_2)_2$	NH_3	1067, 1636, 1639, 1689, and 2231–2234
		$W_2(NMe_2)_6$	NH_3	2235
	W_xC	$W(N^tBu)_2(NMe_2)_2$	H_2, N_2[e]	2236
	WN_xC_y	WF_6	$NH_3 + Et_3B$	945, 1939, 2173, and 2384–2389
		$W(CpEt)(CO)_2(NO)$	H_2[e]	2237
	WS_2	WF_6	H_2S	2238 and 2239
77	Iridium			
	Ir	$Ir(acac)_3$	O_2	95, 98, 276, 832, 835, and 2240–2245
		$Ir(acac)_3$	$O_3 + H_2$	2246
		$Ir(acac)_3$	[q]	2247
		Ir(CpEt)(cod)	O_2	2248
		Ir(CpEt)(cod)	NH_3[e]	2249
		Ir(CpMe)(chd)	O_2	2250
	IrO_2	Ir(CpEt)(chd)	$O_2 + H_2$[e]	2251
		Ir(CpEt)(cod)	O_2	2248 and 2249
		$Ir(acac)_3$	O_3	2252
78	Platinum			
	Pt	$Pt(CpMe)Me_3$	O_2	95, 98, 295, 350, 803, 1642, 1644, 1651, 1676, 2013, 2015, 2243, 2245, and 2253–2271
		$Pt(CpMe)Me_3$	O_2[e]	2260
		$Pt(CpMe)Me_3$	H_2	2266 and 2272
		$Pt(acac)_2$	H_2	1092
		$Pt(acac)_2$	O_3	2273
	PtO_x	$Pt(CpMe)Me_3$	O_2[e]	2260
		$Pt(acac)_2$	O_3	2273
80	Mercury			
	HgTe	$HgMe_2$	MeAyTe	1748 and 1750
82	Lead			
	PbO_2	$Pb(thd)_2$	H_2O	915 and 2274
		$Pb(thd)_2$	O_3	2275
		$Pb(tod)_2$	H_2O	913
		$PbPh_4$	O_3	2275
		$Pb(dmamp)_4$	H_2O	2276
	PbS	$PbBr_2$	H_2S	2277
		PbI_2	H_2S	2277
		$Pb(OAc)_2$	H_2S	2277
		$Pb(O^tBu)_2$	H_2S	2278
		$Pb_4O(O^tBu)_6$	H_2S	2278
		$Pb(thd)_2$	H_2S	2277–2281
		$Pb(dedtc)_2$	H_2S	2277 and 2278
83	Bismuth			
	BiO_x	$Bi[N(SiMe_3)_2]_3$	H_2O	2282
		$Bi(thd)_3$	H_2O	2283
		$Bi(O^tBu)_3$	H_2O	2283
		$Bi(dmb)_3$	H_2O	2283

TABLE I. (*Continued.*)

Z	Material	Reactant A[a]	Reactant B	References
	Bi_2Se_3	$BiCl_3$	$(Et_3Si)_2Se$	1148
	Bi_2Te_3	$BiCl_3$	$(Et_3Si)_2Te$	1148
	$Bi_xSi_yO_z$	$Bi(CH_2SiMe_3)_3$	O_3	662 and 663

[a]The metal thd complexes may have been slightly oligomerized and contain a neutral adduct molecule.
[b]Diamond.
[c]Atomic hydrogen produced by a hot tungsten filament.
[d]R refers to alkyl chains of various lengths.
[e]Plasma.
[f]This process is atypical for ALD: It deposits nanolaminates of Al_2O_3 and SiO_2, where the growth-per-cycle is many nanometers, through a catalytic mechanism.[43]
[g]Cat. = catalyst.
[h]HMDS = hexamethyldisilazane, $(Me_3Si)_2NH$.
[i]Controlled desorption.
[j]Irradiation.
[k]Temperature treatment, such as flash heating or temperature modulation.
[l]He plasma treatment.
[m]Reactant B was formaline, containing ∼37% of formaldehyde HCHO and some ethanol in water.
[n]Photo-assisted.
[o]Decomposed.
[p]Preliminary surface science investigation.
[q]Only one half-reaction of an ALD reaction cycle was carried out.

reactive (e.g., O_2, H_2, N_2) can be used. The conformality of PEALD films is however limited due to the finite lifetime of radicals and charged species, as discussed in recent work by Knoops *et al.*[42] In addition to these classes of compounds, various organic compounds have also been used, with reducing or oxidizing characteristics, as needed.

C. Overview of recent two-reactant ALD material studies

Recently, the development of ALD processes has been more need- than curiosity-driven. Microelectronics has been the most important application area motivating the ALD research. In microelectronics, high-κ oxides for both transistors and DRAM capacitors have formed the largest focus area. New processes have been developed for Group 4 metal (Zr, Hf) oxides but doping of the oxide films, making of nanolaminates, and ternary compounds have been important part of the research aiming for increase of the κ-value and more stable films. Publication-wise the most active ALD research topic has recently been the integration of high-κ oxides with various III–V and Ge channel materials.

Microelectronics has also motivated the development of ALD processes for metal and conducting metal oxide films to be used as electrodes. Noble metal and noble metal oxide films, and especially the interfacial behavior between dielectrics and metal electrodes, have received a lot of attention. Phase-change materials for non-volatile electronic memories present a new area for ALD. For that application, new chemistry and processes to deposit germanium and antimony tellurides have been developed.

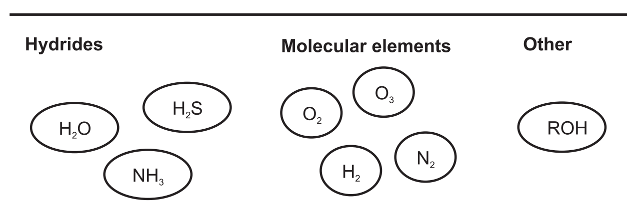

FIG. 6. Overview of the classes of non-metal reactants used in ALD.

MEMS (nanoelectromechanical systems, NEMS) and different nanotechnology applications are naturally suited for ALD. In these studies, no new processes have been developed but existing well-working processes such as those for Al_2O_3, TiO_2, and ZnO have been applied. Protective (barrier) layers and optics form other important and increasing application areas. They also mostly rely on existing processes but for optics new chemistry has been studied for metal fluoride films. In the case of protective films, the substrate to be covered is often polymer material, setting limitations for the process temperature.

Energy applications form yet another increasing interest area, which has motivated ALD thin film research. Both nanostructured dye-sensitized solar cells (DSSC) and Li-ion batteries could utilize the conformality of ALD films. In case of batteries, new processes have been developed for lithium-containing films while DSSC could utilize the existing oxide processes. In photovoltaics, the surface passivation of silicon solar cells with ALD-Al_2O_3 has shown excellent results and has been extensively studied.

Both for PV applications and barrier layers for high volume products, the productivity of conventional ALD even in batch mode is of a concern. Therefore, increasing effort has been devoted towards fast spatial ALD, where the substrate is moved under spatially separated continuous precursor and purge gas flows. So far, this work has focused almost exclusively on Al_2O_3, and it will be interesting to see how well other ALD processes can be transferred to this mode.

The literature contains extensive amounts of information on the development of new ALD processes and the

characteristics of existing processes. The collection of two-reactant ALD processes made in 2005 in Ref. 2 has now been updated to correspond to the situation about 5 years later, at the end of 2010. Accordingly, Table I collects information on ALD processes based on two reactants. The data collection principles have been the same as in the earlier review:[2] Investigations published in scientific journals have been included; we have not judged the contents of the paper as to whether the process really obeys the principles of ALD but have included it if the authors present it as ALD; and investigations have been included if they bring some information or data on the particular ALD process which could be useful to others using the same process. Therefore, conference papers are generally not included in the table (with some exceptions); some investigations may be included which in fact do not correspond to ALD but rather some other form of CVD; and many publications are not referred to, where ALD layers have merely been used as part of a multi-layer process flow while the properties of the ALD layers have not been individually investigated. Restricting the table contents mainly to two-reactant processes leaves out information on ALD processes based on three or more reactants, laminates, mixed oxides, and doping. Therefore, although the table is meant to cover all two-reactant ALD processes, it is not an overview of *all* ALD processes.

The metal reactants used in the processes collected in Table I contained almost a hundred different ligands. To assist reading the table, the ligands have been collected in their own figures. Figure 7(a) contains the inorganic, organometallic, and some metal-organic ligands, and Figure 7(b) completes the set of metal-organic ligands. Also, to assist reading Table I, a quick overview of the different types of materials grown in two-reactant ALD processes is given in Figure 8 in the form of a periodic table.

Examination of Table I reveals several popular ALD processes, which are repeatedly used in various applications. The process that appears to be used the most often is the $AlMe_3 - H_2O$ to deposit Al_2O_3: Since the previous review,[2] this process has gained over 200 new citations. $AlMe_3$ is also often combined with ozone or O_2 plasma. Examples of other widely applied ALD processes are the $ZnEt_2 - H_2O$ process to deposit ZnO (almost 100 new citations), the $HfCl_4 - H_2O$ process to deposit HfO_2 (almost 80 citations), and $TiCl_4 - H_2O$ process to deposit TiO_2 (about 70 new citations).

Many new binary compound materials have been demonstrated since the previous review.[2] Of oxides, lithium oxide has been deposited from $Li(O^tBu)$ and H_2O,[44] barium oxide from cyclopentadienyl-based reactants $Ba(Cp^tBu_3)_2$ and $Ba(Cp^nPrMe_4)_2$ combined with H_2O,[1519,1830] and ytterbium oxide from $Yb(thd)_3$ and ozone.[1877] ALD of noble metal oxide films has been demonstrated for rhodium,[1695] iridium,[2252] and platinum.[2273] Also PEALD processes to deposit oxides of iridium and platinum have been developed.[2251,2260] Of nitrides, copper nitride has been grown from $Cu(^sBuAMD)$ and NH_3 and $Cu(hfac)_2$ and $NH_3 + H_2O$.[1140,1141] Of sulphides, titanium sulphide has been deposited from $TiCl_4$ and H_2S,[999] tin sulphide from $Sn(acac)_2$ and H_2S,[384] and antimony sulphide from $Sb(NMe_2)_3$ and H_2S.[1226,1829] The selection of ALD-made fluoride materials has been extended substantially using titanium and tantalum fluorides as fluorine sources: Magnesium fluoride was deposited from $Mg(thd)_2$ and TiF_4 or TaF_5,[66,68] yttrium fluoride from $Y(thd)_3$ and TiF_4,[1534] and lanthanum fluoride from $La(thd)_3$ and TiF_4.[67,1858] Many new selenides have been deposited: bismuth selenide from $BiCl_3$ and $(Et_3Si)_2Se$,[1148] copper selenide from $Cu(O_2C^tBu)_2$ or CuCl and $(Et_3Si)_2Se$,[1148] and indium selenide from $InCl_3$ and $(Et_3Si)_2Se$.[1148] Also new tellurides have been deposited: germanium telluride from $GeCl_2 \cdot (C_4H_8O_2)$ and $(Et_3\ Si)_2Te$,[1148] antimony telluride from $SbCl_3$ and $(Et_3Si)_2Te$,[1148] and bismuth telluride from $BiCl_3$ and $(Et_3Si)_2Te$.[1148] Of arsenides, manganese arsenide has been deposited from $Mn(CpMe)_2$ and $As(NMe_2)_3$,[1052] and of silicides, cobalt silicide has been deposited from $CoCp_2$ and $NH_3 - SiH_4$ plasma.[1091]

In addition to compound materials, also many new elemental metals have been demonstrated since the previous review.[2] Rhodium has been deposited from $Rh(acac)_3$, employing similar oxidative O_2-based chemistry as in the other noble-metal ALD processes.[1694] The corresponding O_3-based process leads to oxidation of rhodium.[1695] Silver films have been deposited by PEALD using $Ag(O_2C^tBu)(PEt_3)$ and H_2 plasma,[1705] and silver nanoparticles by thermal ALD using a Ag(hfac)(cod)–propanol process.[1706] Metallic zinc has been detected in a film made by the $ZnEt_2 - H_2O$ process, when large $ZnEt_2$ doses were used.[1149] The main product of this process remains ZnO, however, and since pure metallic zinc films will be still difficult if not impossible to make by ALD; elemental zinc deposition is not shown with black but with gray background in Figure 8. Most recently in 2011 (not shown in Table I), antimony films have been grown by combining the $SbCl_3$ and $Sb(SiEt_3)_3$ reactants.[534]

Several trends can be seen by examining Table I regarding the development of new ALD processes. Significant attention has been given since the previous review[2] to the development of new processes to deposit noble metals Ru, Pd, Ir, and Pt. Also metallic Co and Ni have clearly been in the center of attention. Regarding compound materials, especially active process development has been made for SiO_2, HfO_2, FeO_x, and TaN. For groups of reactants, active development has been made with cyclopentadienyls (e.g., to deposit Yb_2O_3, ZrO_2, Ru, and La_2O_3), alkylamides (e.g., to deposit Al_2O_3, AlN, TiO_2, ZrO_2, and HfO_2), and on the nonmetal precursor side with alkyl silyl chalcogenides to deposit various selenides and tellurides. PEALD is at a rapidly developing stage, with new processes developed since the previous review for Al_2O_3, AlN, SiO_2, TiO_2, TiN, V_2O_5, Co, $CoSi_2$, Ni, Cu, ZnO, Ga_2O_3, Y_2O_3, ZrO_2, ZrN_x, NbN, Ru, Pd, Ag, SnO_2, La_2O_3, Er_2O_3, HfO_2, HfN, Ta, Ta_2O_5, TaN, Ir, IrO_2, Pt, and PtO_x.

D. Growth of ternary compounds by ALD

The number of ternary compounds studied in ALD is limited but increases. The compounds studied are mainly multi-component oxides, as shown in Table II. Interest in them stems from their high dielectric constant, ferroelectric properties, or magnetic properties. Ternary chalcogenides

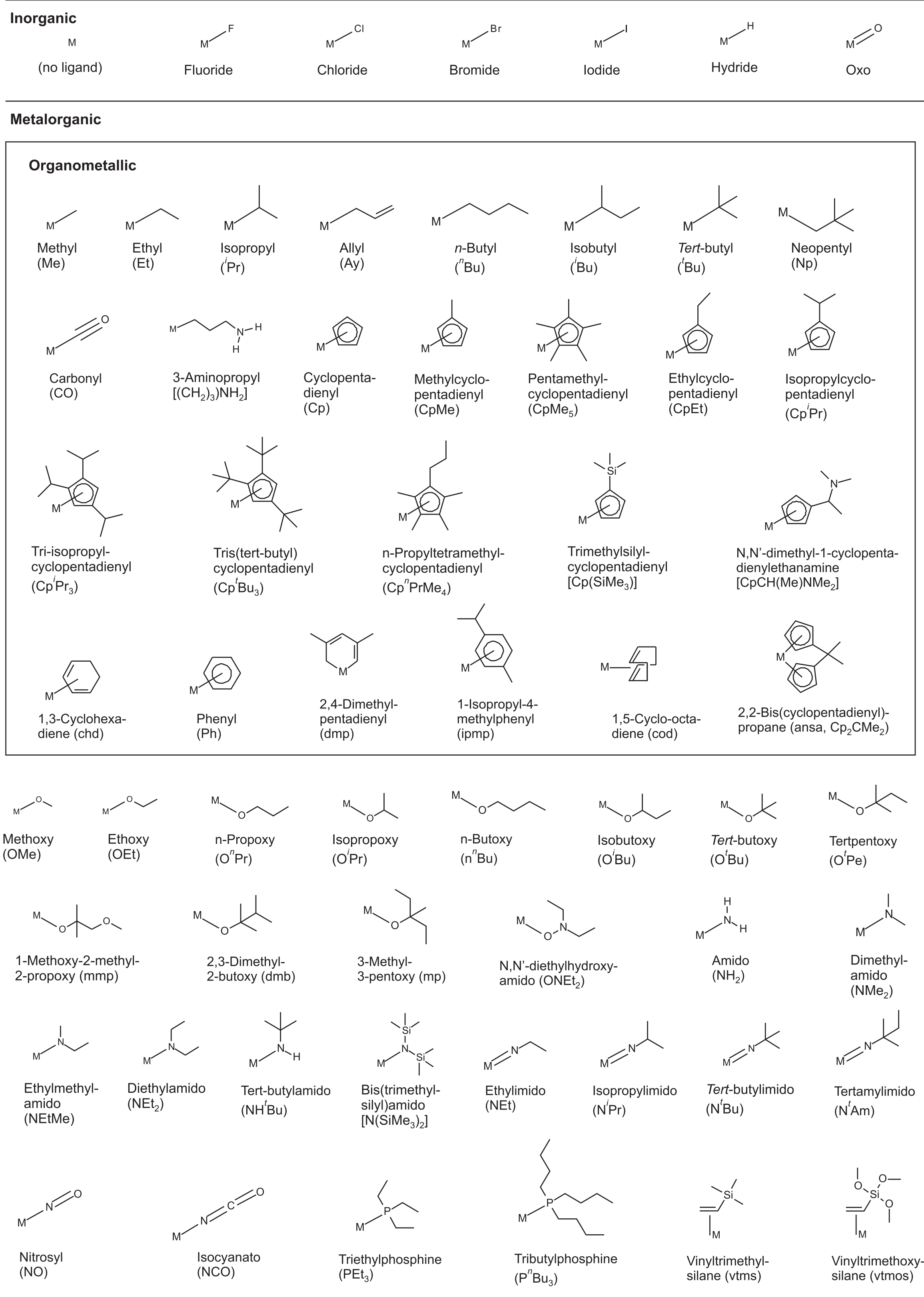

FIG. 7A. Ligands for ALD metal reactants, part 1 of 2.

have recently been studied for phase-change memory applications.[1148] Lithium-containing ternary or quaternary films gain interest because of their possible use in all-solid-state thin-film Li-ion batteries.[45]

There are several ways how the ternary compounds can be deposited. The most straightforward way is the one where separate binary deposition cycles are mixed to get the right stoichiometry. Since the growth rates per cycle differ in the

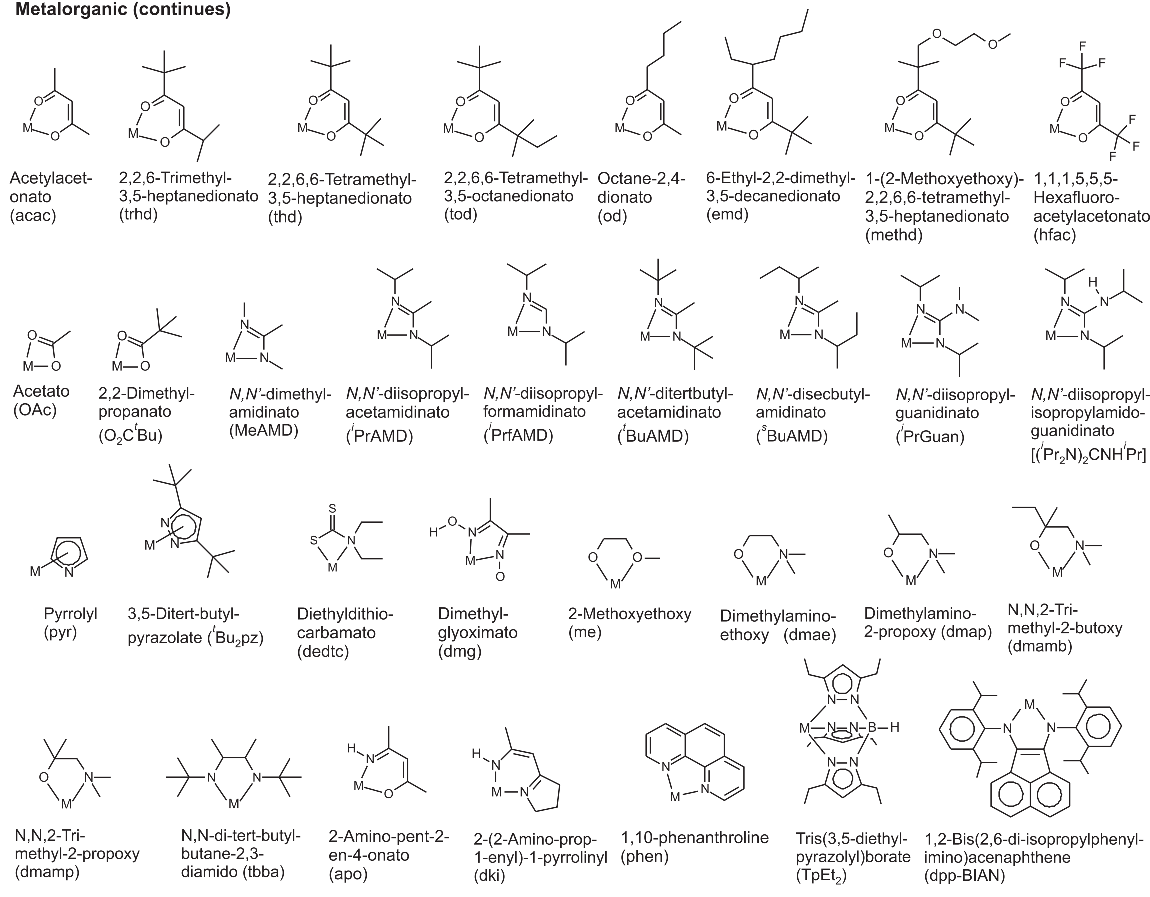

FIG. 7B. Ligands for ALD metal reactants, part 2 of 2.

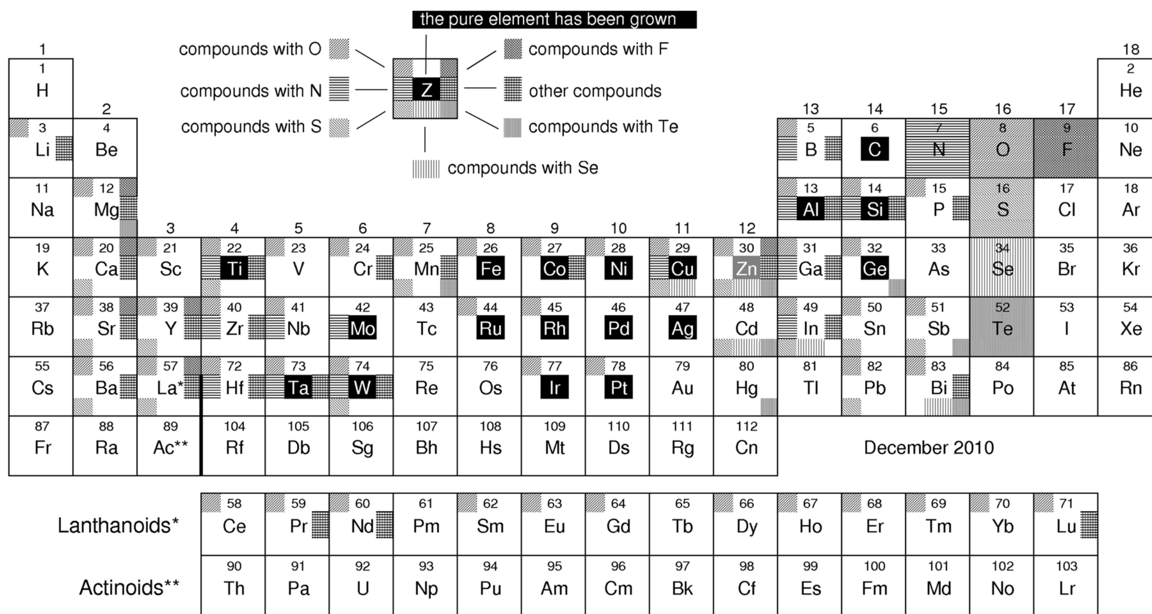

FIG. 8. Overview of the materials grown by ALD. Classification according to Reactant A, with details of the investigations in Table I. Growth of pure elements as well as compounds with oxygen, nitrogen, sulphur, selenium, tellurium, fluorine, and other compounds grouped together is indicated through shadings of different types at different positions. The elements are named according to the recommendations of The International Union of Pure and Applied Chemistry (IUPAC, http://www.iupac.org/reports/periodic_table/, dated 21 January 2011).

TABLE II. Examples of ALD processes for ternary and quaternary compounds.

Compound	Precursors	References
$MgAl_2O_4$	$Mg(C_5H_5)_2 + Al(CH_3)_3 + O_3$	59
$LaAlO_3$	$La(thd)_3 + Al(acac)_3 + O_3$	2284
	$La(^iPr)AMD)_3 + Al(CH_3)_3 + H_2O$	2285
$NdAlO_3$	$Nd(thd)_3 + Al(CH_3)_3 + O_3$	1865
$MSiO_4$ (M = Zr, Hf)	$MCl_4 + Si(OEt)_4$	103
	$MCl_4 + Si(OEt)_4 + H_2O$	103
	$MCl_4 + Si(O^nBu)_4$	103
	$MCl_4 + H_2O + NH_2(CH_2)_3Si(OEt)_3 + O_3$	2286
$Bi_4Si_3O_{12}$ (Bi_2SiO_5)	$Bi(CH_2SiMe_3)_3 + O_3$	662
$Ca_{10}(PO_4)_6(OH)_2$	$Ca(thd)_2 + O_3 + (CH_3O)_3PO + H_2O$	673
$RScO_3$	$R(thd)_3 + Sc(thd)_3 + O_3$	2287
(R = lantanide)	$R(MeC_5H_4)_3 + Sc(C_5H_5)_3 + H_2O$	2288
$MTiO_3$ (M = Sr,Ba)	$M(^iPr)_3C_5H_2)_2 + Ti(O^iPr)_4 + H_2O$	2289
	$M(^tBu)_3C_5H_2)_2 + Ti(OMe)_4 + H_2O$	1505
	$M(thd)_2 + O_3 + Ti(O^iPr)_4 + H_2O$	1508
	$M(thd)_2 + Ti(O^iPr)_2(thd)_2 + H_2O$	2290
$Bi_4Ti_3O_{12}$	$Bi(CH_2SiMe_3)_3 + Ti(OMe)_4 + H_2O$	2291
	$Bi(Ph)_3 + O_3 + Ti(O^iPr)_4 + H_2O$	663
$PbTiO_3$	$Pb(Ph)_4 + O_3 + Ti(O^iPr)_4 + H_2O$	2292
	$Pb(dmamp)_2 + H_2O + Ti(O^tBu)_4\ H_2O$	2293
$LaMnO_3$	$La(thd)_3 + Mn(thd)_3 + O_3$	1038
$LaCoO_3$	$La(thd)_3 + Co(thd)_2 + O_3$	1088
$LaNiO_3$	$La(thd)_3 + Ni(thd)_2 + O_3$	1111
$LaGaO_3$	$La(thd)_3 + Ga(thd)_3 + O_3$	2294
$PbZrO_3$	$PH_4Pb + Zr(thd)_4 + O_3$	2295
$LiLaO_x$	$Li(thd) + La(thd)_3 + O_3$	45
$LaLuO_3$	$La(thd)_3 + Lu(thd)_3 + O_3$	2296
$SrTa_2O_6$	$SrTa_2(OEt)_{10}(dmae)_2 + H_2O$	2297
$SrBi_2Ta_2O_9$	$SrTa_2(OEt)_{10}(dmae)_2 + H_2O + Bi(N(SiMe_3)_2)_3 + H_2O$	2282
$Ge_2Sb_2Te_5$	$GeCl_2{\cdot}L + SbCl_3 + (R_3Si)_2Te$	1148
$CuGaS_2$	$CuCp{\cdot}PEt_3 + Et_3Ga + H_2S$	2298

binary processes, careful adjustment in pulsing is needed.[2289] The optimum process temperatures may also be different in the binary processes, making the temperature window for ternary process narrow.[2297] The second possibility for deposition of ternary compounds is the use of bimetallic single-source precursors. The approach is common in CVD but very sparsely studied in ALD, the most well-known case being the deposition of $SrTa_2O_6$ from $SrTa_2(OEt)_{10}(dmae)_2$ and water.[2282] The reason for the limited use of bimetallic precursors is the lack of volatile, thermally stable, and reactive compounds. The third way to make ternary compounds is to use reactants, which supply two of the three elements into the film. This has been exemplified with silicon alkoxides, which when reacting with metal chlorides produce metal silicates.[103]

In transistors and DRAMs, a shift from SiO_2 to binary high-κ oxides, like HfO_2 and ZrO_2 or multilayer structures ($ZrO_2 - Al_2O_3 - ZrO_2$), has already occurred. The further step to higher-κ materials is the adoption of ternary oxides. Aluminates, especially $LaAlO_3$, have been of interest, because it makes a stable structure on Si, contrary to pure La_2O_3.[2284] The κ value is not very high, being something between those of the binary oxides (9–27).[2285] As a very high-κ material, $SrTiO_3$ is attracting constant interest as a possible material for DRAMs. The limited Sr precursor chemistry has slowed down the industrial use of ALD $SrTiO_3$. The oldest chemistry is based on Sr cyclopentadienyls with water as the oxygen source.[1505,2289] Later, Sr β-diketonates have been used in combination with ozone or oxygen plasma.[1508,2299] Since the application of ALD $SrTiO_3$ has been delayed, mixed rare-earth oxides have received attention as possible candidates for high-κ materials in microelectronics. Combination of large rare-earth ions with small ones can produce separate ternary compounds with perovskite structure, which may have κ-values above 20. Scandium as the smallest rare-earth ion forms perovskites with all larger lanthanide ions (La–Gd), and lanthanum, for example, forms perovskites even with the smallest lanthanides ($LaLuO_3$).[2296] Ternary rare-earth perovskite oxides can be made with both cyclopentadienyl and β-diketonate chemistry, with water or ozone, respectively, being the oxygen source.[2287,2288]

Ferroelectric materials are usually ternary of quaternary oxides. $BaTiO_3$ is one of the most widely studied ferroelectric materials, and ALD films have been deposited similarly to $SrTiO_3$.[2289,2297,2300] Bismuth titanates form another group of ALD ferroelectric materials. In their deposition, the precursor chemistry of bismuth has played a major role,[2291,2301,2302] and so far the most versatile precursor has been $Bi(OCMe_2^iPr)_3$.[2283] Quaternary compound $SrBi_2Ta_2O_9$ is an interesting ferroelectric material. In its ALD process, the bimetallic $SrTa_2(OEt)_{10}(dmae)_2$ has been utilized.[2297] $PbZrO_3$ films have been grown Ph_4Pb, $Zr(thd)_4$ and ozone at around 300 °C.[2295] $PbZrO_3$ is an interesting material due to its applications in sensors and transducers but especially as a component for lead zirconate titanate (PZT) ferroelectrics.

Ternary oxide films are usually deposited at low temperatures, because of the low thermal stability of the precursors. As-deposited films are amorphous. Crystalline films are obtained after high-temperature annealing, which for example in case of mixed rare earth oxides requires 800 − 1000 °C. It would be beneficial from the electrical property point of view to get as-deposited crystalline films. By selecting precursors tolerating higher temperature, crystalline $SrTiO_3$ films have been deposited at 370 °C.[2290] Electrical performance of these films is better than that of films crystallized by post-deposition annealing.

The number of other ternary compounds studied in ALD is limited, some early work on $CuGaS_2$ (Ref. 2298) and recent studies on germanium antimony tellurides[1148] being the examples. Because of the importance of many ternary compounds, it is obvious that much more ALD studies will be devoted to this area.

III. CRYSTALLINITY OF INORGANIC ALD MATERIALS: OVERVIEW

This section analyzes the published experimental data for information on the crystallinity and phase of inorganic materials deposited by ALD from different reactants at different temperatures. The data are tabulated for easy reference. Case studies are presented to illustrate the effect of different process parameters on crystallinity for

representative materials: aluminium oxide, zirconium oxide, zinc oxide, titanium nitride, zinc sulfide, and ruthenium.

A. Metal oxides

1. General

Though the list of various oxides deposited by ALD is long, as is the list of metal precursors used in these processes, from the oxygen precursor point of view the processes divide into three main groups: those using water, ozone, and oxygen plasma. While the water processes proceed through protonation of the ligands that remain intact, with ozone and oxygen plasma the mechanism is more complicated and more combustion-like. Besides these three main groups, one can also distinguish various alternative oxygen precursors like H_2O_2, alcohols, and N_2O. A special case includes processes that do not exploit a separate oxygen precursor but one of the metal precursors (metal alkoxide or carboxylate) serves both as a metal and oxygen source. This chemistry was developed originally to avoid oxidation of the substrate surface, silicon in particular, by the common oxygen sources.[103]

High-κ oxides for both transistors and DRAM capacitors have formed one of the most important driving forces for ALD oxide research. New processes have been developed for Group 4 metal oxides, the focus being first in hafnium oxide and then shifting to zirconium oxide since its tetragonal and cubic forms have higher κ-values than the corresponding hafnium oxide phases. Meanwhile, attention has also been paid to TiO_2, because its rutile phase can have very high κ values 80–150. The problem is the small band gap of TiO_2 and accordingly high leakage currents. Doping with aluminum has given promising results.[904] From titanates, $SrTiO_3$ has received much attention as a potential next-generation dielectric material for DRAMs. The study of ALD of rare-earth oxide films as potential high-κ oxides has been existing during the last years. Almost all lanthanide oxides have been studied. In particular, interesting results in respect of stability and reasonably high κ-value have been obtained with ternary compounds having the perovskite structure, which have been formed from rare earth aluminates and from ternary oxides containing two rare earth elements. Ferroelectric, magnetic, and multiferroic oxide films have been very recently of interest. These ternary compounds have needed new precursors and process development especially for bismuth and iron.

The increasing interest towards organic and flexible electronics and displays is reflected in studies of transparent conducting oxides. ALD of ZnO:Al is of vital interest.

The properties of the oxide films strongly depend on the crystal structure. Not only the difference between amorphous and crystalline phases but also the difference between the different crystalline phases of the oxides has to be taken into account. The Group 4 metal oxides TiO_2, ZrO_2 and HfO_2 are illustrative examples, as they all can exist as amorphous or as 3–4 different crystalline phases in the ALD films. In the dielectric properties, the difference is seen in the κ-values and leakage currents, amorphous films having the lowest κ-value but the best leakage current properties. Therefore, it is of utmost importance to know the conditions where different crystalline phases are formed, and the dependencies between the precursor chemistry, process parameters, and crystalline phases.

An overview of crystallinity of metal oxide films grown by two-reactant ALD processes is shown in Table III. This table, similarly to the corresponding crystallinity tables later for other classes of materials, has been drawn up for two-reactant ALD processes on the basis of experimental evidence for the presence of amorphous and crystalline phases in films made by particular ALD processes on particular substrates at a specified ALD temperatures. The data are collected for ALD films in their as-deposited state; post-deposition treatments are excluded. The presence or absence of different crystalline phases is indicated on the basis of diffraction or spectroscopy investigations. The investigations include Raman scattering and x-ray absorption spectroscopies as well as x-ray, neutron, and electron diffraction. All notations of crystallinity or amorphicity in the table should therefore be reliable. In many cases the phase interpretation is not straightforward due to overlapping of peaks, however. Problematic interpretations are noted in the table inputs if the ambiguity has been reported in original papers.

2. Case: Aluminum oxide

Aluminum oxide is the most studied and used material in ALD. The deposition is usually performed using TMA (trimethylaluminum) and water as precursors. This is almost an ideal self-limiting ALD process and considered as a model system for ALD. The first report dates back to late 1980s[107] but since then the process has been extensively studied and characterized in detail both *ex situ* and *in situ*. The details of the TMA-water process have been discussed by Puurunen in the earlier review.[2] Besides water, ozone and recently also oxygen plasma have been extensively used with TMA. Other aluminum precursors, such as chloride, bromide, different alkoxides, alkylamides, amidinates and mixed ligand compounds ($AlMe_2Cl$, $Al(OR)_xR'_y$), have been studied in ALD of Al_2O_3, but TMA as a high-vapour-pressure liquid has many advantages over the other precursors. ALD Al_2O_3 films are widely used as protective, barrier, and dielectric layers as such or as component in nanolaminates.

The TMA–water process can be used at $30-300\,^{\circ}C$, and $300\,^{\circ}C$ is a common upper limit for all organometallic aluminum precursors, but in some reports temperatures as high as $500\,^{\circ}C$ have been used. Chlorides tolerate much higher temperatures, but for practical reasons, also with $AlCl_3$ the growth temperatures have usually been $\leq 500\,^{\circ}C$ or below. All aluminum oxide films grown below $600\,^{\circ}C$ are amorphous regardless the type of the substrate. Crystallization of the dielectric ALD Al_2O_3 upon post-deposition annealing has been studied to increase the dielectric constant (Figure 9). Crystallization of 5 nm thick films on 1 nm chemical SiO_2 oxide occurs at $900\,^{\circ}C$. The temperature depends on film thickness, and thicker films crystallize at lower temperatures possibly because thicker films have a higher likelihood to contain seeds to originate the nucleation.[151] Besides the thickness, the substrate has some effect: On 4H-SiC wafers epitaxial $\gamma-Al_2O_3$ was obtained at

TABLE III. Crystallinity studies of ALD binary oxide films made by diffraction or spectroscopy.[a]

Reactant A	Reactant B	Substrate(s)	Phases				References
Li_2O (LiOH)			Amorphous				
Li(O^{t}Bu)	H_2O	Si, SLG	225 °C				44
B_2O_3			Amorphous	Hexagonal			
BBr_3	H_2O	Si, SLG	20–50 °C				47
MgO			Amorphous	Cubic			
$Mg(thd)_2$	O_3	Si, SLG		225–250 °C			65
$Mg(thd)_2$	H_2O_2	SLG		325–425 °C			64
$Mg(Cp)_2$	H_2O	H-Si, Si, SLG		105–375 °C			58, 60, and 61
$Mg(Cp)_2$	H_2O	Si	370–500 °C	500–900 °C			55–57
$Mg(CpEt)_2$	H_2O	Si		150 °C			63
Al_2O_3			Amorphous	Hexagonal	Cubic	Tetragonal	
$AlCl_3$	H_2O	Si, SiO_2, glass, ITO, Al, PC	100–500 °C		600–800 °C[b]	600–800 °C[b]	79, 83, 86, 88, 93, and 2303
$AlCl_3$	O_2	Sapphire[f]		660 °C			101
$AlCl_3$	O_2	Nb[f]		450 °C			101
$AlCl_3$	^{t}BuOH	Si, SLG, ITO, Al	500 °C				88
$AlCl_3$	$Al(OEt)_3$	H-Si	400 °C				103
$AlCl_3$	Al(O^{i}Pr)$_3$	SLG, H-Si	150–375 °C				103 and 104
$AlMe_3$	H_2O	Si, H-Si, GaAs, SiC, MgO, ZnO, CuO, $LiMnO_2$, $LiCoO_2$, SiO_2, Al_2O_3, TiO_2, PC, PMMA, PEEK, PTFE, PET, PP, GaQ3, cotton, paper, CNC, MWCNT, Fe, W, SS	25–500 °C				86, 94, 107, 108, 118, 123, 158, 159, 179, 187, 189, 207, 209, 214, 219, 223–226, 243, 250, 258, 262, 268, 273, 279, 282, 298, 302, 309, 315, 321, 339, 348, 361, 362, 367, 387, 392, 624, 657, 770, 832, 1922, and 2304–2309
$AlMe_3$	H_2O[d]	Cr	200 °C				292
$AlMe_3$	H_2O	ITO			300 °C[j]		313
$AlMe_3$	H_2O_2	Si,H-Si, GaAs, SiO_2, SS, Cu, Al	24–700 °C	700–750 °C[b]			399 and 402
$AlMe_3$	^{i}PrOH	Si	250 °C				460
$AlMe_3$	O_3	SiC, SiO_2	300–450 °C				421 and 2309

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases				References
$AlMe_3$	O_2[c]	Si, borosilicate, ITO, PE, PP, wool	25–300 °C				432
$AlMe_3$	$Al(O^iPr)_3$	H-Si	300 °C				103
$AlMe_2Cl$	H_2O	SLG	125–500 °C				462
$AlMe_2(O^iPr)$	H_2O	H-Si	100–250 °C				465
$AlEt_3$	H_2O	Si	600–750 °C				56
$Al(O^nPr)_3$	H_2O	Si, SLG, ITO, Al	500 °C				88
$Al(O^nPr)_3$	^tBuOH	Si, SLG, ITO, Al	500 °C				88
$Al(mmp)_3$	H_2O	Si	250 °C				466
$Al(NMe_2)_3$	H_2O	Si	200–400 °C				469
$Al(NEt_2)_3$	H_2O	Si	250–325 °C				470
$Al(NEt_2)_3$	O_3	Si	200–325 °C				472 and 473
$Al(N^iPr_2)_3$	H_2O	Si	250–325 °C				470
SiO_2			Amorphous				
$Si(OMe)_4$	H_2O	Au-TiO_2 catalyst	150 °C				602
$SiH(NMe_2)_3$	H_2O_2	TiO_2	500 °C				624
$SiH_2(NMe_2)_2$	O_3	H-Si	225 °C				2310
CaO			Amorphous	Cubic			
$Ca(thd)_2$	O_3	Si, Glass	225–350 °C	350 °C			672 and 673
$Ca(Cp^iPr_3)_2$	H_2O	Si, borosilicate		205–300 °C			674
Sc_2O_3			Amorphous	Cubic			
$Sc(thd)_3$	O_3	SLG, Si		335–375 °C			682
$Sc(Cp)_3$	H_2O	SLG, Si		250–350 °C			682
$Sc(^iPrAMD)_3$	H_2O	H-Si		290–360 °C			683
TiO_2			Amorphous	Anatase	Orthorhombic	Rutile	
TiF_4	H_2O	borosilicate		300–500 °C			687
TiF_4	H_2O	SLG		300–400 °C		400–500 °C	687
$TiCl_4$	H_2O	Si, H-Si, Ge, GaAs,ZnO, SiO_2, Al_2O_3, glass, PC, PS, MWCNT, Ni, Mo, Ti, Ta, Pt	25–600 °C	125–680 °C		275–680 °C	44, 94, 128, 207, 365, 719, 720, 722–724, 727, 737, 738, 742, 744, 746–749, 753, 756, 758–760, 762, 767, 769, 770, 773, 775–778, 780–782, 785, 786, 788–790, 795, 800, 801, 803, 805, 806,

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases				References
							808, 809, 814, 1011, and 2311–2313
$TiCl_4$	H_2O	RuO_2	150 °C			275–600 °C	780 and 799
$TiCl_4$	H_2O	RuO_2 on sapphire, Sapphire(0001)				425–450 °C[f]	799 and 812
$TiCl_4$	H_2O	Al_2O_3	100–150 °C	150–350 °C		425–500 °C[f]	748 and 754
$TiCl_4$	H_2O	MgO[f]		300 °C			757
$TiCl_4$	H_2O	Si, SiO_2, SS, KBr	100–350 °C	150–427 °C	300–427 °C	400–427 °C	123, 207, 728, 732, 733, 739, 750, 751, 772, and 807
$TiCl_4$	H_2O[h]	Si	27–67 °C			27–67 °C	731
$TiCl_4$	H_2O_2	Fe	100 °C				490
$TiCl_4$	H_2O_2	H-Si, MgO[f]		340–490 °C			815
$TiCl_4$	MeOH	Si, SiO_2		375 °C			817
$TiCl_4$	MeOH	Al_2O_3				375 °C	817
$TiCl_4$	O_2[c]	H-Si		110–200 °C			818 and 819
TiI_4	H_2O_2	Si, Al_2O_3[f], SiO_2, SLG	135 °C	165–375 °C		300–490 °C	820, 823, and 824
TiI_4	H_2O	Si, Al_2O_3[f], SiO_2, AAO, CNS	135 °C	165–375 °C		445 °C	820–822, 824
TiI_4	O_2	H-Si		235–457 °C		457 °C	826
TiI_4	O_2	Al_2O_3[f]				300–455 °C	827
TiI_4	O_2	MgO[f]		375–455 °C			827
$Ti(CpMe_5)(OMe)_3$	O_3	Ru	280 °C			280 °C	828
$Ti(CpMe_5)(OMe)_3$	O_3	TiN		280 °C			828
$Ti(OMe)_4$	H_2O	Glass, Ni, Si, Ti	200–300 °C	250–350 °C			830, 834, 836, and 837
$Ti(OEt)_4$	H_2O	Si, H-Si, SiO_2, SLG, paper, Mo, Ti	100–300 °C	180–350 °C			128, 760, 773, 782, 831, 832, 837, 839–841, 845
$Ti(OEt)_4$	H_2O_2	H-Si, Mo	125–175 °C	225–350 °C			773 and 782
$Ti(O^iPr)_4$	H_2O	Si, SiO_2, Glass, PC, PVP, Al_2O_3, ZnO, SS, Ru, Pt, CrN, $TiSi_2$, CNC	50–225 °C	150–350 °C			128, 273, 328, 362, 788, 846, and 847, 848, 850, 852, 855, and 856, 859–861, 864–868, 870, 875, 877, 881, 914, and 2314

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases				References
$Ti(O^{i}Pr)_4$	H_2O	RuO_2, ISM	70–160 °C	225–300 °C		225–300 °C	318 and 859
$Ti(O^{i}Pr)_4$	H_2O_2	Si, SiO_2, PE, Fe, Ni	77–150 °C	180–300 °C			334, 848, and 882
$Ti(O^{i}Pr)_4$	O_2	silica	100–165 °C				736 and 884
$Ti(O^{i}Pr)_4$	O_3	Si, Pt		250 °C			899 and 1612
$Ti(O^{i}Pr)_4$	O_3	Ru				250 °C	899, 900, 1612, and 2315
$Ti(O^{i}Pr)_4$	O_3	Ru, TiN		250 °C		250 °C	906
$Ti(O^{i}Pr)_4$	O_2[c]	Si, SiN_x, borosilicate, Pt, RuO_2, PC, PP, wool	50–275 °C	250–300 °C			439, 864, 885, 886, 891, and 892
$Ti(O^{i}Pr)_4$	O_2[c]	H-Si, Ru, Ir		150–250 °C		150–250 °C	888, 890, and 892
$Ti(O^{i}Pr)_4$	O_2[c]	IrO_2				250 °C	892
$Ti(O^{i}Pr)_4$	H_2O[c]	Si	50–210 °C				864
$Ti(O^{i}Pr)_4$	N_2O[c]	Ru, Al_2O_3		250–280 °C		250–280 °C	890 and 907
$Ti(O^{i}Pr)_4$	HCOOH	Si, $SrTiO_3$, $LaAlO_3$, MgO, wool, cellulose, latex, CNT	150–200 °C				908
$Ti(O^{i}Pr)_4$	CH_3COOH	Si, $SrTiO_3$, $LaAlO_3$, MgO, wool, cellulose, latex, CNT	150–200 °C				908–911
$Ti(O^{i}Pr)_4$	NH_3	xerogel	140 °C				898
$Ti(thd)_2(O^{i}Pr)_2$	H_2O	Pt	220–420 °C				913
$Ti(thd)_2(O^{i}Pr)_2$	H_2O	Pt		340–470 °C			916
$Ti(trhd)_2(O(CMe_2Et)_2$	H_2O	Pt	220–420 °C				913
$Ti(NMe_2)_4$	H_2O	Si, H-Si, CeO_2, ZnO, Ni	50–250 °C	250–350 °C		300–350 °C	864, 914, 918, 919, 921, and 923
$Ti(NMe_2)_4$	H_2O[c]	Si	50–210 °C				864
$Ti(NMe_2)_4$	O_2[c]	Si, Kapton, SS, Zn	50–210 °C	250–400 °C		300–400 °C	864, 928, and 930
VO_x			Amorphous	Orthorhombic V_2O_5	Monoclinic VO_2		
$VOCl_3$	H_2O	silica		490 °C	490 °C		1005
$VO(acac)_2$	O_2	glass			400–475 °C		1019
$VO(O^{i}Pr)_3$	H_2O	SnO_2, SiO_2, Ti	50–150 °C				1006, 1008, and 1010
$VO(O^{i}Pr)_3$	H_2O[c]	SiO_2	150 °C				1010
$VO(O^{i}Pr)_3$	O_2	silica, titania	90–120 °C				1012–1014
$VO(O^{i}Pr)_3$	O_2[c]	SiO_2		150 °C			1010

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases						References
$VO(O^nPr)_3$	CH_3COOH	CNT, CNF	200 °C	200 °C					910, 1015, and 1016
CrO_x			Amorphous	α-Cr_2O_3					
$Cr(acac)_3$	air	alumina	200 °C						1033 and 1034
CrO_2Cl_2	MeOH	Al_2O_3[f]		330–465 °C					1030 and 1031
CrO_2Cl_2	MeOH	SiO_2, Si, TiO_2	330–375 °C	330–465 °C					817 and 1031
MnO_x			Amorphous	β-MnO_2	Mn_3O_4	α-MnO_2	ε-MnO_2	MnO	
$Mn(thd)_3$	O_3	SLG, Si		162–257 °C	235–331 °C				1039
$Mn(thd)_3$	O_3	NaCl, KCl, KBr				186 °C			1040
$Mn(thd)_3$	O_3	SiO_2, Al_2O_3, muscovite, MgO, SLG, Si		186 °C					1040
$Mn(thd)_3$	O_3	Al_2O_3					186 °C		1040
$Mn(thd)_3$	O_3	Al_2O_3[f]					186 °C		1041
$Mn(CpEt)_2$	H_2O	Si						150 °C	1037
FeO_x			Amorphous	α-Fe_2O_3	γ-Fe_2O_3	Fe_3O_4			
$FeCl_3$	H_2O	Pt		500 °C					1054
$FeCp_2$	O_2	Si, Al_2O_3		350–600 °C	350–600 °C[b]	350–600 °C[b]			1062
$FeCp_2$	O_2	TiO_2		400–500 °C		400 °C			821
$FeCp_2$	O_2	ZrO_2	367–534 °C						1063
$Fe(thd)_3$	O_3	SLG, Si, Al_2O_3		160–210 °C					1057 and 1058
$Fe(thd)_3$	O_3	MgO			160–210 °C				1058
$Fe(thd)_3$	O_3[i]	SLG, Al_2O_3		186 °C					1057
$Fe(O^tBu)_3$	H_2O	Al_2O_3		130–170 °C					1066
CoO_x			Amorphous	Co_3O_4	CoO				
CoI_2	O_2	SiO_2, MgO[f]		475–700 °C					1078
$Co(acac)_3$	O_2	silica		400 °C					1083
$Co(thd)_2$	O_3	Si, SLG, Corning 7059		114–450 °C	250–400 °C				1088 and 1089
$Co(thd)_3$	O_3	Al_2O_3[f], MgO[f], $SrTiO$[f]		138–283 °C					1090
NiO			Amorphous	Cubic					
$Ni(acac)_2$	H_2O	glass	190–310 °C						1107
$Ni(acac)_2$	CH_3COOH	glass	190–310 °C						1107
$Ni(acac)_2$	O_3+H_2O	glass		190–310 °C					1107
$Ni(dmg)_2$	H_2O	glass	190–310 °C						1107
$Ni(dmg)_2$	O_3	glass		190–310 °C					1107
$Ni(apo)_2$	O_3	glass		190–310 °C					1107
$Ni(dmamp)_2$	H_2O	H-Si	100–160 °C						1102 and 1103
$Ni(CpEt)_2$	O_3	H-Si	150 °C	200–300 °C					1100

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases		References
$Ni(Cp)_2$	O_3	H-Si, Ni, Pt, W, TiN		150–300 °C	1098–1100
$Ni(thd)_2$	H_2O	SiO_2, Si		205–275 °C	1108 and 1110
$Ni(thd)_2$	H_2O	MgO(100)[f], α-Al_2O_3(001)[f]		200–275 °C	1109
CuO_x			Amorphous	Cu_2O	
$Cu(^nBu_3P)_2(acac)$	O_2+H_2O	Ta/TaN		100–150 °C	1139
ZnO			Amorphous	Hexagonal	
Zn	H_2O	SLG		430 °C	1151
$ZnCl_2$	O_2	Sapphire(0001)[f], GaN[f]		450–550 °C	1153–1155
$ZnMe_2$	H_2O	Si, glass, GaN/Al_2O_3[f]		80–300 °C	1157, 1160–1162
$ZnMe_2$	O_2[c]	H-Si, Si		25–120 °C	1163 and 1164
$ZnEt_2$	H_2O	Si, H-Si, glass, SiO_2, Al_2O_3, TiO_2, SnO_2, ZnO, MgO, ZnTe, PS, CNT, ISM		23–400 °C	60, 282, 297, 318, 1149, 1157, 1160, 1161, 1186, 1190, 1192–1194, 1196, 1197, 1199, 1201, 1203, 1204, 1207–1210, 1213, 1215–1220, 1222, 1223, 1225, 1228, 1231–1233, 1235–1238, 1241, 1243, 1245, 1248, 1250, 1253, 1257, 1259, 1261–1265, and 2316–2318
$ZnEt_2$	H_2O	Sapphire(0001)		40–300 °C	1200, 1214, 1246, 1247, and 2316
$ZnEt_2$	H_2O	Sapphire(0001)[f]		25–450 °C	812, 1226, 1229, 1246, 1247, and 1251
$ZnEt_2$	H_2O	GaN/Al_2O_3[f], p-GaN[f], YSZ		180–300 °C	1162, 1202, 1225, 1242, 1244, and 1248

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases		References
$ZnEt_2$	H_2O	silica, Bi_2O_3	150–200 °C	150–200 °C	1149, 1211, and 1239
$ZnEt_2$	H_2O[c]	SiO_2		100–250 °C	1266
$ZnEt_2$	H_2O-O_2	Al_2O_3		180 °C	1186
$ZnEt_2$	H_2O[h]	Sapphire(0001)[f]		600 °C	1181
$ZnEt_2$	H_2O_2	TiO_2, SiO_2		50–100 °C	1223, 1268, and 1269
$ZnEt_2$	H_2O+NH_3[d]	Al_2O_3		200 °C	292
$ZnEt_2$	$H_2O+NH_3+O_2$[d]	Al_2O_3		200 °C	457
$ZnEt_2$	O_3	SiO_2, TiO_2		170–300 °C	1193, 1208, and 1209
$ZnEt_2$	O_2[c]	H-Si, ZnO, ITO, Glass		75–200 °C	1270–1272, 1274, and 2308
$ZnEt_2$	O_2[c]	PES	100 °C	150–250 °C	1275
$ZnEt_2$	N_2O	Al_2O_3, Sapphire(11-20)/ZnO buffer		300–600 °C	1276 and 1278
$ZnEt_2$	N_2O[c]	Al_2O_3		200 °C	458
$Zn(CH_3COO)_2$	H_2O	Sapphire(0001), SLG, Si, GaN, GaAs		280–400 °C	1160, 1279, 1282, 1284, and 1285
Ga_2O_3			Amorphous		
$Ga(acac)_2$	H_2O	SLG, Corning 7059	365–380 °C		1368
$Ga(acac)_2$	O_3	SLG, Corning 7059	350–375 °C		1368
$Ga_2(NMe_2)_6$	H_2O	Si	170–250 °C		1373
$[GaMe_2NH_2]_3$	O_2[c]	Si, SiO_2, Sapphire	50–250 °C		1370–1372
Y_2O_3			Amorphous	Cubic	
$Y(Cp)_3$	H_2O	Si		250–400 °C	1515
$Y(CpMe)_3$	H_2O	Si		200–400 °C	1515
$Y(CpEt)_3$	H_2O	Si		250 °C	1522
$Y(thd)_3$	O_2	Si, SLG, Corning 7059		425–600 °C	1524
$Y(thd)_3$	O_3	Si, glass		200–600 °C	1524–1526
$Y(thd)_3$	O_2[c]	Si		350 °C	1530 and 1531
$Y(^iPrAMD)_3$	H_2O	H-Si		280 °C	683

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases					References
ZrO_2			Amorphous	Cubic	Tetragonal	Monoclinic	Orthorhombic	
$ZrCl_4$	H_2O	SLG, SiO_2	300–500 °C					128, 1536, and 1555
$ZrCl_4$	H_2O	Ta_2O_5, $Ta_xNb_yO_z$	300 °C[b]	300 °C[b]	300 °C[b]			1563
$ZrCl_4$	H_2O	H-Si, SiO_2, Al_2O_3	160–450 °C	180–300 °C	300–600 °C	300–600 °C		1537, 1538, 1544, and 1545
$ZrCl_4$	H_2O	Si, SLG	160–325 °C		250–350 °C	300–350 °C		85, 1549, 1558, 1559, and 1561
$ZrCl_4$	H_2O	Si, H-Si, SiO_2, glass, Ta_2O_5, sapphire, MgO			230–500 °C	230–500 °C		140, 1539, 1540, 1548, 1554, 1556, and 1557
$ZrCl_4$	H_2O	$(Nb_{1-x}Ta_x)_2O_5$				325 °C		1539
$ZrCl_4$	H_2O	Ge(100)[f]		300 °C[b]	300 °C[b]			1547
$ZrCl_4$	H_2O_2	H-Si	160–450 °C	180–210 °C	210–600 °C	300–600 °C		1544 and 1545
ZrI_4	$H_2O+H_2O_2$	H-Si	272 °C	272–350 °C	272–350 °C			1569
ZrI_4	$H_2O+H_2O_2$	H-Si	250 °C	250–275 °C	325–500 °C	325–500 °C		1568
ZrI_4	$H_2O+H_2O_2$	H-Si		250–300 °C	325–500 °C	500 °C		1567
ZrI_4	$H_2O+H_2O_2$	H-Si, Pt		250 °C	250–500 °C	250–500 °C		1570
$Zr(^tBuO)_4$	H_2O	Si, SLG, Corning 7059	175 °C		200–300 °C	200–300 °C		861 and 1588
$Zr(^tBuO)_4$	H_2O	Al_2O_3, PC			120–160 °C			273
$Zr(^tBuO)_4$	H_2O[g]	PET	20 °C					1594
$Zr(^tBuO)_2(dmae)_2$	H_2O	SLG	190–240 °C	340 °C[b]	340 °C[b]	290–340 °C[b]		1599
$Zr(dmae)_4$	H_2O	SLG, borosilicate	190–240 °C	340 °C[b]	340 °C[b]	300–500 °C[b]		1587
$Zr(^iPrO)_2(dmae)_2$	H_2O	SLG, borosilicate	190–240 °C	340 °C[b]	340 °C[b]	300–500 °C[b]		1587
$Zr(thd)_4$	O_3	Si, SLG	275–300 °C			300–500 °C	300–500 °C	1571
$ZrCp_2Me_2$	H_2O	Si		200–300 °C[b] and 400–500 °C[b]		200–500 °C[b]	200–500 °C[b]	1574
$ZrCp_2Me_2$	O_3	Si, SLG	250–300 °C			300–500 °C	250–500 °C	1571
$ZrCp_2Me(OMe)$	O_3	Si			300 °C			1577
$ZrCp_2Cl_2$	O_3	Si, SLG	250–300 °C			250–500 °C	275–500 °C	1571
$ZrCp(NMe_2)_3$	O_3	Si	300 °C[b]	300 °C[b]	300 °C[b]			1578
$Zr(CpMe)_2Me(OMe)$	H_2O	Si	300 °C					2319
$Zr(CpMe)_2Me(O^tBu)$	H_2O	Si	300 °C					2319
$Zr(CpMe)_2Me_2$	H_2O	Si		350 °C[b]	350 °C[b]	350 °C	350 °C[b]	1579
$Zr(CpMe)_2Me_2$	O_3	SiO_2	300–400 °C	300–400 °C	300–400 °C	300–400 °C	300–400 °C[b]	1580
$Zr(CpMe)_2Me(OMe)$	H_2O	Si		350 °C[b]	350 °C[b]	350 °C	350 °C[b]	1579
$Zr(CpMe)_2Me(OMe)$	O_3	SiO_2	300–400 °C	300–400 °C	300–400 °C	300–400 °C	300–400 °C[b]	1580
$Zr(CpMe)_2Me(OMe)$	O_3	SiO_2		300–350 °C[b]	300–350 °C[b]	300–350 °C		1583 and 1584
$Zr(CpMe)(NMe_2)_3$	O_3	Si	300 °C[b]	300 °C[b]	300 °C[b]			1578
$Zr(CpEt)(NMe_2)_3$	O_3	Si	300 °C[b]	300 °C[b]	300 °C[b]			1578
$Zr[(Me_3Si)_2N]_2Cl_2$	H_2O	H-Si	250 °C					1620

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases				References
Zr(NMe$_2$)$_4$	H_2O	Si	50 °C	100–200 °C[b]	100–200 °C[b]		1601 and 1602
Zr(NEtMe)$_4$	H_2O	Si	300 °C				1609
Zr(NEtMe)$_4$	O_3	TiN		225–300 °C[b]	225–300 °C[b]	225–300 °C[b]	1612 and 1613
Zr(NEtMe)$_4$	O_3	Si	275 °C				1614
Zr(NEtMe)$_4$	O_2[c]	H-Si		100–250 °C[b]	100–280 °C[b]		1617 and 1619
Zr(NEt$_2$)$_4$	O_2	Si, Ge	300 °C				1607
Zr(MeAMD)$_4$	H_2O	Si	300 °C				1609
Nb_2O_5			Amorphous				
Nb(OEt)$_5$	H_2O	SLG	215–275 °C				1626
RuO_2			Amorphous	Rutile			
Ru(CpEt)$_2$	O_2	Si, SiO_2, TiN		265–270 °C			1652, 1654, and 1692
Ru(CpEt)$_2$	O_2	Ta_2O_5		300–350 °C			1687
Ru(thd)$_2$(cod)	O_2	Si		290 °C			1693
Rh_2O_3			Amorphous				
Rh(acac)$_3$	O_3	Si, SLG	160–180 °C				1695
In_2O_3			Amorphous	Cubic			
$InCl_3$	H_2O	SLG		400–500 °C			1753
$InCl_3$	$H_2O + H_2O_2$	Corning 7059		300–500 °C			1757
InCp	O_3	Si, glass, Al_2O_3		200–450 °C			1759 and 1760
In(acac)$_3$	H_2O	Si	175 °C	200–225 °C			1761
In(acac)$_3$	O_3	Si	200 °C				1761
SnO_2			Amorphous	Tetragonal			
$SnCl_4$	H_2O	Ga_2O_3	350 °C				1812
$SnCl_4$	H_2O	SiO_2, Graphene	180–200 °C	300–400 °C			747, 1802, and 1810
$SnCl_4$	H_2O	Si		500 °C			1369
$SnCl_4$	H_2O	ZnS, CuO_x	350 °C				1808 and 1811
$SnCl_4$	H_2O	SLG, Corning 7059		300–600 °C			1796, 1799, and 1801
$SnCl_4$	H_2O	Al_2O_3[f]		400–600 °C			1804 and 1805
$SnCl_4$	H_2O+O_3 (hexane cat.)	SLG		500 °C			1799
$SnCl_4$	H_2O_2	Si	150–430 °C				1816
$SnCl_4$	H_2O_2	Al_2O_3[f]		600–700 °C			1804 and 1813
SnI_4	H_2O_2	Al_2O_3[f]		600 °C			1804
SnI_4	O_2	SiO_2		400–750 °C			1820
SnI_4	O_2	Al_2O_3[f]		400–750 °C			1804, 1813, 1820, and 1821

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases					References
$Sn(NMe_2)_4$	H_2O_2	Si, glass	50–300 °C					1826
Sn(tbba)	H_2O_2	SiN, glass		120 °C				1828
$Sn^nBu_2(OAc)_2$	O_2[c]	SiO_2, TiO_2[k]		200–400 °C				1822, 1823, and 1825
Sb_2O_5			Amorphous					
$Sb(NMe_2)_3$	O_3	Si	120 °C					1829
$BaO(Ba(OH)_2)$[e]			Amorphous $Ba(OH)_2$	Monoclinic $Ba(OH)_2$	Orthorhombic $Ba(OH)_2$			
$Ba(Cp^tBu_3)_2$	H_2O	Al_2O_3	240 °C	290–340 °C	340 °C[b]			1830
La_2O_3			Amorphous	Cubic	Hexagonal	Hexagonal $La(OH)_3$	Monoclinic La(O)OH	
$La(thd)_3$	O_3	SLG, Si	200–300 °C	300–450 °C	400–450 °C			44, 1088, and 1577
$La(thd)_3$	O_3	Corning 7059	200–300 °C	300–350 °C	400–450 °C			1088
$La(thd)_3$	O_3	Corning 7059	225–300 °C	300–425 °C				1833
$La(thd)_3$	O_3	SiO_2[l]		350–500 °C	350–500 °C			1835
$La(thd)_3$	O_3	SiO_2[m]		350 °C		350 °C	350 °C	1835
$La(thd)_3$	H_2O	H-Si, TiN	230–350 °C					237, 1836, and 1837
$La[N(SiMe_3)_2]_3$	H_2O	H-Si	250 °C	250 °C				1838
$La[N(SiMe_3)_2]_3$	H_2O	H-Si, Si, borosilicate	200–400 °C					1840
$La[N(SiMe_3)_2]_3$	H_2O	Si			300 °C			1841
$La(Cp)_3$	H_2O	SiO_2[l]		260 °C	260 °C			1835
$La(Cp)_3$	H_2O	SiO_2[m]				260 °C		1835
$La(CpEt)_3$	O_2[c]	Si	350 °C		350–400 °C			1847
$La(Cp^iPr)_3$	O_3	Si	200 °C			200 °C		1852
$La(Cp^iPr)_3$	O_3	Ge	200 °C					1852
$La(^iPrfAMD)_3$	O_2	Si, Ge	300 °C					1607
CeO_2			Amorphous	Cubic				
$Ce(thd)_4$	O_3	SLG, Si		175–250 °C				1859
$Ce(thd)_3phen$	O_3	SLG, Si		225–275 °C				1859
PrO_x			Amorphous					
$Pr[N(SiMe_3)_2]_3$	H_2O	borosilicate, Si	200–400 °C					1861 and 1862
$Pr(^iPrAMD)_3$	H_2O	H-Si	200–315 °C					1863
Nd_2O_3			Amorphous	Cubic				
$Nd(thd)_3$	O_3	Si, SLG	200–250 °C	290–325 °C				1864 and 1865

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases					References
Sm_2O_3			Amorphous	Cubic				
$Sm(thd)_3$	O_3	Si		300 °C				1864
Eu_2O_3			Amorphous	Cubic				
$Eu(thd)_3$	O_3	Si		300 °C				1864
Gd_2O_3			Amorphous	Cubic	Monoclinic			
$Gd(thd)_3$	O_3	Si		300 °C				1864 and 1866
$Gd(CpMe)_3$	H_2O	Si		150–250 °C				1866
$Gd(mmp)_3$-tetraglyme	H_2O	Si		200–250 °C				1867
$Gd[OCMe_2CHMe_2]_3$	H_2O	Si, H-Si, SLG, Al_2O_3	350 °C	300–400 °C	250–300 °C			1868 and 1869
$Gd[(N^iPr)_2CN(CH_3)_2]_3$	H_2O	Si	200 °C	160–250 °C				1871 and 1873
Dy_2O_3			Amorphous	Cubic				
$Dy(thd)_3$	O_3	Si		300 °C				1864
Ho_2O_3			Amorphous	Cubic				
$Ho(thd)_3$	O_3	Si		300 °C				1864
Er_2O_3			Amorphous	Cubic				
$Er(thd)_3$	O_3	Si		300–350 °C				1584 and 1864
$Er(thd)_3$	O_2[c]	Si		350 °C				1530 and 1531
$Er(CpMe)_3$	H_2O	Si, SLG		250–300 °C				1875
$Er(^tBuAMD)_3$	O_3	Si	250 °C	300 °C				1876
Tm_2O_3			Amorphous	Cubic				
$Tm(thd)_3$	O_3	Si		300 °C				1864
Yb_2O_3			Amorphous	Cubic				
$Yb(thd)_3$	O_3	Si, glass		300–350 °C				1877
Lu_2O_3			Amorphous					
$Lu[Cp(SiMe_3)]_2Cl$	H_2O	SiO_2	360–370 °C					1879 and 1880
HfO_2			Amorphous	Cubic	Tetragonal	Monoclinic	Orthorhombic	
$HfCl_4$	H_2O	H-Si	300 °C					1897 and 1947
$HfCl_4$	H_2O	Si, H-Si, SiO_2, GaAs, TiN, Pt, Ir, Ru	200–350 °C			250–600 °C		1918, 1919, 1922, 1930, 1931, 1970, and 2320
$HfCl_4$	H_2O	H-Si, Ge, SiO_2	226–300 °C	226–300 °C		300–750 °C		1915, 1935, and 1974

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases					References
$HfCl_4$	H_2O	H-Si, SiO_2	300 °C	600 °C		300–600 °C		1901
$HfCl_4$	H_2O	Si	600–880 °C	880–940 °C	500–600 °C[b]	500–940 °C	500–600 °C[b]	1888
$HfCl_4$	H_2O	Si, H-Si, SiO_2, HfO_2	225–300 °C	300–750 °C[b]	300–750 °C[b]	300–940 °C	300–750 °C[b]	1554, 1887, 1906, 1918, and 2321
$HfCl_4$	H_2O	Si		300 °C[b]	300 °C[b]	300 °C	300 °C[b]	1559
$HfCl_4$	H_2O	Si, SiO_2, sapphire, MgO		600 °C[b]	600 °C[b]	600 °C	600 °C[b]	1556 and 1557
$HfCl_4$	H_2O	H-Si			300 °C[b]	300 °C	300 °C[b]	1566
$HfCl_4$	H_2O	Si, H-Si			300 °C	300 °C		189, 1561, and 1959
$HfCl_4$	H_2O	Si, H-Si, glass, Pt, TiN				300–350 °C		1883, 1948, and 1971
$HfCl_4$	H_2O	Ge				375 °C	375 °C	1917
$HfCl_4$	H_2O	Pt	350 °C[b]			350 °C[b]	350 °C[b]	1970
$HfCl_4$	H_2O	Si, SiO_2, glass	300–500 °C		500 °C[b]	300–600 °C	500 °C[b]	1558, 1884, and 1932
$HfCl_4$	H_2O	SiO_2	300 °C[b]		300 °C	300 °C		1555
$HfCl_4$	O_3	H-Si	300 °C			300 °C	300 °C	1897
$HfCl_4$	O_3	Ge	375 °C					1917
HfI_4	H_2O	H-Si, SiO_2	300 °C	600 °C		300–600 °C		1566 and 1901
HfI_4	H_2O	poly-Si	225 °C	400–500 °C[b]	400–500 °C[b]	300–500 °C	400–500 °C[b]	1981
HfI_4	H_2O	MgO				400–500 °C		1981
HfI_4	H_2O_2	poly-Si	225 °C	400–500 °C[b]	400–500 °C[b]	300–500 °C	400–500 °C[b]	1981
HfI_4	H_2O_2	MgO				400–500 °C		1981
HfI_4	O_2	H-Si				570–750 °C		1984
$HfCp_2Me_2$	H_2O	Si				300–425 °C		1989 and 1990
$HfCp_2Me_2$	O_3	Si				300–425 °C		1989 and 1990
$Hf(CpMe)_2Me_2$	H_2O	Si		350 °C[b]	350 °C[b]	350 °C	350 °C[b]	1579
$Hf(CpMe)_2Me_2$	O_3	Si				300–500 °C		1991
$Hf(CpMe)_2(OMe)Me$	H_2O	Si		350 °C[b]	350 °C[b]	350 °C	350 °C[b]	1579
$Hf(CpMe)_2(OMe)Me$	O_3	Si, TiN		350 °C[b]	350 °C[b]	300–500 °C[b]		1521 and 1991
$Hf(CpMe)_2(O^iPr)Me$	H_2O	Si	250–440 °C					1993
$Hf(CpMe)_2(mmp)Me$	H_2O	Si	250–440 °C					1993
$HfCp(NMe_2)_3$	O_3	Si	250 °C	300–350 °C[b]	300–350 °C[b]	300–350 °C		1994
$Hf(CpMe)(NMe_2)_3$	O_3	Si	250 °C	300–350 °C[b]	300–350 °C[b]	300–350 °C		1994
$Hf(^tBuO)_4$	O_2	H-Si	350–480 °C					2322
$Hf(mmp)_4$	O_2	Si				550 °C		684
$Hf(mmp)_4$	H_2O	borosilicate, Si	225–325 °C			325–425 °C		1867 and 2004
$Hf(mmp)_4$	H_2O_2	borosilicate, Si	300 °C			325–425 °C		2004
$Hf(^tBuO)_2(mmp)_2$	H_2O	borosilicate, SiO_2	275–300 °C		360 °C[b]	300–400 °C		2001
$Hf(OCEtMe_2)_4$	H_2O	H-Si	250–350 °C					2005
$Hf(OCEtMe_2)_4$	O_2[c]	H-Si			250 °C	250 °C		2006

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases					References
$Hf(O^iPr)_4$	O_2	H-Si				300 °C		1995
$Hf(O^tBu)_4$	HCOOH	Si, $SrTiO_3$, $LaAlO_3$, MgO, wool, cellulose, latex, CNT	100–125 °C					908
$Hf(O^tBu)_4$	CH_3COOH	Si, $SrTiO_3$, $LaAlO_3$, MgO, wool, cellulose, latex, CNT	175–275 °C					908–911
$Hf(O^tBu)(NEtMe)_3$	O_3	Si	300 °C			300 °C		2002
$Hf(O^tBu)(NEtMe)_3$	O_3	TiO_2(rutile)	250 °C		250 °C			355
$Hf(O^tBu)(NEtMe)_3$	O_3	TiO_2(anatase)				250 °C		355
$Hf(NMe_2)_4$	H_2O	Si	50–150 °C			200–300 °C		1601 and 1602
$Hf(NMe_2)_4$	H_2O	CNC				150 °C		362
$Hf(NMe_2)_4$	H_2O	H-Si, SiO_2	200–400 °C			205–400 °C[b]		250, 2011, 2023, and 2320
$Hf(NMe_2)_4$	H_2O	Si	250 °C		350 °C[b]	350 °C	350 °C[b]	2010 and 2016
$Hf(NMe_2)_4$	H_2O	Ge	225 °C					2021
$Hf(NMe_2)_4$	O_2[c]	Si, H-Si	250 °C			280 °C		1619 and 2041
$Hf(NEtMe)_4$	H_2O	Si	250 °C			250 °C[b]		2023
$Hf(NEtMe)_4$	H_2O	borosilicate, ITO, Si, Al_2O_3	150–200 °C		200–300 °C[b]	200–300 °C	200–300 °C[b]	1601, 2071, 2072, and 2082
$Hf(NEtMe)_4$	H_2O	H-Si	200–250 °C					2023 and 2081
$Hf(NEtMe)_4$	H_2O	InP			350 °C	350 °C		2094
$Hf(NEtMe)_4$	D_2O	H-Si, Si_xN_y	60–100 °C					2074 and 2323
$Hf(NEtMe)_4$	O_3	H-Si, SiO_2	180–275 °C		350 °C[b]		350 °C[b]	2102
$Hf(NEtMe)_4$	O_2[c]	Si	290 °C					963
$Hf(NEt_2)_4$	H_2O	Si	150–250 °C					1522 and 1932
$Hf(NEt_2)_4$	O_3	Si, H-Si	200–275 °C					472, 473, and 2310
$Hf(NEt_2)_4$	O_2[c]	H-Si			250 °C	250 °C		2006
$Hf(ONEt_2)_4$	H_2O	borosilicate, Si	300 °C			300 °C[b]		2007
$Hf(NO_3)_4$	H_2O	H-Si	180 °C					2114
Ta_2O_5			Amorphous	Hexagonal	Tetragonal	Orthorhombic		
TaF_5	H_2O	H-Si	400–450 °C	400–450 °C		400 °C		2136
$TaCl_5$	H_2O	Glass, Si	300–500 °C					392, 1291, and 1883
$TaCl_5$	H_2O	Glass, Si	80–160 °C	400–500 °C	400–500 °C			2130
$TaCl_5$	H_2O	Corning 7059	300–325 °C	300–350 °C		400 °C		2133
TaI_5	H_2O	H-Si	250–400 °C	350–400 °C		350–400 °C		2137
TaI_5	H_2O	SiO_2	250–400 °C					2137
$Ta(OEt)_5$	H_2O	Si, SLG, SS, $Nb_xZr_yO_z$	170–325 °C					123, 1563, 2139, 2145, and 2146

TABLE III. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases			References
$Ta(OEt)_5$	H_2O[g]	Si	170–400 °C			2142
$Ta(OEt)_5$	$TaCl_5$	Corning 7059	275–450 °C			2135
$Ta(OEt)_5$	O_2[g]	Si, SiO_2	190–285 °C			2149
$Ta(OEt)_5$	O_2[c]	Si, borosilicate, Pt	150–250 °C			2148
$Ta(OEt)_5$	O_2[c] + H_2O	Si, borosilicate, Pt	150–250 °C			2148
$Ta(OEt)_4(dmae)$	O_3	N-Si	400 °C			2150
$Ta(NMe_2)_5$	H_2O	H-Si	150–350 °C			918, 2152, and 2153
$Ta(NMe_2)_5$	O_2[c]	H-Si, Si, SiO_2	100–350 °C			918, 1650, 2152, and 2155
$Ta(NEt)(NEt_2)_3$	H_2O	Si, SiO_2	50–350 °C			2156
$Ta(NEt_2)_5$	H_2O	Si, SiO_2	50–250 °C			2156
$Ta(N^tBu)(^tBu_2pz)_3$	O_3	Si	300–450 °C			2157
$Ta(N^tBu)(^iPrAMD)_2(NMe_2)$	H_2O	Si	275–350 °C			2158
WO_x			Amorphous	Monoclinic		
$WF_6 + WO_3$	H_2O	sapphire		200 °C		2220
$W_2(NMe_2)_6$	H_2O	Si	160–200 °C			2223
IrO_2			Amorphous	Tetragonal		
$Ir(acac)_3$	O_3	Si, SLG, Al_2O_3		165–200 °C		2252
Ir(CpEt)(cod)	O_2	SiO_2		290 °C		2248
PtO_x			Amorphous			
$Pt(acac)_2$	O_3	Si, SLG, Al_2O_3	120–130 °C			2273
$Pt(CpMe)Me_3$	O_2[c]	Si, SiO_2	300 °C			2260
PbO_2			Amorphous	Tetragonal	Orthorhombic	
$Pb(thd)_2$	O_3	Si		150–300 °C[b]	150–300 °C[b]	2275
$PbPh_4$	O_3	Si		185–400 °C[b]	185–400 °C[b]	2275

[a]H-Si, HF-etched Si; MWCNT, multi-walled carbon nanotube; CNS, carbon nanosheet; CNC, carbon nanocoil; CNT, carbon nanotube; SLG, soda lime glass; SS, stainless steel; PE, polyethylene; PET, polyethylene terephtalate; PMMA, polymethylmethacrylate; PP, polypropylene; PS, polystyrene; PVC, polyvinylchloride; PVP, polyvinylpyrrolidone; PC, polycarbonate; PES, polyethersulfone; PTFE, polytetrafluoroethylene; ISM, Inner shell membrane from hen's egg; YSZ, Yttria-stabilized zirconia; Precursors dash-separated, pulsed separately; Two precursors connected with +, fed in the same pulse.

[b]Ambiguous interpretation due to reflection overlap or weak intensity.

[c]Plasma.

[d]Coating-head type deposition.

[e]Oxide was formed after 1 day delay after deposition.

[f]Epitaxy.

[g]Photo-assisted deposition.

[h]Electric field enhanced deposition.

[i]Magnetic field enhanced deposition.

[j]XRD was measured after depositing 10-nm HfO_2 film on top at the same temperature.

[k]Rutile single crystals of various orientations.

[l]Film was capped with ALD-Al_2O_3 *in situ*.

[m]Bare film without ALD-Al_2O_3 *in situ* capping.

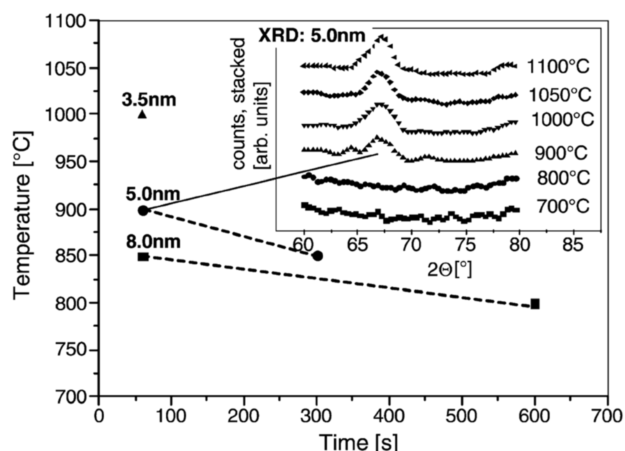

FIG. 9. First appearance of long-range order in Al_2O_3 films, as detected by XRD. The inset shows XRD spectra for 5 nm Al_2O_3 layers annealed for 60 s at different temperatures. Reprinted with permission from S. Jakschik, U. Schroeder, T. Hecht, M. Gutsche, H. Seidl, and J. W. Bartha, Thin Solid Films **425**, 216 (2003). Copyright 2003 Elsevier.

1100 °C,[2324] while on ZnO nanotubes crystalline alumina was obtained at 800 °C.[268]

ALD of alumina films that are crystalline already in the as-deposited state requires growth temperatures above 600 °C. That is possible when $AlCl_3$ is employed as the precursor. Dueñas *et al.*[2325] grew films on HF-treated silicon and obtained crystalline material in thick films (30–100 nm) at $600-800$ °C, while thin films (3–9 nm) did not show crystallinity at any temperature. The structure of the aluminum oxide could not be unambiguously detected since the x-ray diffraction (XRD) and reflection high-energy electron diffraction (RHEED) reflections could be attributed either to cubic $\gamma-Al_2O_3$ or tetragonal $\delta-Al_2O_3$. Oya *et al.*[100,101] have succeeded in epitaxial growth of $\alpha-Al_2O_3$ on sapphire at 600 °C and on single crystal Nb at $450-500$ °C. The process employed was $AlCl_3+O_2$, and the reason for the low temperature in the case of the Nb substrate was explained by catalytic effect of the substrate.

3. Case: Zirconium oxide

Zirconium oxide (ZrO_2) has been studied as possible high-κ oxide material for future microelectronic circuits owing to its high permittivity. ALD ZrO_2 is already used in DRAM capacitors in ZAZ (ZrO_2-Al_2O_3-ZrO_2) multilayer structures. ZrO_2 has different crystal structures, from which the monoclinic phase has permittivity value of 23, while tetragonal and cubic phases show nearly twice as high values of 42 and 40.[2326] ZrO_2 is a good oxide ion conductor which has been utilized, for example, in fuel cells as a solid electrolyte.[1517]

For bulk ZrO_2, monoclinic phase is the most stable polymorph at temperatures below 1150 °C, whereas tetragonal and cubic phases are found at high temperatures.[2327,2328] In addition, orthorhombic phases have been characterized at elevated pressures.[2328] In thin films and particles at submicrometer scale, additional parameters such as surface energy and strain step in, however, and the phase behavior differs from the bulk.

In submicron architectures, such as nanoparticles and thin films, the ratio of surface area to volume is high. Therefore, the surface-related parameters, such as surface energy, start to affect the overall energy and further the phase transition conditions. The effect of surface energy to the phase behavior has been formulated by Garvie.[2327,2329,2330] According to Garvie, the low surface energy stabilizes the tetragonal phase and decreases the monoclinic to tetragonal phase transition temperature when the feature size of ZrO_2 is decreased. Cubic phase has even lower surface energy, making it stable in very small particles.[2331] Both tetragonal and cubic nanoparticles have been synthesized.[2332–2339] In thin film deposition, especially in ALD, physical dimensions of the crystallites can be controlled by laminating ZrO_2 with an other, often amorphous layer such as Al_2O_3, which interrupts the crystallite growth.[1539]

On the other hand, there are reports on syntheses of monoclinic[2340,2341] and orthorhombic[2342,2343] ZrO_2 nanoparticles as well. Besides the surface energy, there appear to be also other factors contributing to the phase, such as crystallite morphology, oxygen vacancies in the lattice, and strain.[2334,2335,2338,2339,2341,2344–2346] Oxygen vacancies can be generated by doping cations with an oxidation state of two or three into the crystal lattice of ZrO_2. This method has been widely applied for stabilizing cubic and tetragonal phases of ZrO_2. Typical stabilizing dopants are yttrium, magnesium, and calcium.[2347]

Atomic layer deposited zirconium oxides, and the closely related hafnium oxides, have been doped with cations to stabilize their high permittivity cubic and tetragonal phases. With ALD, the doping level can be straightforwardly controlled by introducing the doping oxide cycles in between the matrix oxide cycling. Through the alternate cycling, accurate and reproducible dopant concentration is achieved. In a paper by Putkonen *et al.*, ZrO_2 was prepared from Cp- and thd-complexes together with ozone, while yttrium doping was enabled by $Y(thd)_3$.[2347] They found phase-pure cubic ZrO_2 over wide range of dopant concentration. Niinistö *et al.* stabilized amorphous ZrO_2 by strontium doping in the as-deposited state, and the films were crystallized in cubic/tetragonal phase upon post-deposition annealing.[14] In the same paper, HfO_2 grown from $Hf(NMe_2)_4$-O_3 was doped with yttrium using $Y(CpMe)_3$. The films were amorphous as-deposited and crystallized into cubic/tetragonal phase when annealed above 400 °C. Similar phase behavior was found when novel $HfCp(NMe_2)_3$ and Hf(CpMe)$(NMe_2)_3$ were used as a hafnium source, as reported in a separate paper by the same authors.[1994] Cubic HfO_2 can be also stabilized by doping with Er and Dy or Sc.[684,2348]

In ALD, the most widely studied precursor system for ZrO_2 is $ZrCl_4$ and water.[1536] Besides chloride, zirconium tetraiodide (ZrI_4) has been utilized as another metal halide precursor for ZrO_2 ALD.[1567] With ZrI_4, hydrogen peroxide (H_2O_2) was also applied as the oxygen source.[1567] Zirconium alkoxides,[1588] aminoalkoxides,[1587] alkylamides,[1601] and cyclopentadienyl (Cp) complexes[1571] as well as complexes of their combinations have been utilized as metal sources. In addition to water and hydrogen peroxide, ozone has been used as a source for oxygen.[1571,1610] Several ALD processes

exploiting plasma have been also reported.[1595,1596] Processes reported for ZrO_2 ALD are collected in Table I.

ALD of zirconium oxide starts as amorphous phase regardless of precursor system utilized.[1536,1544,1545,1549,1569,1587,1599,1620] After a certain thickness is deposited, crystalline ZrO_2 is observed. The phase evolution is not straightforward, and also the overlap in diffraction patterns complicates the interpretation. The literature data on ALD-ZrO_2 crystallization are collected in Table III.

Nucleation of the film is anyhow different on oxide-terminated and HF-etched H-terminated silicon. This is clearly seen in Figure 10. On oxidized silicon, ZrO_2 layer is smooth yet clearly polycrystalline. On HF-etched silicon, the nucleation density has been lower and the film shows more or less separate crystallites and is much rougher, because the nuclei have had more time to grow independently before coalescing. There are amorphous regions in between the crystallites and also an amorphous SiO_x film at the film-substrate interface.[1540] A special case where a strong effect of substrate has been seen is local epitaxy of ZrO_2 on germanium substrates at 300 °C.[1547]

The thickness required for crystalline ZrO_2 to form depends on the precursor combination and deposition temperature. For the $ZrCl_4$–H_2O process, the film thickness needed for crystallization is as high as 100 nm at 185 °C, and it monotonically decreases as deposition temperature increases, being around 1 nm at 600 °C.[1544] The crystallization threshold thickness is only slightly higher at the low deposition temperatures, when hydrogen peroxide is used in place of water. The threshold thickness as a function of temperature is illustrated in Figure 11. Kukli *et al.*[1545] suggested

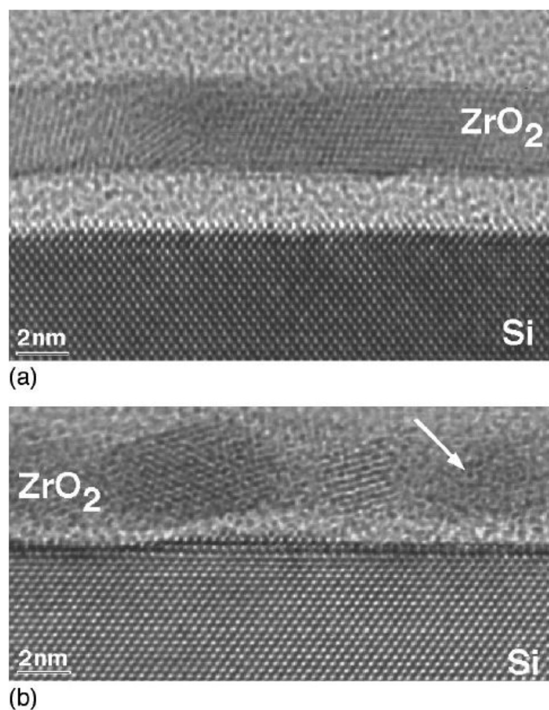

FIG. 10. HR-TEM images of thin ZrO_2 layers deposited on (a) oxidized and (b) HF-etched silicon. Arrow indicates amorphous region.[1540] Reprinted with permission from Appl. Phys. Lett. **76**, 436 (2000). Copyright 2000 American Institute of Physics.

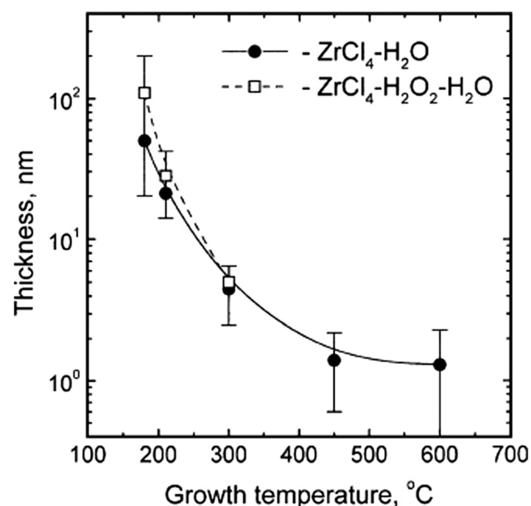

FIG. 11. Minimum film thickness required for crystalline ZrO_2 phases to be observed by RHEED as a function of temperature. Films were deposited on Si(100). Reprinted with permission from J. Aarik, A. Aidla, H. Mändar, T. Uustare, and V. Sammelselg, Thin Solid Films **408**, 97 (2002). Copyright 2002 Elsevier.

that hydrogen peroxide has its effect on the silicon-film interface, but shows similar chemistry as water as the film growth proceeds further. Hydrogen peroxide most probably oxidizes the substrate in the early stages of the film growth more effectively than water, and forms a thicker amorphous interfacial layer.

With the ZrI_4–H_2O_2/H_2O process, the thickness needed for film crystallization is higher, being 20 nm at 272 °C,[1569] while with $ZrCl_4$, crystallization took place for less than 10 nm thick film at the same temperature.[1544] Films deposited from cyclopentadienyl-based precursors and ozone start to grow as amorphous as well, since only weak diffraction if any was observed in the patterns measured from very thin films.[1576,1578,1580] Films deposited from alkoxides, alkyl- and silylamides, and aminoalkoxides are mainly amorphous throughout.[1587,1588,1599,1602,1620,2319] Reason for the amorphicity can be impurities, which originate from the thermally unstable precursors. Recently, alkylamidinate complex showed good thermal stability and the films deposited from it and water yielded amorphous films.[1609] The films were just 3.9 nm thick, which is below the common crystallization limit, however.

Whether the crystals grow from new nuclei formed on the already-deposited amorphous layer, or the original amorphous layer is crystallized as well, is not extensively studied. However, there are reports[1540,1554,1558,2349] presenting cross-sectional transmission electron microscopy (TEM) images of few-nanometer thick films showing through-film crystallites (Figure 10), supporting the latter option. The apparent amorphicity in very thin films can originate also from crystallite sizes below detection limit of the applied methods.

The first phase appearing in the zirconia films, as the film thickness increases above the crystallization threshold, is typically cubic or tetragonal. In films deposited from $ZrCl_4$, especially at low deposition temperature, the first phase appearing is cubic.[1544] Evidently, the cubic phase appears as a very thin layer and is transformed to the

tetragonal form as the film thickness increases, since reflections from the cubic phase are not visible together with tetragonal phase.[1544] Due to the similarity of the tetragonal and cubic phases (see, for example, ICSD (inorganic crystal structure database) Cards 17-0923 and 27-0997), the interpretation between the two is challenging, however.

In the films grown from ZrI_4 and hydrogen peroxide, the cubic phase is found for much higher thicknesses: 125-nm film grown at 275 °C shows a strong (200) reflection from the cubic phase.[1567] Kukli *et al.* suggested several reasons for the phase behavior different from the $ZrCl_4$ process: the chemical mechanism of the crystal growth, kinetics of the phase formation, effect of grain size, and mobility of surface species affecting the nucleation density. It was reported by the same authors that neither the ZrI_4 pulse nor the peroxide-water pulse did completely saturate, as evidenced by quartz crystal microbalance (QCM) data. Also the film mass decreased when the purge period after the ZrI_4 pulse was elongated, which was concluded to be caused by iodine desorption from the film surface.[1567]

The QCM observations from the $ZrCl_4$–water process were somewhat different. First, the elongation of the purge after the metal precursor did not affect the film mass. On the other hand, the $ZrCl_4$ pulse did not fully saturate even if the pulse was elongated to the scale of tens of seconds. The mass gain was nevertheless diminishingly small after a few seconds. Second, reaction between the film surface and water was found to saturate.[1544] The film growth mechanisms are obviously different when $ZrCl_4$ and ZrI_4 are applied as precursors, however.

The orientation of the cubic crystallites is much more evident when deposited from ZrI_4 than in the $ZrCl_4$-based films. This is probably because the crystallites can grow larger in the ZrI_4 than in the $ZrCl_4$ process. The first signs of the cubic phase, as observed by RHEED, refer to randomly oriented crystallites.[1569] This is also supported by XRD studies of thin films.[1567] As the thickness of the cubic layer increases, the (200) reflection becomes the most intense one, indicating the [100] orientation of cubic crystallites.[1567–1570]

The films deposited from Cp_2ZrMe_2 and ozone show as their first trace of crystallization a weak diffraction slightly above 2θ of 30°, which was interpreted as cubic or orthorhombic (111) reflection.[1576] In the same study, films were deposited also from Cp_2ZrCl_2 together with ozone, and similar phase behavior was found. Very thin films deposited from $(MeCp)_2ZrMe_2$ and $(MeCp)_2Zr(OMe)Me$ and ozone as oxygen source showed reflections from tetragonal and/or cubic phase as the first signs of crystalline formation.[1580] Similar phase evolution was found when ozone was replaced by water as an oxygen source.[1579] Kukli *et al.* reported recently that novel cyclopentadienyl-alkylamido complexes $CpZr(NMe_2)_3$, $(CpMe)Zr(NMe_2)_3$, and $(CpEt)Zr(NMe_2)_3$ yielded tetragonal zirconium oxide when utilized together with ozone.[1578] The thickness range was 5.2-7.8 nm. The films deposited from $Zr(thd)_4$ and ozone showed weak reflections in XRD, which were addressed to the monoclinic or orthorhombic phases.[1571]

As the film thickness increases, the tetragonal phase starts to appear. In the films deposited from $ZrCl_4$ and water, the tetragonal phase dominates at thicknesses around 50 nm.[1544,1545,1548,1557] At moderate deposition temperatures (around 300 °C), the first traces of the monoclinic phase are found as films reach thickness around 100 nm.[1544,1557] The monoclinic and tetragonal phases are found in the films simultaneously over a wide range of thicknesses. For the films deposited from Cp_2ZrMe_2, the monoclinic and orthorhombic phases were the most visible, similarly when deposited with water or ozone.[1571,1574] When $(MeCp)_2ZrMe_2$ and $(MeCp)_2Zr(OMe)Me$ were used together with water or ozone, cubic and monoclinic phases were reported to start showing as these films had grown thicker.[1579,1580] The (111) reflection of the cubic phase at 30.2° was alternatively addressed to the orthorhombic phase.[1579,1580]

In a paper by Tkachev *et al.*,[1557] it is concluded from findings with Raman spectroscopy that part of the originally formed tetragonal phase transformed to monoclinic, since the absolute amount of the tetragonal phase decreased upon appearance of the monoclinic phase. All of the tetragonal phase was not, however, transformed but remained underneath the monoclinic layer. Non-equilibrium crystal growth conditions have been suggested to be responsible for stabilizing the tetragonal phase in ALD ZrO_2.[1548] In addition, there were small crystallites, which had been formed at the early stage of film deposition. The small size together with intercrystallite strain could stabilize the tetragonal phase in these crystallites and inhibit the transformation to monoclinic. When ZrO_2 is deposited in nanolaminates in the thickness of about 10 nm, the tetragonal phase is the only one observed.[85,1539] The peaks are reported to be relatively wide indicating small crystallite size. The tetragonal crystallites in the nanolaminate structure are randomly oriented, since the (111) reflection is the strongest one similar to the powder pattern (JCPDS (joint committee on powder diffraction standards) Card 27-0997).[85,1539]

The crystallites of the monoclinic phase in thick films deposited from $ZrCl_4$ seem to prefer [001] orientation, since the (002) reflection becomes the most intense as the film gets thicker, while ($\bar{1}$11), the strongest peak in powder pattern (JCPDS Card 36-0420) diminishes.[1544,1557] The monoclinic phase, when appearing in ZrI_4-based films, shows random orientation.[1568] The studied films were, however, in the thickness range where the cubic and tetragonal phases are dominant and the monoclinic phase was just starting to form. The films deposited from some precursors containing cyclopentadienyl ligands show monoclinic ($\bar{1}$11) as their most intense reflection in thickness range over 100 nm, when deposited at 350 °C or higher.[1571]

Interpretation of some reflections of ZrO_2 phases is complicated due to overlap of diffraction lines. The presence of the orthorhombic phase in ALD ZrO_2 films has been suggested by Putkonen *et al.*,[1571,1574] Niinistö *et al.*,[1576,1580] and Dezelah *et al.*,[1579] while several papers[1544,1545,1557] do not suggest the appearance of that phase. The orthorhombic phase is indeed a high pressure phase found only in high pressure as bulk material.[2327,2328] There are, however, reports on nanostructured and thin film ZrO_2 appearing in the orthorhombic phase also in ambient conditions.[2342,2343,2350] Therefore neither of the interpretations cannot be ruled out.

4. Case: Zinc oxide

Zinc oxide (ZnO) is a versatile material owing to its beneficial electrical, physical, and chemical properties. ZnO is a semiconductor with a wide band gap of 3.37 eV and large exciton binding energy, 60 meV, making it a promising optoelectronic material. It can be applied in ultraviolet (UV) light emitting diodes.[2351] It is also an efficient gas sensor material.[2352] Hexagonal ZnO as a piezoelectric material can be utilized, for example, as a force sensor.[2353] Aluminum-doped ZnO is a widely studied transparent conducting oxide.

The crystal structures occurring for ZnO are cubic zinc blende, cubic rock salt, and hexagonal wurtzite. The zinc blende phase is stable only on cubic substrates, and the rock salt structure occurs only at high pressures.[2351] The hexagonal phase is stable at ambient conditions. Crystalline ZnO films produced by ALD are all of hexagonal phase, as seen in Table III.

The most studied precursor combination for ZnO ALD is diethyl zinc (DEZ)–water. The deposition temperature range lies from around 100 to 350 °C. Zinc oxide thin films are typically crystalline even when deposited at low temperature. Amorphous zinc oxide films can be deposited only as very thin films on certain substrate materials.[1149,1211,1239] With the DEZ-water process, ZnO films have been deposited on wide variety of substrate materials, including glasses, silicon, and different oxide materials (for details, see Table III). Besides water, oxygen,[1186] oxygen radicals,[1270] N_2O,[1276] and ozone[1193,1208,1209] have been utilized as oxygen sources together with DEZ.

Resistivity of ZnO is sensitive to stoichiometry. Usually, oxide films prepared in vacuum are oxygen deficient and therefore rather conducting. In fact, it is difficult to prepare strictly stoichiometric, insulating ZnO needed in, for example, piezo applications. Ozone and oxygen radicals can improve the stoichiometry and thereby increase the resistivity.[1193,1270] When DEZ is replaced by its methyl analogue dimethyl zinc (DMZ), higher GPC is obtained due to the smaller size of DMZ.[1157] At low deposition temperature, the films deposited from DMZ were more transparent than the ones grown from DEZ. Resisitivity, in turn, was higher in the films grown from DMZ.

In addition to the alkyl compounds, zinc acetate has been applied as a zinc precursor for ZnO ALD.[1279,1282,1285] Resistivity was reported to be higher in the films grown from the acetate than in the films grown from DEZ.[1282] With zinc chloride ($ZnCl_2$) and oxygen gas (O_2), epitaxial growth of zinc oxide was found on both sapphire[1153,1154] and GaN[1155] at temperature range $450 - 550$ °C. In addition, atmospheric deposition pressure was applied in these experiments.[1153,1154] Crystalline ZnO films have been also deposited from elemental zinc and water.[1151]

In zinc oxide thin films prepared by ALD, the microstructure is crystalline hexagonal. The orientation of crystallites in the film can be modified by substrate material and by tailoring the ALD growth parameters. The extreme case of substrate effect on the film orientation is epitaxy. ZnO films have been deposited epitaxially on sapphire (1120), $(10\bar{1}0)$, and $(11\bar{2}0)$ faces as well as on GaN.[1162,1229,1246,1278,1282] Also on ZnO nanowires and seed crystals, the film growth shows high degree of orientation in the same direction as the substrate.[297,2316] Interestingly, by depositing ALD aluminum oxide of different thicknesses on the ZnO nanowires the substrate effect on the film orientation could be modified. The thicker the alumina layer between the substrate and the film, the more random was the orientation in the ALD ZnO film.[297]

When deposited on silicon, glass, or, e.g., TiO_2, ZnO films are polycrystalline and typically orientate towards [100] or [001] direction. The orientation appears to depend, first, on growth temperature. At low temperature, random or [100] orientation is favored whereas at higher temperatures, film orientates to [001]. The phenomenon seems to be universal when epitaxial growth is excluded. Very similar orientation behavior has been found for several precursor systems. The temperature limit for the orientation change from [100] to [001] depends on the precursors being around 100 °C for processes exploiting oxygen plasma together with DEZ or DMZ,[1164,1270,1272,1275] slightly above 200 °C for DEZ-water,[1157,1160,1176,1220,1228] and above 300 °C for zinc acetate-water.[1285] Further increase in deposition temperature improves the film crystallinity and orientates the film more strongly towards [001], as illustrated in Figure 12. The temperature dependence in the case of zinc acetate is illustrated in Figure 13. In addition to (100), and (002) reflections, strong (110) reflections have been reported to arise from films whose thickness lies in the range from hundreds to thousands of nanometers, especially at low growth temperatures.[1160,1233] There are deviations from the above mentioned crystallite orientation trends, however. The strongest reflection from the film has been also reported to be (101).[1180,1181,2126]

Besides deposition temperature and the precursors, film orientation can be modified by other deposition conditions. At low growth temperatures, the [100] orientation can be turned to [001] by elongating the purge time between the precursor pulses. This has been found for both diethyl zinc-water[1213] and zinc acetate-water[1285] processes. Malm *et al.* deposited films from DEZ and water with long purges in temperature range starting from room temperature up to 140 °C and reported [001] preferred orientation throughout the studied range.[1265] Furthermore, an additional pulse of molecular oxygen switched the orientation from [100] to [001] in the case of DEZ-water at 180 °C.[1186] In addition, ZnO could be deposited epitaxially onto sapphire $(10\bar{1}0)$ and (0001) faces from DEZ-water only when the precursor exposure time was elongated by isolating the reaction chamber during the precursor pulse.[1229,1246] Liu *et al.* reported that the film grown from DEZ-water by regular ALD is more or less randomly oriented, but by applying an electric field over the substrate, epitaxial films can be grown on (0001)-cut sapphire without modifying any other process conditions.[1180,1181] It was suggested that polarization of precursor molecules would align them and that way direct the film growth on the substrate.

It has been reported that the (001) surface is thermodynamically the most favorable, i.e., it has the lowest surface energy.[1193] The (100) crystal face, in turn, has been reported

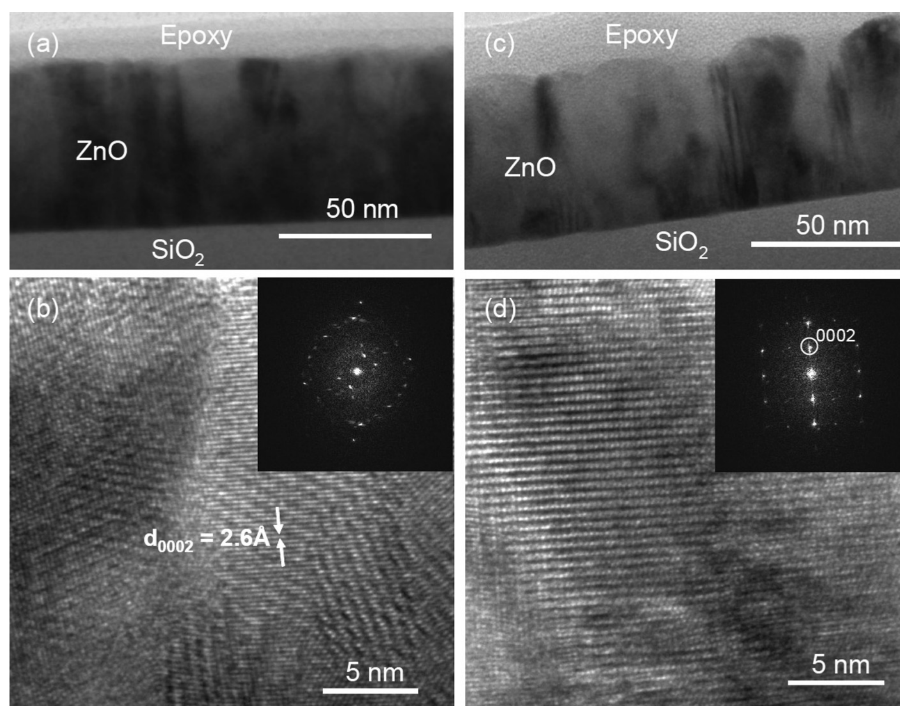

FIG. 12. Low resolution [(a) and (c)] and high resolution [(b) and (d)] TEM images of ZnO films deposited from DEZ and water at 250 °C [(a) and (b)] and 400 °C [(c) and (d)] on SiO_2/Si. Insets show digital diffractograms from the corresponding high resolution TEM images.[1250] Reprinted with permission from Y.-S. Min, C. J. An, S. K. Kim, J. Song, and C. S. Hwang, Bull. Korean Chem. Soc. **31**, 2503 (2010). Copyright 2010 The Korean Chemical Society.

to have stepped structure.[1176] Pung *et al*. suggested that at low deposition temperature the dissociation products of DEZ could block the surface sites of the (001) face while at higher temperature these species would further react into desorbable molecules revealing the (001) face for ALD growth.[1220] Makino *et al*. suggested that a decrease of the hydroxyl group density on the substrate surface at higher temperature would increase the surface mobility.[1161] Yousfi *et al*. reported that when the ZnO growth process was interrupted for 10 min and then restarted, a nucleation period was observed, while after a shorter pause, the growth recovered immediately.[1176] They suggested that the surface could rearrange to a less reactive form during the longer interrupt. Wójcik *et al*. concluded that elongated purge time allows longer time for the surface species to diffuse.[1285] As discussed above, many papers report that elongation of purge or exposure times, or addition of an intermediate oxygen pulse switches the ZnO crystallite orientation from [100] to [001]. However, origin of the phenomena is not widely discussed in the literature and still requires further investigations.

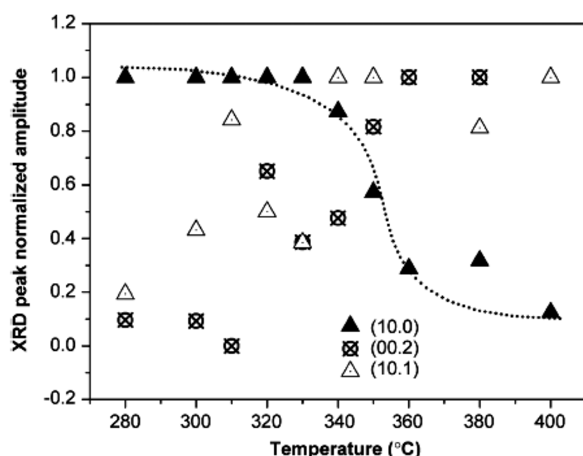

FIG. 13. Relative ratio of XRD peak intensities of ZnO films as a function of temperature deposited from zinc acetate and water.[1285] Reprinted with permission from A. Wójcik, M. Godlewski, E. Guziewicz, R. Minikayev, and W. Paszkowicz, J. Cryst. Growth **310**, 284 (2008). Copyright 2008 Elsevier.

Film thickness as a factor in the development of the ZnO film orientation has not been as extensively studied as temperature and other reaction conditions. Makino *et al*. reported that as the film grows thicker, the orientation seems to decrease and becomes more random.[1222] They applied PEALD process at low deposition temperature, and the orientation was not observed to be strong in the earlier stage of the growth either. Przézdziecka *et al*. studied the effect of thickness when the film was grown from DEZ and water.[1233] At 100 °C, (110) reflection was notable and became stronger as films grew thicker. At 200 °C, the effect of the film thickness on the (110) reflection was found to be opposite. Song *et al*. deposited ZnO on indium tin oxide (ITO) from DEZ and O_2 plasma.[2308] They reported that the thinnest films (100 nm) showed [001] orientation and the orientation became random as the film reached 200 nm thickness but arranged again at 300 nm thickness, now towards [100].

As above demonstrated, characteristically hexagonal ALD zinc oxide shows diverse behavior as it comes to crystal orientation. The orientation appears to be a function not only of substrate and deposition temperature but also of other deposition conditions, such as precursor purge time.

B. Metal nitrides

1. General

Metal nitrides are hard, chemically resistant, sometimes catalytically active, and often electrically conductive materials.[2354] Since the pioneering work on the growth of metal nitrides by ALD published in 1988,[933] there has been

increasing interest in nitride deposition by ALD, especially for application as diffusion barriers and electrodes in microelectronics.[2355] An overview of the crystallinity of metal nitride films grown by ALD is shown in Table IV.

Metal nitrides have most typically been grown by ALD using metal chloride reactants or other halides and ammonia (Table IV). These processes give nitrides at relatively high temperatures (typically ca. $350-500\,^{\circ}$C) with some residues of the respective halogens. For transition metals (TiN, ZrN_x, MoN_x, HfN_x, TaN_x, WN_x), many recent efforts have concentrated on metalorganic alkylamide-based compounds as reactants, with the goal of eliminating halogen residues. With the alkylamides, the deposition temperatures are typically lower than with the halides (typically ca. $150-250\,^{\circ}$C). A drawback of the amide compounds is their thermal decomposition at low temperatures (even during storage),[976,2357] resulting in a non-self-limiting CVD component in addition to the ALD growth and high impurity contents. The nitrogen of the alkylamide reactants has in many cases been observed to incorporate in the film, serving as an additional or even the main nitrogen source.[995,996,2042,2118] Organometallic reactants (with direct metal–carbon bonds), mainly alkyls, have been used to grow the nitrides of Main Group III elements (Al, Ga, In). Decomposition of the organometallic reactants at the temperatures needed for ammonia to complete the reactions may be a problem also for metal alkyls, however.[484]

In many metal nitride ALD processes, the metal center needs to be reduced, since in the reactant the metal often is in a higher oxidation state than in the product nitride. Ammonia works as a reducing agent, at least in some processes (e.g., TiN and NbN). If several stable nitrides exist for a given metal, ammonia leads generally to the one with the highest oxidation state, of this the Ta_3N_5 (dielectric) and TaN (conductive) pair being perhaps the most well-known example. Additional reducing agents (e.g., Zn, Me_3Al, B_2H_6, Si_2H_6) may be employed to assist the metal nitride deposition, although these may leave behind residues in the film. In nitride ALD, plasma-enhanced processes are often useful, in many cases facilitating the growth of the desired phase with the metal in the reduced state and at the same time decreasing the impurity levels and extending the ALD temperature range towards lower temperatures.[960] Drawbacks of PEALD are the more complex process control, decreased conformality of the film, and possible plasma damage to the substrate.

2. Case: Titanium nitride

TiN is one of the most investigated ALD nitride processes. As in many other transition metal nitride processes, also in TiN ALD, reduction of the metal center is needed, since the oxidation state of titanium in the reactants is +IV (see Table IV), whereas in the desired product nitride, TiN, it is +III. In many cases, the nitrogen source (e.g., NH_3) acts also as a reducing agent, while in other processes, separate reducing agents have been added.

In the pioneering work on metal nitride ALD by Hiltunen *et al.* in 1988,[933] TiN was grown from titanium tetrachloride ($TiCl_4$) and ammonia (NH_3) at $500\,^{\circ}$C. The $TiCl_4-NH_3$ process has remained the most commonly applied process to deposit TiN to date, while according to Table IV the temperature range of the $TiCl_4-NH_3$ process has been extended down to about $300\,^{\circ}$C and up to $550\,^{\circ}$C.[938–940,946,951,952,955] Higher temperatures should also be possible since decomposition of the reactants is not expected. Growth per cycle values between ca. 0.02 and 0.04 nm have been reported. In some studies, Zn[934,935] or $AlMe_3$[957,2358] has been added as a reducing agent to the $TiCl_4-NH_3$ process, with the goal to improve the TiN characteristics such as the electrical conductivity. While little zinc residues were left in the TiN films deposited with Zn, several atomic percents of aluminum and carbon were left in the films from $AlMe_3$. In addition to $TiCl_4$, also TiI_4 can be used together with NH_3 to deposit TiN.[968] In combination with $TiCl_4$ and TiI_4, dimethylhydrazine, tert-butylamine, and allylamine have been explored as alternative nitrogen sources with more reducing power than ammonia.[966,967] Plasma of H_2 and N_2 has also been used in combination with $TiCl_4$, extending the deposition temperature range down to $100\,^{\circ}$C.[958,960,962,963]

In addition to inorganic halides, also metal-organic reactants have been used for TiN deposition. Metal-organic reactants are desired in cases where the corrosive by-products of the halide-based processes cannot be tolerated either by the underlying materials (e.g., copper pitting by HCl[944]) or by the ALD reactor itself (e.g., stainless steel reactors). Titanium alkylamides $Ti(NMe_2)_4$ [tetrakis(dimethylamido) titanium, TDMAT][973,975,980] and $Ti(NEtMe)_4$ [tetrakis (ethylmethylamido) titanium, TEMAT][996] have been used to deposit TiN together with ammonia at temperatures typically around $150-250\,^{\circ}$C. TiN has also been grown by PEALD processes from $Ti(NMe_2)_4$.[988,989,994,995] Growth per cycle values from 0.2 nm up to several nanometers per cycle, corresponding to several TiN monolayers per cycle, have typically been reported. The high absolute values and the high variation of the GPC originate from the fact that the alkylamide-based processes do not fulfill the basic saturation criterion of ALD because decomposition of the metal precursor occurs.[976]

TiN is one of the materials whose amorphous/crystalline nature depends on the ALD process parameters; both crystalline and amorphous films have been frequently reported. Investigating the data in Table IV shows two trends. TiN films deposited from the metal halide reactants are mainly crystalline, while TiN films deposited from the alkylamide reactants are mainly amorphous—both with some exceptions. In the following, we analyze the reasons for these trends as well as for their exceptions. To assist this analysis, published data for the growth characteristics, impurities, and density of TiN films made by different ALD processes have been collected in Table V together with information on film crystallinity.

When grown from the metal halide reactants and ammonia, crystalline TiN films are always obtained (Tables IV and V). Also the corresponding PEALD TiN process gives crystalline films, even at 100 °C.[960] The films are polycrystalline, with cubic columnar crystals that extend throughout the film.[938,945,952] An example of a TiN film made with the

TABLE IV. Crystallinity studies of ALD nitride films made by diffraction or spectroscopy.[a]

Material reactant A	Reactant B	Substrate(s)	Phases		References
BN			Amorphous	Turbostratic	
BBr_3	NH_3	silica	400 °C	750 °C	49
BBr_3	NH_3	Al_2O_3		250–600 °C	50
BBr_3	NH_3[e]	Al_2O_3		250–600 °C	50
AlN			Amorphous	Hexagonal	
$AlCl_3$	NH_3	SLG		500 °C	477
$AlCl_3$	$NH_3 + H_2$[c]	H-Si		350 °C	480
$AlMe_3$	NH_3	Corning 7059		325–425 °C	484
$AlMe_3$	NH_3	Si	325–400 °C		488
$AlMe_3$	NH_3	Sapphire		1200 °C	489
$AlEt_3$	NH_3	Sapphire		450 °C	495
$Al(NMe_2)_3$	NH_3	Ge	200–250 °C		501
$Me_2EtN{:}AlH_3$	NH_3	Al_2O_3		300–380 °C	498
$Me_2EtN{:}AlH_3$	NH_3	Si	300–380 °C		498
TiN			Amorphous	Cubic	
$TiCl_4$	NH_3	SLG, Si, H-Si, SiO_2, $HfSiO_x$, HfO_2		300–550 °C	933, 938–940, 946, 951, 952, and 955
$TiCl_4$	NH_3-Zn	SLG		400–500 °C	934 and 935
$TiCl_4$	NH_3-$AlMe_3$	GaAs	275 °C		957
$TiCl_4$	$H_2 + N_2$[c]	Si, SiO_2		100–400 °C	958, 960, and 962–965
$TiCl_4$	DMHy	SLG	250 °C	300–400 °C	966
$TiCl_4$	$^tBuNH_2$	SLG, borosilicate	400 °C	400 °C	967
$TiCl_4$	$allylNH_2$	SLG, borosilicate	400 °C	400 °C	967
TiI_4	NH_3	SLG		400–500 °C	968
TiI_4	$^tBuNH_2$	SLG, borosilicate	400 °C	400 °C	967
TiI_4	$allylNH_2$	SLG, borosilicate	400 °C	400 °C	967
$Ti(NMe_2)_4$	NH_3	H-Si	200–300 °C	350 °C	975
$Ti(NMe_2)_4$	NH_3	Si, SiO_2	180 °C	180 °C	973 and 980
$Ti(NMe_2)_4$	NH_3[c]	SiO_2	250 °C		988 and 989
$Ti(NMe_2)_4$	$NH_3 + H_2$[c]	Al_2O_3		150–300 °C	493 and 990
$Ti(NMe_2)_4$	N_2[c]	SiO_2	250 °C		989
$Ti(NMe_2)_4$	N_2[c]	SiO_2		200 °C	994
$Ti(NMe_2)_4$	H_2[c]	Si, SiO_2	150–250 °C	150 °C	989 and 995
$Ti(NEtMe)_4$	NH_3	Si, SiO_2	150–220 °C		996 and 997
Cu_3N			Amorphous	Cubic	
$[Cu(^sBuAMD)]_2$	NH_3	Ru		140–300 °C	1140
$Cu(hfac)_2$	H_2O-NH_3	SiO_2		210–300 °C	1141

TABLE IV. (*Continued.*)

Material reactant A	Reactant B	Substrate(s)	Phases				References
GaN			Amorphous	Cubic			
GaCl	NH_3	GaN		300–400 °C			1376 and 1377
GaCl	NH_3	GaAs	300–400 °C				1377
$GaCl_3$	NH_3	GaAs(001)	500 °C				1378
$GaCl_3$	NH_3	GaAs(111)[d]		500 °C			1378
$GaCl_3$	NH_3	H-Si		650 °C			1379
$GaMe_3$	NH_3	AlN[d]		550–650 °C			1380
$GaMe_3$	NH_3	AlN[d]		800–900 °C			1384
$GaMe_3$	NH_3	GaAs(001)		500–650 °C			1383
$GaEt_3$	NH_3	AlN[d]		450–900 °C			1385
$GaEt_3$	NH_3	SiC(0001)[d]		250–500 °C			1387
$GaEt_3$	NH_3	SiC(0001)	150–170 °C	170–250 °C			1387
ZrN_x			Amorphous				
$Zr(NMe_2)_4$	NH_3	Si	150–250 °C				1622
$Zr(NEtMe)_4$	NH_3	Si	150–250 °C				1622
$Zr(NEt_2)_4$	NH_3	Si	150–250 °C				1622
$Zr(NEt_2)_4$	N_2[c]	Si	300 °C				1623
NbN			Amorphous	Cubic	Hexagonal		
$NbCl_5$	NH_3	SLG, SiO_2, Al_2O_3		400–500 °C			933, 1630, and 1632
$NbCl_5$	NH_3	SLG, borosilicate	250–300 °C				1631
$NbCl_5$	NH_3-Zn	SLG		500 °C[b]	500 °C[b]		935 and 1630
$NbCl_5$	DMHy	SLG		400 °C[b]			966
MoN			Amorphous	Cubic	Hexagonal	Tetragonal	
$MoCl_5$	NH_3	SLG, Si		350–500 °C	450–500 °C		933 and 1635
$MoCl_5$	NH_3-Zn	SLG		500 °C	500 °C		966
$MoCl_5$	DMHy	SLG		500 °C	500 °C		966
$Mo(^tBuN)_2(NMe_2)_2$	NH_3	Si, SiO_2, Ni, Cr	260–300 °C			260–300 °C	1636
$Mo(^tBuN)_2(NEt_2)_2$	NH_3	Si	285–300 °C			285–300 °C[b]	1638
InN			Amorphous	Hexagonal			
In	N_2[e]	Sapphire[d]		440 °C			1762
$InMe_2Et$	NH_3	AlN[d]		480 °C			485
$InMe_2Et$	NH_3	AlN		480 °C			485
HfN_x			Amorphous	Cubic			
$Hf(NMe_2)_4$	NH_3	Si	150–250 °C				1622
$Hf(NEtMe)_4$	NH_3	Si, Ge	150–250 °C				501 and 1622
$Hf(NEtMe)_4$	H_2[c]	SiO_2		250 °C			2118
$Hf(NEt_2)_4$	NH_3	Si	150–250 °C				1622

TABLE IV. (*Continued.*)

Material reactant A	Reactant B	Substrate(s)	Phases			References
TaN_x			Amorphous	Cubic	Orthorhombic	
$TaCl_5$	NH_3	SLG		500 °C[b]		933
$TaCl_5$	NH_3	SLG, SiLK^{TM}	300–500 °C		400–500 °C	2160 and 2161
$TaCl_5$	NH_3-Zn	SLG		400–500 °C		2160
$TaCl_5$	DMHy	SLG	300–400 °C			966
$TaCl_5$	^{t}BuNH$_2$	SLG	350 °C	400–500 °C		2163
$TaCl_5$	^{t}BuNH$_2$ + NH_3	SLG		400–500 °C		2163
$TaCl_5$	H_2 + N_2[c]	H-Si, SiO_2		100–400 °C		2164 and 2166
$TaCl_5 \cdot SEt_2$	NH_3 - $AlMe_3$	Si		400–500 °C		2169
$TaBr_5$	^{t}BuNH$_2$	SLG	400 °C	450–500 °C		2163
$TaBr_5$	^{t}BuNH$_2$ + NH_3	SLG		400–500 °C		2163
TaF_5	H_2 + N_2[c]	SiO_2		350 °C	350 °C	2170
TaF_5	H_2[c]-NH_3	SiO_2		200–350 °C		2125–2127
TaF_5	H_2 + NH_3[c]	SiO_2		200 °C	200 °C	2127
$Ta(NMe_2)_5$	H_2[c]	H-Si, SiO_2, Si		150–250 °C		964, 2152, 2182, and 2183
$Ta(NMe_2)_5$	N_2 + H_2[c]	H-Si, SiO_2	250 °C		150–250 °C	2182 and 2183
$Ta(NEtMe)_5$	NH_3	H-Si, SiO_2	220–250 °C	250 °C		2184
Ta(^{i}PrN)(NEtMe)$_3$	N_2 + H_2[c]	SiO_2	400 °C			2185
Ta(^{t}BuN)(NEt$_2$)$_3$	NH_3	SiO_2, Si	150–260 °C	350–400 °C		2187, 2188, 2192, and 2198
Ta(^{t}BuN)(NEt$_2$)$_3$	N_2H_4	Si	150–250 °C			2188
Ta(^{t}BuN)(NEt$_2$)$_3$	H_2[c]	SiO_2, Si	250–350 °C	250–350 °C		1669, 2191, 2192, and 2195
Ta(^{t}BuN)(NEt$_2$)$_3$	NH_3[c]	SiO_2	250 °C	350–400 °C		2198
Ta(^{t}AmN)(NMe$_2$)$_3$	H_2[c]	SiO_2		230 °C		1671
WN_x			Amorphous	Cubic W_2N	Hexagonal WN	
WF_6	NH_3	Si		327 °C		2224
WF_6	NH_3 (ethene, silane cat.)	Si		350 °C	350 °C	2225
WF_6	NH_3	SiO_2		350 °C[b]		944
WF_6	NH_3 + B_2H_6	SiO_2	300 °C	300 °C[b]	300 °C[b]	2356
WF_6	NH_3 + B_2H_6	SiO_2		200–350 °C	200–350 °C	2230
W(^{t}BuN)$_2$(NMe$_2$)$_2$	NH_3	Si, SiO_2, Ni, Cr	250–380 °C			1067, 1689, and 2231
$W_2(NMe_2)_6$	NH_3	Si	180–210 °C			2235

[a]SLG, soda lime glass; H-Si, HF-etched Si; Precursors connected with +, fed in the same pulse; Precursors connected with dash, pulsed separately; DMHy, dimethylhydrazine.
[b]Ambiguous interpretation due to reflection overlap, low film thickness, or weak intensity.
[c]Plasma.
[d]Epitaxy.
[e]Photo-assisted.

TABLE V. Results for some ALD and PEALD TiN films: film type (crystalline/weakly crystalline/amorphous), average growth per cycle (GPC), stoichiometry, impurities, and density.

ALD (°C)	Film type	GPC (nm)	N:Ti	Impurities, at. % C	H	O	Cl (I)	Density ($g\ cm^{-3}$)	References
Halide reactants									
Thermal ALD									
$TiCl_4$–NH_3									
300	Cryst.	0.03	…	…	…	…	7.7[a]	…	955
350	Cryst.	0.04	0.9	∼0	…	∼0	2.5	4.2	938
400	Cryst.	0.017	…	…	…	< d.l.	1.2	…	944
400	Cryst.	0.017	1.16	∼0	…	∼0	1.2	∼5.4	950
400	Cryst.	0.04	0.9	∼0	…	∼0	0.5	4.7	938
450	Cryst.	0.03	…	…	…	…	1.8	…	955
450	Cryst.	0.04	0.9	∼0	…	∼0	0.3	4.9	938
500	Cryst.	0.02	…	…	<0.4	…	<0.5	…	934
500	Cryst.	0.02	0.9	∼0	…	∼15	<0.05	…	935
500	Cryst.	0.03	…	…	…	…	1.0	…	955
TiI_4–NH_3									
400	Cryst.	0.012	0.5	…	…	∼30	<0.5 (I)	…	968
500	Cryst.	0.03	0.8	…	…	∼10	<0.5 (I)	…	968
$TiCl_4$–allylNH_2									
400	Amorph.[b]	0.015	0.7	9	≳ 3[c]	≳ 13	7	…	967
$TiCl_4$–DMHy									
250	Amorph.	0.022	…	…	…	…	18	…	966
300	W. cryst.	0.022	…	6	2	…	7	…	966
400	W. cryst.	0.026	1.0	13	2	14	2	…	966
Plasma ALD									
$TiCl_4$–N, H plasma									
100	Cryst.	0.026	1.1	…	16	2.6	2.1	3.9	960
200	Cryst.	0.027	1.0	…	…	1.4	0.96	…	960
300	Cryst.	0.036	1.1	…	…	2.8	0.42	…	960
320	Cryst.	0.030	1[d]	∼0	…	∼0	∼1.0	4.9	950
400	Cryst.	0.050	1.0	…	3	1.8	0.07	4.8	960
Alkylamide reactants									
Thermal ALD									
$Ti(NMe_2)_4$–NH_3									
150	Amorph.	0.3	1.0	21	…	20	…	…	975
180	Amorph.	0.2	…	10	…	20[e]	…	…	973
180	W. cryst.	0.7	0.9	6	…	37	…	3.1	980
200	Amorph.	0.5	1.0	20	…	20	…	…	975
200	…	0.06	0.5	9	…	37	…	…	986
240	W. cryst.	1–11	0.8	7	…	37	…	3.0	976
350	W. cryst.	7.6	1.0	45	…	7	…	…	975
$Ti(NEtMe)_4$–NH_3									
200	Amorph.	0.5	0.6	4	6	…	…	…	996
Plasma ALD									
$Ti(NMe_2)_4$–H_2 plasma									
150	Cryst.	0.035	0.7	11	…	0	…	4.1	995
250	Amorph.	…	1	<5	…	25	…	…	989
$Ti(NMe_2)_4$–N_2 plasma									
250	Amorph.	…	1.3	4.5	…	10	…	…	989
$Ti(NMe_2)_4$–NH_3 plasma									
200	…	0.08	0.9	1	…	5	…	…	986
250	Amorph.	0.2	1.2	<3	…	20	…	…	988
250	Amorph.	…	1.1	1	…	20	…	…	989

[a]Reduction of Cl by pump-down to 2.3 at. %.
[b]Results for 7 sccm flow rate. Higher flow rate of 16 sccm gave weakly crystalline film.
[c]For a similar process using NH_3 with co-reactant, about 3 at. % H and 13 at. % O were reported. With the allylNH_2 alone, these impurities are most likely at a similar or higher level.
[d]N:Ti adjustable, 0.93–1.1.
[e]40 at. % O after 30d air exposure.

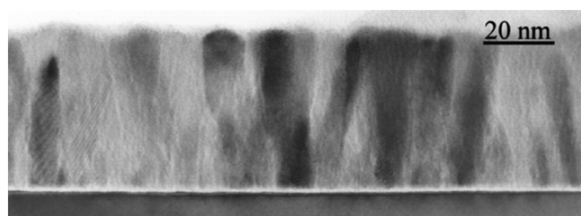

FIG. 14. TEM cross-section of TiN grown on native silicon oxide from $TiCl_4$ and NH_3 at 400 °C. Reprinted with permission from K.-E. Elers, V. Saanila, W.-M. Li, P. J. Soininen, J. T. Kostamo, S. Haukka, J. Juhanoja, and W. F. A. Besling, Thin Solid Films **434**, 94 (2003). Copyright 2003 Elsevier.

$TiCl_4 - NH_3$ process on silicon with columnar crystallites is shown in Figure 14. Crystallite diameter is on the order of 20 nm.[945] The orientation of the lattice planes varies depending on the exact process details—e.g., substrate, temperature,[938,952,955,960] and the presence of additional reducing agents.[934] The most abundant orientation is [100] in most studies, but other orientations are seen to coexist. Examples of x-ray diffractograms measured for TiN made by the $TiCl_4 - NH_3$ process are shown in Figure 15.

Temperature is among the most important factors affecting crystallinity, both directly as well as indirectly through the impurity contents. Common to the metal halide-based processes is the ALD-wise relatively high process temperature: Almost all studies with thermal ALD have been made at temperatures above 300 °C. Although high for ALD, this temperature is still low compared to the melting point of TiN of about 2930 °C. The minimum ALD temperature is set by thermodynamics: At lower temperatures, the Gibbs energy of the possible reaction paths of metal halides with ammonia is positive, and completing the necessary reactions is difficult.[934] The relatively high ALD temperature can in principle be one factor that may explain the crystalline nature of TiN films grown from the halide reactants: Qualitatively seen, higher temperatures allow the atoms to find the most relaxed locations during the ALD cycle.

Another important factor that affects film crystallinity is the impurity concentrations: High concentrations might prevent crystallization altogether. It may be difficult to separate

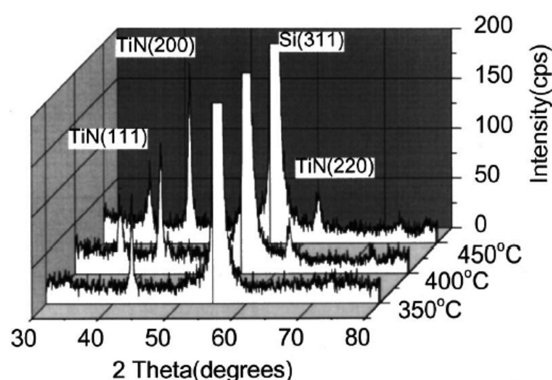

FIG. 15. X-ray diffractograms of TiN films deposited from $TiCl_4$ and NH_3 at in 1000 ALD cycles at temperatures 350, 400, and 450 °C. Reprinted with permission from H. Jeon, J.-W. Lee, Y.-D. Kim, D.-S. Kim, and K.-S. Yi, J. Vac. Sci. Technol. A **18**, 1595 (2000). Copyright 2000 American Vacuum Society.

the effect of temperature from the effect of impurities, since these often go together: The higher the ALD temperature, the lower the impurity concentrations often (but not always) are. This decreasing trend in impurity contents with increasing ALD temperature is clear also for the titanium halide–ammonia TiN ALD processes (Table V). However, polycrystalline TiN has been reported even in cases where there are significant concentrations of impurities present in the layers, e.g., 7 at. % Cl for thermal ALD at 300 °C and 2 at. % Cl and 16 at. % H for PEALD at 100 °C. It is clear, therefore, that significant quantities of Cl and H impurities do not prevent the crystallization of ALD TiN films. Also the oxygen concentrations measured for some TiN films have been high (tens of percents). However, most likely this oxygen has not been incorporated during the film growth but has come through oxidation of the TiN films upon their exposure to air.[2359] Also, in general in polycrystalline TiN films, oxygen has been found not to be uniformly distributed but concentrated on the surface and grain boundaries, thereby leaving crystallinity of grain interiors unaffected.[2360,2361] Based on a recent paper, this seems to be the case for ALD TiN films also.[2359] On the basis of the TiN growth results with the halide–ammonia processes, one cannot therefore conclude on the effect of O impurities on the crystallization behavior, but the fact that Cl and H impurities do not prevent crystallization seems clear.

The few exceptions related to the deposition of amorphous or weakly crystalline TiN from halide reactants (Tables IV and V) are all from processes where organic groups are present in the reducing agent. Carbon residues have been incorporated, and apparently hampered crystallization. This has been the case for the $TiCl_4$–DMHy process[966] and the $TiCl_4$–allylNH_2 process.[967] The carbon impurity levels were 6–13 at. %, while other impurities and reaction temperatures were similar to the other $TiCl_4$-based studies. No impurity analysis was reported for the film grown by the $TiCl_4 - AlMe_3 - NH_3$ process at 275 °C,[957] but the low density of 3.2 g cm^{-3} reported points to high impurity contents in that case as well. On the basis of this analysis, it seems that C contents of some atomic percents hinder the crystallization of TiN films, also in films grown at higher temperatures (400 °C).

TiN films grown from the alkylamide reactants have been reported in most cases to be amorphous (Table IV). Another difference compared to the halides is the markedly lower ALD temperature range: Films have been grown by the alkylamide-based processes at 150 − 250 °C whereas halides typically require temperatures above 300 °C.

The data in Table V indicate that the amorphous TiN films grown by thermal ALD from the alkylamide reactants are loaded with impurities. Typical are carbon contents of 10–20 at. %. Hydrogen content has been determined only in one study, being 6 at. %.[996] Oxygen contents are in almost all cases high (tens of percents), but similarly to the halide-based processes, the oxygen probably originates from oxidation after the growth upon exposure of the films to ambient air.[973,987] The density is low, just around 3 g cm^{-3}.[976,980] The high carbon content must originate from incomplete removal of the ligands of the alkylamide reactants or from

thermal decomposition. $Ti(NEtMe)^4$, for example, decomposes at about 240 °C, depositing a TiN-type film without any co-reactant.[996] In a few experiments, some crystallinity has been observed, and this has been accompanied with a somewhat lower carbon concentration, being <10 at. %.[976,980] (The film grown at 350 °C with crystallinity and 45 at. % C is an exception to this "rule," but this film has been grown at a temperature far higher than decomposition temperature of the alkylamide reactant, and it is therefore a CVD film rather than ALD film.) The TiN films made from the alkylamide reactants by thermal ALD are another example of films, which are amorphous and simultaneously have a high C content.

Plasma-enhanced ALD processes based on alkylamide reactants have similarly to the thermal ALD processes most often given amorphous material with significant impurities. All films with a high oxygen content have also been amorphous (Table V); the amorphous and impure nature of the film probably allows easy oxidation in ambient air. In one study, crystalline film which was not oxidized in air was obtained, however. In this study, the conditions were optimized for removal of the amino groups of the alkylamide reactants by hydrogen plasma.[995] Simultaneously, the GPC was much lower (0.035 nm) than in other alkylamide-based studies (and similar to the halide-based processes), and crystalline, dense and conductive films were obtained at 150 °C. These films contained a significant amount of carbon, however, 11 at. %.[995] It seems therefore that even the plasma process cannot remove all carbon that results from the decomposition of the alkylamide reactant. The plasma process has probably converted the carbon to carbidic, conductive form, and the material is more correctly described as Ti(C,N) solid solution instead of TiN.

In conclusion, TiN films deposited by ALD seem to grow in a crystalline form, unless there are high amounts of C impurities present that prevent crystallization. Temperature as such does not seem to dictate the crystallinity. Also Cl and H impurities even in high concentrations (over ten percents) do not seem to affect the crystallinity. Crystalline films are obtained by either halide-based processes at higher temperatures (>300 °C) or PEALD at lower temperatures (<300 °C). Amorphous, TiN-type films can be obtained in a wide temperature range by thermal ALD using reactants that contain organic groups—either titanium halide combined with organic reducing agents or alkylamides with any (or no) reducing agent—but this comes with the cost of significant carbon contamination of the films.

C. Metal chalcogenides

1. General

From metal chalcogenides, the most attention has been paid for zinc sulphide, and even the very early ALD experiments were with elemental zinc and sulfur to grow ZnS.[2362] The motivation for the development of the whole ALD thin film deposition technology was the need for a better method to fabricate thin film EL displays. The light-emitting layer in monochrome yellow display is Mn(II) doped ZnS. The development work was very successful and in addition to the ZnS:Mn, strong contribution to the success was coming from the pinhole-free ALD Al_2O_3 and especially Al_2O_3/TiO_2 nanolaminate dielectric layers.[81,2363] The need to develop multi-color and full color EL displays led to studies on rare earth doped ZnS films and doped alkaline earth sulfides.[675,1512,2364,2365] The interest towards cadmium and indium sulfide originates from their use as buffer layer in solar cells, especially in those based on $CuInSe_2$.[831,1766] ALD is one option for their fabrication.

ALD of selenides and tellurides was studied already in 1980s. The interest stems from thin film solar cells and blue light-emitting diode (LED) materials. CdTe is an important photovoltaic material, and most of the ALD studies have focused on the preparation of CdTe films from the elements.[1042] ZnSe, on the other hand, was the most promising blue LED material in 1980s, and its studies concentrated on epitaxial films on single crystal substrates.[1323] ALD studies on epitaxial II-VI compounds did not lead to industrial applications, and ALD of selenide and telluride films was scarcely studied until 2009, when a new process based on alkylsilyl chalcogen compounds was discovered.[1148] This new interest stems from phase change materials, which are aimed for electronic data storage.

An overview of crystallinity of metal sulfide, selenide, and telluride films grown by two-reactant ALD processes is shown in Table VI.

2. Case: Zinc sulfide

The first ZnS ALD experiments were made with elemental zinc and sulfur. However, it was soon realized that this process was not industrially applicable for the production of EL displays. Therefore, a new process, based on compound reactants $ZnCl_2$ and H_2S, was developed.[76] There were indications that EL displays may suffer in performance over time due to chlorine residues in the films. Since then, zinc acetate,[1279] DMZ,[1306] $Zn(thd)_2$ (thd = 2,2,6,6-tetramethyl-3,5-heptanedionate),[2367] and DEZ,[1318] have been studied as a zinc precursor, H_2S being the sulfur source. The use of $ZnBr_2$ as a precursor has been mentioned in the literature and ZnI_2 has been used for deposition of ZnS:Mn.[1305,2367]

The different zinc precursors affect strongly the growth rate and process temperatures. For zinc chloride and diethyl zinc, similar growth rates have been reported (about 1 Å/cycle) at 500 °C and 150 °C, respectively.[1294,1298,1299,1316,2368] Higher growth rates have been reported for films deposited at lower temperatures. The growth rates decrease with increasing temperature, and with DEZ this behavior is stronger than with halides. The deposition temperatures and growth rates with DMZ are similar to those of DEZ, but the temperature dependence is weaker.[2368] Acetate yields higher growth rates (2.6 Å/cycle), which has been attributed to the fact that slowly heated zinc acetate molecules can form a volatile tetrameric complex that is referred to zinc oxoacetate (ZnOAc).[2369] The complex adsorbs on the surface, and because the zinc atoms in the complex are at appropriate positions from each other for the ZnS lattice, high growth rate can be achieved.[1304] One

TABLE VI. Crystallinity studies of ALD chalcogenide films made by diffraction or spectroscopy.[a]

Reactant A	Reactant B	Substrate(s)	Phases			References
Sulfides						
CaS			Amorphous	Cubic		
$Ca(thd)_2$	H_2S	SLG, Al_2O_3		325–450 °C		675, 676, 678, and 679
TiS_2			Amorphous	Cubic	Hexagonal	
$TiCl_4$	H_2S	Rh	400 °C			999
$TiCl_4$	H_2S	Ru		400 °C[b]	400 °C[b]	999
$TiCl_4$	H_2S	SLG, ZnS, Ir, Pd, Pt, TiN			400 °C	999
CuS_x			Amorphous	Pseudocubic	Hexagonal	
$Cu(thd)_2$	H_2S	Si, SLG			120–160 °C	1144
$Cu(thd)_2$	H_2S	Corning 7059, SnO_2:F, TiO_2		175–280 °C	125–175 °C	1145 and 1146
$Cu(^sBuAMD)_2$	H_2S	Al_2O_3	130 °C		130 °C	1147
ZnS			Amorphous	Cubic	Hexagonal	
Zn	S	GaAs[c]		140–340 °C		1288
Zn	H_2S	GaP[c]		420–600 °C		1289
$ZnCl_2$	H_2S	GaAs[c]		510 °C		1300 and 1301
$ZnCl_2$	H_2S	glass, SLG, phlogopite, ITO, silica, Al_2O_3			390–500 °C	84, 1290–1292, 1295, 1297–1299, 1302, 1304, and 2134
$ZnCl_2$	H_2S	Al_2O_3		360–500 °C[b]	360–400 °C[b]	1304
ZnI_2	H_2S	Al_2O_3		300–490 °C	300–490 °C	1305
$ZnMe_2$	H_2S	GaAs[c]		150–310 °C		1308 and 1311
$ZnMe_2$	H_2S	H-Si[c]		125–175 °C		1313 and 1314
$ZnMe_2$	H_2S	Si, Corning 7059		25–500 °C[b]	25–500 °C[b]	1306
$ZnMe_2$	H_2S	Corning 7059		200–340 °C		1309
$ZnEt_2$	H_2S	glass		120 °C[b]	120 °C[b]	1194
$ZnEt_2$	H_2S	$Al_xTi_yO_z$, Al_2O_3		250–400 °C	350–400 °C	1317 and 1318
$ZnEt_2$	H_2S	H-Si			110 °C	1215
$ZnEt_2$	H_2S	Si		100–400 °C	300–400 °C	1320
$Zn(CH_3COO)_2$	H_2S	SLG, ITO		290–390 °C		1292, 1295, and 1297
$Zn(CH_3COO)_2$	H_2S	SLG, Al_2O_3		290–360 °C[b]	290–360 °C[b]	1279, 1304, and 1322
$Zn(OAc)_2$	H_2S	Al_2O_3		250–350 °C[b]	250–350 °C[b]	1304
SrS			Amorphous	Cubic		
$Sr(^iPr_3Cp)_2$	H_2S	Al_2O_3		260–400 °C		1509
$Sr(Me_5Cp)_2$	H_2S	Al_2O_3		280–350 °C		1509
$Sr(thd)_2$	H_2S	Al_2O_3, SLG		350–450 °C		675 and 1512

TABLE VI. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases			References
CdS			Amorphous	Cubic	Hexagonal	
Cd	S	GaAs[c]		340 °C		1707
$CdCl_2$	H_2S	GaAs[c]		510 °C		1300
$CdCl_2$	H_2S	ZnS		510 °C		1300
$CdMe_2$	H_2S	ZnSe[c]		RT		1709, 1710, and 1713
$CdMe_2$	H_2S	Si, glass		100–400 °C	100–400 °C	1714
In_2S_3			Amorphous	Tetragonal		
$InCl_3$	H_2S	SLG		300–400 °C		1763
$In(acac)_3$	H_2S	ZnO	160 °C			1178
$In(acac)_3$	H_2S	glass		130–160 °C		1766, 1767, and 1772
$In(acac)_3$	H_2S	Si, $Cu(In,Ga)Se_2$, SLG, Corning 7059		140–260 °C		1768, 1769, and 1771
Sb_2S_3			Amorphous	Orthorhombic		
$Sb(NMe_2)_3$	H_2S	Si	120 °C			1829
$Sb(NMe_2)_3$	H_2S	$Sb_2 S_3$[c]		65–160 °C		2366
$Sb(NMe_2)_3$	H_2S	$Sb_2 Se_3$[c]		90–160 °C		2366
BaS			Amorphous	Cubic		
$Ba(Me_5Cp)_2$	H_2S	$Al_2 O_3$		240–300 °C		1509
$Ba(thd)_2$	H_2S	Al_2O_3, SLG		300–450 °C		675 and 1831
La_2S_3			Amorphous	Cubic		
$La(thd)_3$	H_2S	SLG, Corning 7059	300–400 °C	500 °C		1857
WS_2			Amorphous	Hexagonal		
WF_6	H_2S	ZnS		300 °C		2238
WF_6	$H_2S(ZnEt_2$ cat.)	ZnS		300–350 °C		2239
PbS			Amorphous	Cubic		
$Pb(detc)_2$	H_2S	glass, Al_2O_3, BaF_2		300–350 °C		2277
$Pb(thd)_2$	H_2S	Si		160 °C		2279
Selenides						
ZnSe			Amorphous	Cubic		
Zn	Se	GaAs		200–430 °C		1323, 1327, 1331, and 1335–1337
Zn	Se	GaAs[c]		350–410 °C		1339
Zn	Se	ZnTe[c]		200–400 °C		1323, 1325, and 1327
$ZnMe_2$	H_2Se	H-Si[c]		125–250 °C		1313, 1314, 1352, and 1354
$ZnMe_2$	H_2Se	GaAs[c]		100–200 °C		1350 and 1352
$ZnCl_2$	H_2Se	GaAs		400–500 °C		1342–1344

TABLE VI. (*Continued.*)

Reactant A	Reactant B	Substrate(s)	Phases			References
CdSe			Amorphous	Cubic		
Cd	Se	ZnSe		230–300 °C		1715, 1717, 1718, and 1720
Cd	Se	GaAs[c]		350–410 °C		1722
Tellurides						
MgTe			Amorphous	Cubic		
Mg	Te	CdTe		260–300 °C		69 and 70
ZnTe			Amorphous	Cubic		
Zn	Te	GaAs		200–400 °C		1323, 1327, and 1359
Zn	Te	GaAs[c]		380–390 °C		1363 and 1365
Zn	Te	ZnSe[c]		200–400 °C		1323 and 1327
Zn	Te	CdTe[c]		225–250 °C		1361
Zn	Te	ZnTe[c]		280 °C		1362
GeTe			Amorphous	Rhombohedral		
$GeCl_2 \cdot C_4H_8O_2$	$(Et_3Si)_2Te$	Si		90 °C		1148
CdTe			Amorphous	Cubic		
Cd	Te	CdTe[c], BaF_2[c], GaAs[c]		260–290 °C		70, 1043, 1046, 1723, 1724, 1726, 1730, and 1731
Cd	Te	ZnSe		230–300 °C		1720
$CdMe_2$	$TeEt_2$	GaAs[c], Si		250–320 °C		1367 and 1749
$CdMe_2$	TeMe(allyl)	GaAs[c], Si		250–320 °C		1367 and 1748–1750
Sb_xTe_y			Amorphous	Rhombohedral Sb_2Te_3	Sb-like rhombohedral	
$SbCl_3$	$(Et_3Si)_2Te$	Si		60 °C		1148
$SbCl_3$	$(Et_3Si)_2Te$-$(Et_3Si)_2Sb$	Si			95°C	534
HgTe			Amorphous	Cubic		
$HgMe_2$	TeMe(allyl)	GaAs[c], Si		140 °C		1748 and 1750

[a]SLG, soda lime glass; ITO, indium tin oxide; H-Si, HF-etched Si.
[b]Ambiguous interpretation due to reflection overlap or weak intensity.
[c]Epitaxy.

monolayer per cycle (3.1 Å/cycle) is possible with dimethyl zinc and elemental zinc precursors.[1289,1309] The ZnI_2 molecule is significantly larger than $ZnCl_2$, and therefore the growth rate of ZnS from ZnI_2 is less than half of that from $ZnCl_2$.[1305] Process temperatures can vary from room temperature (alkyl compounds) to >500 °C (halides), depending on the precursor.

ALD zinc sulfide films studied have been polycrystalline. Since the application has been luminescence, there has been no interest in amorphous films, because it is known that crystalline order is beneficial for light emission. ZnS is known to exist in both cubic (zinc blende or sphalerite) and hexagonal (wurtzite) crystal phases. The phase depends on the temperature, and at atmospheric pressure, the low-temperature cubic phase transforms to hexagonal form at 1020 °C. In thin films, the wurtzite phase has been seen at much lower temperatures. However, the cubic phase is known as the low-temperature and the hexagonal phase as the high-temperature form. Distinguishing the phases from each other in thin film samples with x-ray diffraction measurements is difficult, because they both show the main reflection at around 28.5° (CuK_α radiation) which can be attributed to either cubic(111) or hexagonal(002). The appearance of the hexagonal phase is usually determined from the (103) reflection at 51.8°, but this reflection is always relatively weak.[1295,2370,2371]

The XRD patterns show that the polycrystalline ALD ZnS films are preferentially oriented to either c[111] or h[001] direction. In the earlier studies, it has been suggested that ZnS films deposited by ALD below 400 °C are mainly in the cubic form and above 400 °C mainly in the hexagonal form.[1292,1317,2370] The opinion was based on the observations from the chloride process where at 350 °C the cubic phase was seen whereas at 460 °C XRD showed hexagonal reflections. Optical measurements can be used to confirm the phase structure since the cubic and hexagonal forms have slightly different band gaps and Mn emission in ZnS shifts 3–4 nm towards red when the structure changes from cubic to hexagonal.[2372] It is obvious that there is a rather wide temperature range where the both phases can coexist. The films grown from the acetate have been cubic but this may be because the acetate cannot be used above 390 °C due to thermal decomposition. Most of the crystal structure studies with the acetate have been made from the films grown at around 300 °C.[1292,1295,1322]

The structures of ZnS films grown from the alkyl compounds have not been studied thoroughly. The early paper on room temperature DMZ + H_2S process reports that the thin films (<100 nm) showed crystallinity, with the mean grain diameter increasing with increasing deposition temperature.[1306] One report deals with epitaxial monolayer per cycle growth from DMZ at 250 – 310 °C on GaAs(001) surface.[1308] The paper on the DEZ + H_2S process shows that the cubic phase dominates at 250 – 350 °C, and at 400 °C, the hexagonal structure appears in XRD.[1317] The DEZ process, although widely used, has not been described thoroughly in literature. It is obvious that it allows the deposition of ZnS at low temperatures, but at which temperature the crystallization starts is unclear. XRD is not a very good method to study crystallinity in detail, since it requires relatively large ordered areas for detection. Electron diffraction with small beam size can distinguish smaller areas. In order to know exactly when the hexagonal phase appears and how broad the two-phase temperature range is, electron diffraction or careful optical measurements should be carried out. Recently, Bakke *et al*. confirmed by TEM and optical measurements that the cubic and hexagonal phases coexist in a wide temperature range (225 – 400 °C), and traces of hexagonal phase were seen already in films deposited at 200 °C with the DEZ–H_2S process.[1320]

Grain sizes of the films have been studied by XRD and scanning electron microscopy. It seems that higher deposition temperature and thicker film result in larger grains in all processes.[1295,1306,1322,2371] In the chloride process, the tendency is stronger than in the acetate process. It is believed that because of the larger grains, the electroluminescence efficiency is better in films grown from the chloride than in those made from the acetate.[1305] In the DEZ process, the dependence of the grain size on thickness is similar to that of the chloride process.[1317] The SEM and TEM studies show that ALD ZnS films consist of columnar grains that extend through the whole thickness.[1318,2373] The grain shape seems to depend on the process, since the acetate and DEZ processes show smaller conical or columnar grains than ZnS grown with the chloride process.[1317] In films grown at low temperatures, the grains are largely aligned vertically to the sample surface, but at higher growth temperatures, the grains are more randomly oriented, as seen in Figure 16.[1320] Further on, it seems that larger thickness and higher growth temperature result in a rougher surface, as studied by atomic force microscopy (AFM).[1298,1317,1320] It has also been shown that high roughness is promoted by low nucleation density at the beginning of the film growth.

The study of epitaxial growth on single-crystal GaAs(001) shows that the substrate has an effect on the structure and morphology.[1308] However, ZnS films have usually been grown on amorphous substrates (glass, alumina-coated glass, quartz) or native-oxide-covered silicon. The beginning of the growth on the amorphous surface has not been studied carefully. The density measurements by Rutherford backscattering spectrometry (RBS) and ellipsometry have shown that the growth on glass starts mainly as amorphous and crystallization occurs after a few tens of nanometers.[1293] On an Al_2O_3 layer, ZnS is polycrystalline already at the beginning of the growth, but poor crystal quality has been reported for the first 25 nm thickness, as evaluated by luminescence measurements. AFM study at the beginning of the growth in the chloride process shows a difference between glass and mica surfaces.[1298] The nucleation takes place more easily on glass than on the atomically flat crystal surface of mica and, accordingly, the film is smoother on glass than on mica. Angle-resolved x-ray photoelectron spectroscopy (ARXPS) on the DEZ + H_2S process on Al_2O_3-covered Si wafers showed that after 100 growth cycles there is aluminum at the surface. This tells that at first small ZnS agglomerates are formed at randomly distributed nucleation sites and more than 100 cycles are needed before the agglomerates coalesce.[1317]

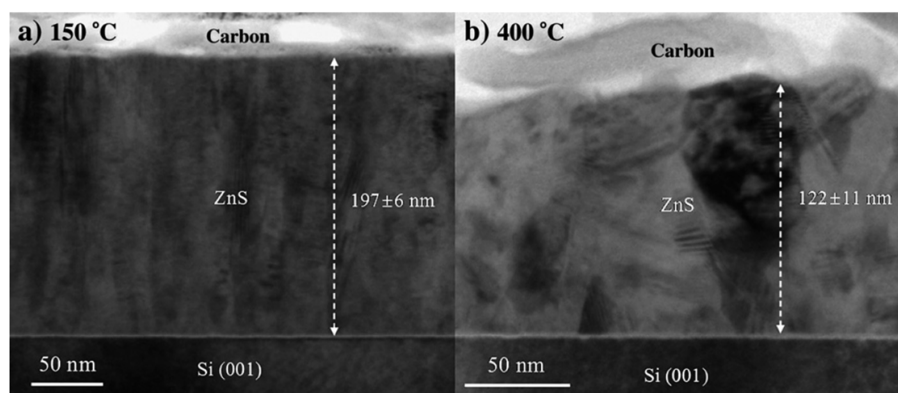

FIG. 16. Bright field TEM images of 2000 cycles of ZnS from Et_2Zn and H_2S at (a) 150 °C and (b) 400 °C. Columnar grains extend from substrate to surface in the film grown at 150 °C, while the grains are more randomly oriented in the film grown at 400 °C. Reprinted with permission from J. R. Bakke, J. S. King, H. J. Jung, R. Sinclair, and S. F. Bent, Thin Solid Films **518**, 5400 (2010). Copyright 2010 Elsevier.

D. Other inorganic ALD materials

1. General

An overview of the crystallinity of elemental films, metal phosphides and arsenides, and fluorides, grown by two-reactant ALD processes, is shown in Tables VII–IX, respectively.

Elemental films divide into metals and Si, Ge (Table VII), and most recently also Sb.[534] For metal ALD no such universal chemistries as available for metal oxides with water- and ozone-based processes, for example, have been established by now. The first clearly successful ALD metal process was based on the reduction of WF_6 with silane and later also borane. Analogous chemistry was used later also for molybdenum.[2376] For noble metals, rather general chemistry that exploits molecular oxygen as the co-reactant has been established, as will be discussed in more detail in Sec. III D 2. For copper, various attempts have been made (Table VII), but the breakthrough process is still to be found. In principle, plasma activation of either hydrogen alone or with, for example, nitrogen (NH_3 or N_2–H_2 mixture) has potential to develop to the desired universal approach to metal ALD, and indeed, promising results have been reported with many metals, including the highly challenging electropositive metals Al and Ta.[71,72,685,2124–2128] However, much effort is still needed to validate this promise and to solve potential problems, both chemistry- and reactor-related, deriving from the exploitation of plasma.

Nearly all the ALD metal films have been polycrystalline in the as-deposited form. An interesting, and potentially technologically important exception, is the amorphous tungsten films that were obtained using B_2H_6 as a reducing agent WF_6 at 300 °C.[2202] Under identical conditions, the use of Si_2H_6 as reducing agent resulted in crystalline W films. One might think that the amorphous structure would be due to higher impurity contents, but the films contained about the same amounts (ca. 5 at. %) of boron and silicon. The rest of the amorphous ALD metal films reported can be ascribed to their low deposition temperatures and/or high impurity contents.

ALD, or in this case more exactly ALE, of Si and Ge, together with the III–V compound semiconductors (arsenides and phosphides) was extensively investigated in the late 1980s and the early 1990s.[1480,2377] With Si and Ge, a substantial amount of surface chemistry studies were conducted but the film properties were not reported in similar detail. The III–V compounds gained even more effort and were thoroughly characterized with respect to both deposition chemistry and film properties. However, as there was no clear advantage compared to competing techniques, metal-organic vapor-phase epitaxy (MOVPE) in particular, and the films suffered from high carbon contamination levels, the interest toward ALE of semiconductors faded away toward mid-1990s. As noted, this research was aimed at epitaxial growth and, accordingly, the crystalline properties of the resulting films were dominated by the epitaxial effects of the substrate material.

Metal fluorides form an important class of thin film materials especially for optics because of their transparency to short UV wavelengths and low refractive index. Yet, ALD studies on metal fluorides have remained rather limited. This must be partly due to concerns related to fluorine precursor. Noting the wide use of hydrides as nonmetal precursors in general would make HF an obvious choice, but it is highly corrosive and toxic. Indeed, in the very first ALD metal fluoride studies, HF was obtained by decomposing NH_4F thermally and reacted successfully with $Ca(thd)_2$, $Sr(thd)_2$, and Zn acetate.[680] Quite recently, Pilvi *et al.* showed that TiF_4 and TaF_5 can serve as fluorine source when reacted with metal-thd precursors.[66–68,681,1534,1858,2378] TiF_4 and TaF_5 are reasonably low-volatility solids and therefore safe to handle and easy to capture from the reactor exhaust. The reaction with metal thd precursors was surprisingly complete, leaving only minor amounts of Ti and Ta residues into the films.

With respect to crystallization, metal fluoride films (LaF_3, YF_3, CaF_2, MgF_2) deposited from the corresponding metal-thd precursors and TiF_4 or TaF_5 showed similar behavior: Films deposited at 225–250 °C were only weakly crystalline and the crystallinity intensified strongly with increasing deposition temperature.[66–68,681,1534,1858,2378] This led to a substantial roughening of the films which is unfortunate with respect to the potential UV optics applications of the films. Only YF_3 could be deposited below 225 °C, down to 175 °C, and these films were amorphous to XRD and had smooth surfaces.[1534] The negative effect of the low deposition temperatures was the contamination of the films by residual elements from the precursors. The fluoride films obtained with HF were all crystalline as the deposition temperatures were 260 °C and higher.[680]

TABLE VII. Crystallinity studies of ALD element films made by diffraction or spectroscopy.[a]

Material reactant A	Reactant B	Substrate(s)	Phases		References
			Amorphous	Crystalline	
Al					
$AlMe_3$	H_2[c]	Si, SiO_2, TiN		250 °C	71 and 72
Si					
Si_2Cl_6	Si_2H_6	Ge(100)[d]		465 °C	548
$SiCl_2H_2$	H_2[c]	Ge(100)[d]		465 °C	548
$SiCl_2H_2$	H_2	H-Si(100)[d]		815–825 °C	542 and 543
$SiCl_2H_2$	H_2	H-Si(111)[d]		890–910 °C	543
Fe					
$Fe(^tBuNCMeN^tBu)_2$	H_2	SiO_2, glass		250 °C	1053
Co					
$Co(^iPrNCMeN^iPr)_2$	H_2	SiO_2, glass		350 °C	1053
$Co(^iPrNCMeN^iPr)_2$	H_2	SiO_2, WN		300 °C	1067
$CoCp_2$	NH_3[c]	Si		300 °C	1071
$CoCp(CO)_2$	NH_3[c]	SiO_2, Si		250–400 °C	1073
$CoCp(CO)_2$	H_2[c]	H-Si		125–175 °C	1076
$Co_2(CO)_8$	H_2[c]	H-Si	75–110 °C		1077
Ni					
$Ni(^iPrNCMeN^iPr)_2$	H_2	SiO_2, glass		250 °C	1053
$Ni(acac)_2$	H_2	Ti, Al, Si		250 °C	1092
$Ni(hfip)_2$	H_2	H-Si		225 °C	1094
Cu					
CuCl	$H_2 + H_2O$	Al_2O_3		425 °C	1114
CuCl	H_2-H_2O	Al_2O_3, SiO_2		375–475 °C	1113 and 1114
CuCl	H_2-H_2O	Al_2O_3[d]		400 °C	2374
CuCl	Zn	Al_2O_3		500 °C	1115
$Cu(hfac)_2$	NH_3-H_2O	SiO_2		283–302 °C	1141
$Cu(hfac)_2{\cdot}XH_2O$	formalin	TiN, TaN, Ta		300 °C	1124
$Cu(hfac)_2 + H_2 + H_2O$	MeOH	TiN, TaN, Cu, Ta, glass		300 °C	1138
$Cu(hfac)_2 + H_2 + H_2O$	EtOH	TiN, TaN, Cu, Ta, glass		300 °C	1138
$Cu(hfac)_2 + H_2 + H_2O$	^{i}PrOH	TiN, TaN, Cu, Ta, glass		300 °C	1138
$Cu(hfac)_2 + H_2 + H_2O$	formalin	TiN, TaN, Cu, Ta, glass		300 °C	1138
$Cu(hfac)_2 + H_2 + H_2O$	CO	TiN, TaN, Cu, Ta, glass		300 °C	1138
Cu(hfac)(vtmos)	H_2[c]	TiN		200–300 °C	1126
$Cu(acac)_2$	H_2	Ti, Al, glass		250 °C	1092
$Cu(acac)_2$	H_2[c]	Si, glass		140 °C	1116
$Cu(thd)_2$	H_2	SLG-Pt/Pd, SiO_2, glass		190–350 °C	1119, 1120, and 1122
$Cu(thd)_2$	H_2[c]	Au, TaN_x, SiO_2		90–250 °C	1123
$Cu(dmap)_2$	$ZnEt_2$	Si		100–120 °C	1132
$[Cu(^iPrNCMeN^iPr)]_2$	H_2	SiO_2, glass, Co, WN		190–280 °C	1053 and 1067
$[Cu(^sBuNCMeN^sBu)]_2$	H_2	Al_2O_3, SiO_2, Si_3N_4, glass, Ru, Co, Cu, WN		150–190 °C	1127
Zn					
$ZnEt_2$	H_2O	silica		200 °C	1149
Ge					
$GeMe_2H_2$	H_2[c]	SiO_2[d]		480 °C	1494
Mo					
$MoCl_5$	Zn	Al_2O_3, SLG		400–500 °C	1634
Ru					
Ru(chd)(ipmp)	O_2	SiO_2		220 °C	1641
$Ru(Cp)_2$	O_2	Al_2O_3, TiO_2, SiO_2, HfO_2, $HfSiO_x$, Ta_2O_5, carbon aerogel		275–400 °C	1643, 1646, 1647, and 1650
$Ru(Cp)_2$	NH_3[c]	TaN_x, Ta_2O_5, H-Si, SiO_2		300 °C	1649
$Ru(EtCp)_2$	O_2	Si, SiO_2, TiN, Ta_2O_5		270–300 °C	1652, 1654, and 1687
$Ru(EtCp)_2$	NH_3[c]	TaN, Si	290 °C[b]		1669
$Ru(EtCp)_2$	NH_3[c]	TiN, TaN_x, Ta_2O_5, H-Si, SiO_2, Si	100–200 °C[b]	270–350 °C	1650, 1661, 1662, 1666, and 1670
$Ru(EtCp)_2$	H_2[c]-O_2	TiO_2		300 °C	900
$Ru(EtCp)_2$	$H_2 + N_2$[c]	O-Si, SiO_2		200–230 °C	994 and 1671

TABLE VII. (*Continued.*)

Material reactant A	Reactant B	Substrate(s)	Phases		References
Ru(EtCp)(dmp)	O_2	Si, Au, Pt, Al_2O_3, SiO_2, TiO_2, TiN		230–290 °C	906, 1672, 1673, and 1677
RuCp(CpCH(Me)(NMe$_2$))	O_2	Al_2O_3		325–500 °C	1680
Ru(thd)$_3$	O_2	Al_2O_3		325–400 °C	1685 and 1686
Ru(^{t}BuAMD)$_2$(CO)$_2$	O_2	O-Si		300–400 °C	1688
Ru(^{t}BuAMD)$_2$(CO)$_2$	NH_3	WN		250–300 °C	1689
Rh					
Rh(acac)$_3$	O_2	Al_2O_3		250 °C	1694
Pd					
Pd(hfac)$_2$	formalin	Al_2O_3		200 °C	1702
Pd(hfac)$_2$	H_2	Ir, TaN		80–130 °C	1696 and 1697
Pd(hfac)$_2$	HCOCOOH	tetrasulfide SAM		200 °C	1696
Pd(thd)$_2$	H_2	Al_2O_3, SiO_2		180 °C	1684
Pd(thd)$_2$	H_2[c]	Ir, W, Si	80 °C		2375
Ag					
Ag(O$_2$C^{t}Bu)(PEt$_3$)	H_2[c]	Si, glass		140 °C	1705
Ag(hfac)(cod)	propanol	glass		110–150 °C	1811
Sb					
$SbCl_3$	(Et$_3$Si)$_3$Sb	Si, SLG		95–250 °C	534
Ta					
$TaCl_5$	H_2[c]	Si, SiO_2	25–400 °C	400 °C[b]	685, 2124, and 2165
TaF_5	H_2[c]	SiO_2		200–350 °C	2125–2127
W					
WF_6	Si_2H_6	Si		327 °C[b]	2208
WF_6	Si_2H_6	Al_2O_3		122–177 °C	187 and 309
WF_6	SiH_4	TiN, SiO_2		300 °C	2201 and 2202
WF_6	B_2H_6	TiN	300 °C		2202
Ir					
Ir(acac)$_3$	O_2	Al_2O_3, TiO_2, SS		225–375 °C	276, 832, 2240, and 2243
Ir(acac)$_3$	O_3	Si, SLG, Al_2O_3		200–225 °C	2252
Ir(acac)$_3$	O_3-H_2	Al_2O_3		165–200 °C	2246
Ir(EtCp)(cod)	O_2	SiO_2, TiN		270–290 °C	2248 and 2249
Ir(EtCp)(cod)	NH_3[c]	TiN		270 °C	2249
Ir(MeCp)(chd)	O_2	Al_2O_3		225–350 °C	2250
Pt					
Pt(MeCp)Me$_3$	O_2	Al_2O_3, Ir, Si, SiO_2, ZrO_2, $SrTiO_3$, SnO_2, borosilicate		200–300 °C	295, 1644, 2243, 2253, 2255, 2260, 2262, and 2270
Pt(MeCp)Me$_3$	O_2[c]	Si, SiO_2		300 °C	2260
Pt(acac)Me$_2$	O_3	Al_2O_3		140–200 °C	2273

[a]DMHy, dimethylhydrazine; SLG, soda lime glass; H-Si, HF-etched Si; SAM, Self-assembled monolayer; Precursors connected with +, fed in the same pulse; Precursors connected with dash, pulsed separately.
[b]Ambiguous interpretation due to reflection overlap or weak intensity.
[c]Plasma.
[d]Epitaxy.

2. Case: Ruthenium

Ruthenium is a potential high-work-function electrode material for DRAM capacitors and MOSFETs, and has therefore gained substantial interest in ALD as well. Further motivation for Ru ALD development comes from fuel cell electrodes, catalysts, and the use of Ru as a seed layer for the electrodeposition of Cu on barrier layers for IC interconnects. The first Ru ALD process exploited $RuCp_2$ and molecular oxygen as precursors.[1643] Exploitation of molecular oxygen as the other precursor for metal deposition is somewhat surprising, as O_2 is not a reducing agent, but this process is more complicated and involves oxidative decomposition of the metal precursor.[1642] Subsequently it was demonstrated that besides organometallic compounds, also β-diketonates could be used as Ru precursors, and also other noble metals (Ir, Pt, Rh, Pd) could be deposited with similar O_2-based processes. A common feature for these metals is that they are catalytically active in dissociating O_2 to atomic oxygen, which apparently is critical for activating O_2 that otherwise has turned out to be often inert in ALD. Deposition temperatures have typically been between 250

TABLE VIII. Crystallinity studies of ALD phosphide, arsenide and antimonide films made by diffraction or spectroscopy.[a]

Material reactant A	Reactant B	Substrate(s)	Phases		References
Phosphides					
GaP			Amorphous	Cubic	
$GaMe_3$	PH_3	GaP[d], GaAs[d]		500 °C	1394
$GaMe_3$	PH_3	Si[d]		450–600 °C	516 and 1392
$GaMe_3$	$P(NMe_2)_3$	SiO_2		450 °C	1395
InP			Amorphous	Cubic	
$InMe_3$	PH_3	GaAs[d]		420–580 °C	1773
$InMe_3$	PH_3	InP[d]		350–360 °C	1775 and 1776
Arsenides					
AlAs			Amorphous	Cubic	
$AlMe_3$	AsH_3	H-Si[d]		500 °C	513
$AlMe_2H$	AsH_3	GaAs[d]		470 °C	520
$AlMe_2H$	AsH_3-NMe_2H	GaAs[d]		460 °C	522
$AlMe_3$	AsH_3	H-Si[d]		500 °C	516 and 1426
$AlMe_3$	AsH_3	As[d]		500–850 °C	516
$(Me_3N)\cdot AlH_3$	$As(NMe_2)_3$	GaAs[d]		310–400 °C	527
$(Me_3N)\cdot AlH_3$	AsH_3	As-Si, GaAs[d]		150 °C	525
$(Me_2EtN)\cdot AlH_3$	AsH_3	GaAs[d]		250–650 °C	528–532
MnAs			Amorphous	Hexagonal	
$Mn(CpMe)_2$	$As(NMe_2)_3$	GaAs[d]		350–500 °C	1052
GaAs			Amorphous	Cubic	
Ga + HCl	AsH_3	GaAs[d]		350–550 °C	1397, 1399, and 1411
$GaCl_3$	AsH_3	GaAs[d]		300–450 °C	1413 and 1414
$GaCl_3$	AsH_3	GaAs		200–250 °C	1413 and 1414
$GaMe_3$	AsH_3	GaAs[d]		450–700 °C	1417, 1420, 1433, and 1438
$GaMe_3$	AsH_3	silica		400 °C	1467
$GaMe_3$	AsH_3	H-Si		500 °C	516, 1426, 1439, and 1463
$GaMe_3$	AsH_3	InAs[d]		500 °C	1394
$GaMe_3$	AsH_3-H_2[c]	GaAs		430–500 °C	1441
$GaMe_3$	AsH_3	GaP[d]		500 °C	516
$GaMe_3$	$^tBuAsH_2$	GaAs[d]		500–580 °C	1470 and 1474
$GaMe_3$	$EtAsH_2$	GaAs[d]		500–540 °C	1468
$GaMe_3$	As	GaAs[d]		370–530 °C	1434
$GaMe_3$[b]	AsH_3	GaAs[d]		370–430 °C	514
$GaEt_3$	AsH_3	GaAs[d]		385 °C	1438
$GaEt_3$	$^tBuAsH_2$	GaAs[d]		550 °C	1486
$GaEt_3$	$As(NMe_2)_3$	GaAs[d]		440–495 °C	527
$GaEt_3$[b]	AsH_3	GaAs[d]		370–430 °C	514
$GaEt_2Cl$	AsH_3	GaAs[d]		450–525 °C	1490
$Ga(C_5H_{11})_3$	$^tBuAsH_2$	GaAs[d]		430–500 °C	1491
InAs			Amorphous	Cubic	
In + HCl	AsH_3	GaAs[d]		350–550 °C	1399 and 1411
$InMe_3$	$^tBuAsH_2$	InP[d]		350–410 °C	1775, 1776, 1788, and 1789
$InMe_3$	AsH_3	InAs[d]		360–420 °C	1394
Antimonides					
AlSb			Amorphous	Cubic	
$AlCl_3$	$(Et_3Si)_3Sb$	Si, SLG		300 °C	534
GaSb			Amorphous	Cubic	
$GaCl_3$	$(Et_3Si)_3Sb$	Si, SLG	150 °C		534

TABLE VIII. (*Continued.*)

Material reactant A	Reactant B	Substrate(s)	Phases		References
			Amorphous	Cubic	
GeSb					
$GeCl_2 \cdot (C_4H_8O_2)$-$SbCl_3$	$(Et_3 Si)_3 Sb$	Si, SLG	95 °C		534

[a]H-Si, HF-etched Si; Precursors connected with +, fed in the same pulse; Precursors connected with dash, pulsed separately.
[b]Photo-assisted deposition.
[c]Plasma.
[d]Epitaxy.

and 350 °C but with *N*,*N*-dimethyl-1-ruthenocenylethylamine, i.e., a derivative of $RuCp_2$ where the alkyl substituent of the Cp-ring has an amine group, self-limiting growth was achieved up to at least 450 °C.[1680] Examples of Ru films grown from this precursor are shown in Figure 17.

Reaction mechanism studies on the $RuCp_2$–O_2 process with quadrupole mass spectrometer (QMS) showed that CO_2 and H_2O were the main byproducts of the process, and that these both were released during both precursor pulses.[1642] This implies that during the oxygen pulse not only the hydrocarbon ligands remaining from the preceding metal precursor pulse are oxidized, but also adsorbed oxygen atoms are formed on the surface to react with the next metal precursor pulse. Interestingly, QCM measurements showed that the amount of oxygen atoms left on the film surface was equal to 2–3 ML, which means that some subsurface oxygen had to be formed as well. The same and following studies have shown that a similar mechanism seems to apply to other O_2-based noble metal processes as well, except that the subsurface oxygen formation seems to be unique to Ru.

In general, the Ru films obtained with the O_2-based processes have contained only small amounts (a few at. % in maximum, often below 1 at. %) of oxygen as an impurity, showing that the atomic oxygen formed on the film surface during the O_2 pulse is almost completely consumed in the following reactions with the hydrocarbon ligands of the metal precursor. However, a combination of a high O_2 dose and low metal precursor dose has sometimes resulted in RuO_2 instead of metallic Ru, but this seems to imply that the metal precursor dose has remained short from saturation.[994] While oxides of other noble metals (IrO_2, RH_2O_3, PtO_x) have been successfully deposited at low temperatures using ozone instead of O_2,[1695,2252,2273] with Ru this is not possible because Ru and RuO_2 are etched by O_3 forming volatile RuO_4. Accordingly, while Ir, Rh, and Pt can be deposited at low temperatures using a cycle sequence metal precursor–O_3–H_2,[2246,2273] this is not applicable to Ru.

In addition to the O_2-based processes, Ru films have been grown by PEALD using ammonia plasma, but the growth rate in these processes has sometimes been modest, though rates comparable to thermal ALD have been reported, too. The plasma processes have exhibited similar nucleation delays as the O_2-based processes.[1670]

A common observation to the Ru and other noble-metal ALD processes has been a long incubation time (nucleation delay) of tens of cycles before the film starts go grow with a constant rate. This is understandable, as in these cases the surfaces on which Ru is deposited are not catalytically active for O_2 dissociation. In fact, it still remains an open question how the growth gets started on a surface that does not

TABLE IX. Crystallinity studies of ALD fluoride films made by diffraction or spectroscopy.

Material reactant A	Reactant B	Substrate(s)	Phases		References
			Amorphous	Tetragonal	
MgF_2					
$Mg(thd)_2$	TiF_4	Si		250–400 °C	66 and 67
			Amorphous	Cubic	
CaF_2					
$Ca(thd)_2$	HF	Al_2O_3		320 °C	680
$Ca(thd)_2$	TiF_4	Si		300–450 °C	67 and 681
$Ca(thd)_2$	TaF_5	Si		225–400 °C	68
			Amorphous	Rutile	
ZnF_2					
$Zn(CH_3COO)_2$	HF	$Al_2 O_3$		300 °C	680
			Amorphous	Rutile	
SrF_2					
$Sr(thd)_2$	HF	Al_2O_3		350 °C	680
			Amorphous	Orthorhombic	
YF_3					
$Y(thd)_3$	TiF_4	Si	175–200 °C	225–325 °C	1534
			Amorphous	Hexagonal	
LaF_3					
$La(thd)_3$	TiF_4	Si		225–250 °C	1858

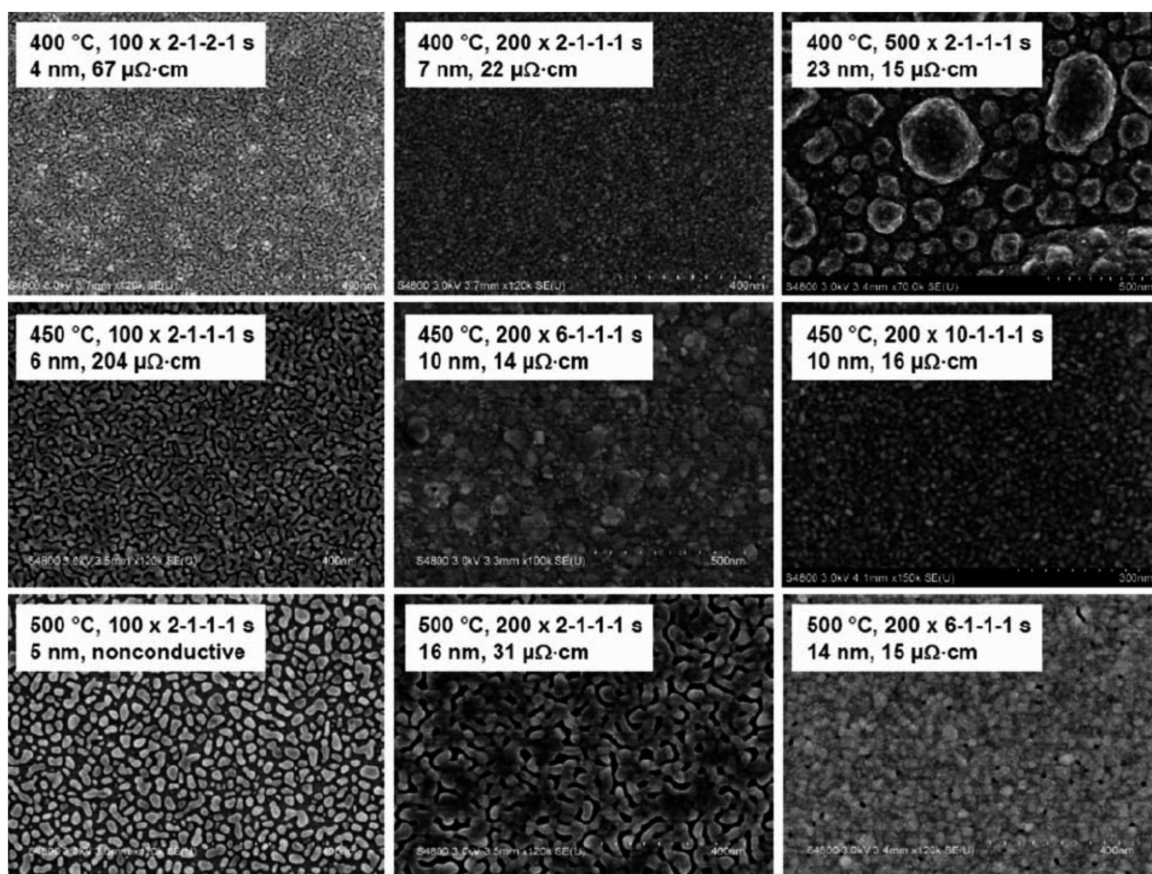

FIG. 17. Top-view SEM images of Ru films grown from $CpRuCpCH(CH_3)N(CH_3)_2$ and air at 400 °C (top row), 450 °C (middle row), and 500 °C (bottom row). The labels indicate the deposition temperature, number of ALD cycles, cycle time (Ru precursor-purge-O_2-purge), Ru thickness, and electrical resistivity. Reprinted with permission from K. Kukli, M. Ritala, M. Kemell, and M. Leskelä, J. Electrochem. Soc. **157**, D35 (2010). Copyright 2010 ECS—The Electrochemical Society.

catalyze O_2 dissociation. Most likely some minor decomposition of Ru precursor occurs first to form the first metallic Ru nuclei that then act as the catalysts. Indeed, with increasing deposition temperature the nucleation delay is shortened. Furthermore, new precursors like (η^6-1-isopropyl-4-methylbenzene)(η^4-cyclohexa-1,3-diene)ruthenium(0)[1641] and (2,4-dimethylpentadienyl)(ethylcyclopentadienyl)ruthenium (DER),[1672] which from the chemistry point of view should be somewhat less stable than the cyclopentadienyls and β-diketonates, have exhibited linear growth almost from the very beginning of the growth, yet the processes do not seem to suffer from precursor decomposition.

Based on their observation that negligible nucleation delay was observed when Ru was deposited from DER and O_2 on gold film even when gold should not activate O_2 dissociation, Kim *et al.*[1677] suggested that instead of O_2 dissociation, it would be the metal precursor adsorption that determines the initial growth. However, while the adsorption of the metal precursor is an important step, it alone does not ensure the onset of the film growth. There are several examples where the metal precursors are clearly adsorbing but no film growth is observed. For example, noble-metal oxides could have been grown at low temperatures with the more reactive co-reactant ozone, but no growth occurs with O_2 at the same temperatures from the same metal precursors.[1694,1695,2252] Therefore, a more plausible explanation is that the gold surface catalyzes the decomposition of the Ru precursor so that the first Ru nuclei form on the surface and then start to catalyze the O_2 dissociation. One should also be aware that not all the gold surfaces are the same. Especially nanostructured gold has become famous about its high catalytic activity that greatly exceeds that exhibited by single crystal surfaces. In summary, one can list the following alternatives for the onset of noble metal ALD growth using O_2 as the other precursor: (1) the surface activates O_2 by catalyzing its dissociation to atomic oxygen, (2) the surface catalyzes metal precursor decomposition to form the first metal nuclei, and (3) the first metal nuclei form by chance with non-catalyzed decomposition reaction.

A positive consequence of the difficulties in nucleation of ruthenium and other noble metals is that selective-area ALD is easier to realize with noble metals than with oxides, for example. For selective-area ALD, chosen areas of the substrate surface are covered with self-assembled monolayers (SAM) or polymers upon which the films do not nucleate and thus the films grow only in those areas that are without these passivation layers.[95,98,835,2241] Another area where poor nucleation and the resulting isolated island deposits have been exploited is nanocrystal floating gates in flash memories.[1648,1659]

All the Ru films deposited by ALD have been crystalline with the common hexagonal structure; no amorphous ALD

Ru films have been reported. The crystalline orientation is more or less random when the deposition is carried out at low temperature. As the temperature is elevated, crystallites tend to orient towards [001] direction.[1642] In PEALD Ru films, the same [001] orientation increases with the plasma power. No major differences have been observed between various Ru precursors. The substrate, in turn, may have quite an effect: The films deposited with PEALD from $Ru(EtCp)_2$ and NH_3 plasma had a random orientation on silicon but were [001] oriented on PEALD TiN film.[1670] Examples of x-ray diffractograms measured for Ru grown from *N*,*N*-dimethyl-1-ruthenocenylethylamine and air are shown in Figure 18.

IV. CRYSTALLINITY OF INORGANIC ALD MATERIALS: GENERAL TRENDS

This section discusses the general trends in the development of ALD film crystallinity as function of ALD process parameters. We have attempted to isolate the effects of different parameters, but often this is not completely possible because several parameters are interrelated, e.g., temperature, reactants, and impurities. Also, while many ALD processes share characteristics with the examples discussed here, one must remember that each ALD process (reactant pair) is unique and may behave completely differently as compared to the examples given here.

A. Effect of temperature

Temperature affects the crystallinity of all solid inorganic materials, as they undergo phase transition from amorphous to crystalline or a crystalline phase to another at characteristic temperatures. In general, the phase transition temperatures are similar for ALD films as for films made by other techniques. In addition to changes in film crystallinity, other changes occur during ALD process with increase of ALD processing temperature, as reaction and mass transport kinetics get faster. Most notably, the impurity contents in the films change, often decreasing with increasing temperature, but sometimes also increasing with temperature if decomposition of the reactants takes place. The choice of process temperature dictates also the choice of available ALD reactants, since not all reactant combinations work at all temperatures, and the reactants largely determine the impurities. The effect of temperature on crystallinity cannot therefore in most cases be completely separated from its effect on other factors.

Aluminum oxide (Sec. III A 2) is one the materials that predominantly grow in an amorphous form by ALD. At the temperatures used typically for the $AlMe_3 - H_2O$ process ($\lesssim 300\,°C$) as well as for the $AlCl_3 - H_2O$ process ($\lesssim 500\,°C$), the films are almost always amorphous. Amorphous Al_2O_3 films can be crystallized by heating. The crystallization temperature is a function of the film thickness, annealing time (and probably also the substrate): the longer the annealing time and thicker the film, the lower the temperature where crystallization is seen. As examples, a 5 nm Al_2O_3 film crystallized at about 900 °C in 1 min anneal,[151] and a 100 nm thick Al_2O_3 film between 750 and 800 °C in 30 min anneal.[228] When crystalline films grow during ALD,

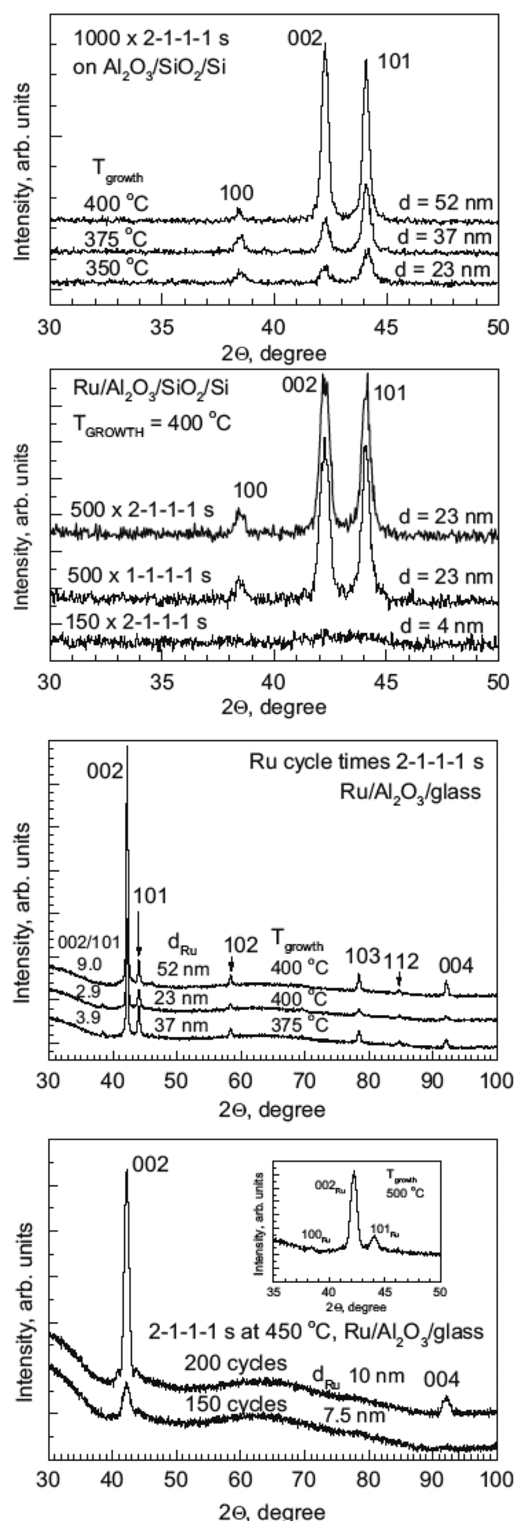

FIG. 18. Representative x-ray diffractograms of Ru films grown at different substrate temperatures, measured in GIXRD mode (uppermost panel); Ru films grown to variable thickness or with different Ru precursor pulse length, measured in GIXRD mode (the second panel); Ru films grown at different substrate temperatures to different thicknesses, measured in Θ–2Θ mode (the third panel); and thin Ru films grown at 450 °C, measured in Θ–2Θ mode (bottom panel). The inset depicts an x-ray diffractogram from a film grown at 500 °C. Ru peaks are denoted with corresponding Miller indexes. Labels denote the amounts of ALD cycles used, cycle times, growth temperatures, diffraction peak intensity ratios, and film thicknesses (*d*). Reprinted with permission from K. Kukli, M. Ritala, M. Kemell, and M. Leskelä, J. Electrochem. Soc. 157, D35 (2010). Copyright 2010 ECS—The Electrochemical Society.

the temperature needed for crystalline growth is similar or somewhat lower than the temperature needed for crystallization of thick films by annealing: For Al_2O_3 this is from approximately 700 °C upwards (Table III). In rare cases, growth of crystalline Al_2O_3 has succeeded at temperatures significantly lower than the general temperature for crystalline growth (even at 450 °C), enabled by the epitaxial growth guided by the single-crystalline substrate.[100,101]

Zinc oxide (Sec. III A 4) gives an example of a material that almost always grows in a crystalline form. This is the case for films deposited at 25 – 600 °C (Table III). Furthermore, the crystalline films always show a hexagonal structure. ZnO is therefore an example of a material where changes in process temperature do not lead to significant changes in crystallinity or phase. Process temperature affects the crystallite orientation in the film, however, as discussed in detail in Sec. III A 4.

Titanium nitride (Sec. III B 2) exemplifies a material that forms both amorphous or crystalline films, depending on the process details. Most processes carried out at "low" temperatures around 200 °C have resulted in amorphous TiN films, and at "high" temperatures around 400 °C to crystalline TiN films. However, as discussed in more detail in Sec. III B 2, it is not the process temperature itself but mainly the carbon impurities, which have determined the amorphous/crystalline nature of the films. Alkylamides can be used only at "low" temperatures because of their thermal decomposition, and halides at "high" temperatures because of thermodynamic requirements of the reactions. The films grown from alkylamides always contain significant amounts of carbon impurities, and it is the impurities, not the ALD temperature itself, which have led to the amorphous TiN films.

Zinc sulphide (Sec. III C 2) is an example of a material where temperature affects the crystallinity. All ZnS films studied have been crystalline. Cubic phase is obtained at low temperatures, at medium temperatures cubic and hexagonal phases coexist, and at higher temperatures hexagonal phase dominates. The transition from the cubic to the hexagonal phase takes place at around 400 °C. In this case, ALD temperature also affects the grain size: The higher the temperature, the larger the grains.

For titanium dioxide, both amorphous and crystalline films have been reported. $TiCl_4$ and H_2O are the reactants first introduced for the growth of TiO_2,[689] and still remain among the most frequently used reactants. Aarik *et al.*[727] have thoroughly studied the reaction between $TiCl_4$ and H_2O. They observed by electron diffraction amorphous TiO_2 films at temperatures below 165 °C, anatase structure at 165 – 350 °C, and rutile structure dominated at temperatures above 350 °C.[727] Besides the temperature itself, an additional explanation for the amorphous structure formed at low temperatures may be that chlorine residues hinder the crystallization. The reason for the existence of rutile structure at such a low temperature remains open; the surface intermediates formed and their mobility may favour the growth of rutile. The same group observed also an anomalous effect of temperature on the ALD of TiO_2: A significant increase in growth rate, roughening, and decrease in refractive index occurred when growth temperature was increased from 150 to 225 °C, a behaviour attributed to crystallization of the film.[744] The crystallization temperature of TiO_2 films is similar regardless of the titanium precursor. With alkoxide (methoxide, ethoxide, isopropoxide) precursors, crystalline anatase together with amorphous phase has been observed at 250 °C, while films deposited at 200 °C have been completely amorphous.[830,839,847] It is evident, therefore, that for TiO_2, higher temperature promotes crystalline growth, but it is not totally clear whether this is a direct consequence of the temperature or does the decreasing impurity content with increasing temperature dominate.

Temperature can also affect the preferred orientation of the films. For example, TiN films grown from TiI_4 and NH_3 showed [100] orientation when deposited at 400 and 425 °C, but changed to [111] orientation when the growth temperature was 450 – 500 °C.[968] This change was suggested to be due to a change in steric hindrances caused by the iodido ligands in the TiI_x surface species formed during the TiI_4 pulse. At low temperatures the steric hindrance was thought to be stronger and thereby prevent the formation of the energetically favorable (111) close packed arrangement, which consists of alternate Ti and N layers. At 450 °C and above, fewer iodido ligands seemed to remain incorporated in the TiI_x chemisorption layer, as both the orientation changed and growth rate increased. Similar behavior has been reported also for ZnO. At low temperature, [100] orientation is found most often whereas at high temperature [001] becomes dominant. The temperature at which the orientation is changed depends on the reactants as well as on other process parameters.

B. Effect of reactants

It is sometimes considered that certain reactants give certain crystalline phases. It is, however, difficult to separate the effect of reactant alone from the effect of temperature on crystallinity: Most ALD reactants have a characteristic range of temperatures where they can be employed, and these temperature ranges are often different for different types of reactants. Another interrelated parameter strongly affecting crystallinity is the film impurities.

One example of an indirect effect of the reactants on the crystallinity is the deposition of V_2O_5 from vanadyl-tri-isopropoxide and H_2O or O_2 at 150 °C by PEALD. When H_2O-plasma was used as reactant, amorphous film resulted, while O_2-plasma led to a crystalline film.[1010] The primary reason why the different reactants led to different crystallinity was their effectiveness for removing the ligands of the metal reactant. Water was not sufficiently reactive and the H_2O-based V_2O_5 films contained 22 at. % C films, while oxygen plasma effectively combusted the ligands and the film was free from carbon impurities.[1010] The purity of the film allowed the crystallization.

There are several other examples where certain classes of reactants lead to amorphous films, while others lead to crystalline films. In the ZrO_2 case study (Sec. III A 3), it was pointed out that films grown from halide reactants are generally crystalline when the film thickness is above a certain threshold, while films grown from alkoxides, alkylamides,

silylamides and aminoalkoxides are generally amorphous throughout. Similarly, the TiN case study (Sec. III B 2) revealed that films grown from the halide reactants are crystalline, while films from the alkylamides are amorphous. In both cases, the amorphous nature of the films made from the metalorganic precursors is most likely accounted for by their carbon impurities. One must note, however, that also the growth temperature was lower with the alkylamides than with the halides.

In addition to defining whether films are crystalline or amorphous, the choice of reactants may affect the phase, orientation and grain size of a crystalline film, and thereby greatly influences the properties of the films. An example of the effect of reactants on the preferred orientation is the addition of Zn to the deposition of nitrides from chloride reactants. TiN films grown from $TiCl_4$ and NH_3 at 500 °C were of cubic TiN and showed [100] as the preferred orientation, while addition of Zn led to [111] as the preferred orientation.[935] Similar to the above-cited temperature effect on the $TiI_4 - NH_3$ process, also here relaxed steric hindrance caused by removal of chlorido ligands with zinc was thought to enable the formation of the energetically favourable [111] orientation. NbN films grown from $NbCl_5$ and NH_3 at 500 °C showed a significantly higher grain size and increased AFM rms roughness when zinc was added as a reducing agent.[935] ZnS films grown by the acetate and DEZ processes show smaller conical or columnar grains than ZnS grown with chloride process.[1317]

C. Effect of impurities

Impurities are one of the main factors influencing the crystallinity of ALD-grown films. The effect of impurities is interlinked with other ALD parameters: the reactants, temperature, the use of plasma, and sometimes also the substrate.

In the TiN case study (Sec. III B 2), the effect of impurities on crystallinity was investigated in detail for various types of processes (halide-based, alkylamide-based) and for various impurities (Cl, C, H). It was found that impurities indeed strongly affect the amorphous/crystalline nature of the films. Not all impurities were alike, however. Rather high chlorine and hydrogen impurities (up to 7.7 at. % Cl and 16 at. % H) could still be tolerated and resulted in crystalline films. Already moderate carbon concentrations (a few atomic percents), in contrast, resulted in weakly crystalline or completely amorphous films. The TiN case therefore indicated that C impurities effectively prevent crystallization, while Cl and H do not.

The deposition of V_2O_5 from vanadyl-tri-isopropoxide and H_2O or O_2 gives another example of the effect of carbon impurities on the growth. When the growth was done at 150 °C, H_2O was used as reactant and the growth was by ALD or PEALD, amorphous film resulted, but when the non-metal reactant was O_2-plasma, PEALD-grown film was crystalline.[1010] The ALD and PEALD-grown H_2O-based V_2O_5 films contained 6.5 and 22 at. % C, respectively, while the film grown with O_2-plasma was free from carbon impurities according to x-ray photoelectron spectroscopy (XPS).[1010] Obviously, it is the high carbon impurity content that prevented the crystallization of V_2O_5 when H_2O was used as a reactant.

D. Effect of plasma enhancement

The use of PEALD processes brings additional energy to the surface processes in the form of radicals as reactive species, and further energy may be provided by electrons, ions and photons coming from the plasma in variable amounts depending on the plasma configuration and operation pressure. PEALD is used instead of ALD mainly either to enable completely new processes, which would not be operational by thermal ALD at all, or to lower the deposition temperature of processes where thermal ALD also works. For films grown at relatively low temperatures by thermal ALD and PEALD, those grown by PEALD often contain less impurities. The deposition of pure metals such as Al, Ti, and Ag is an example of PEALD as an enabling technology. The lowering of the deposition temperature of the $TiCl_4 - NH_3$ process down to 100 °C, again, is an example where the use of plasma enhancement allows the process to operate at a significantly lower temperature than possible by thermal ALD. The main drawbacks of PEALD compared to thermal ALD are the limited film conformality[42] and the more complicated reactor design. Defect generation by energetic photons is possible, too.[2379]

For thermal ALD and PEALD processes carried out from the same or similar reactants at the same (nominal) temperature, PEALD may result in films with a decreased impurity concentration, and thereby allows crystallization. The additional energy may also lead to increased local temperature and thereby induces crystallization. There are examples where PEALD has resulted in crystalline films while thermal ALD results in amorphous films, but as well examples where PEALD has not influenced the film crystallinity. When at the limit between amorphous and crystalline films, the plasma configuration may have an effect, too: Kim *et al.*[2059] compared remote- and direct-plasma PEALD of HfO_2 from $Hf(NEt_2)_4$ and O_2-plasma at 250 °C, and observed that while the 2-3 nm thick films deposited with the remote-plasma were amorphous, those deposited with the direct-plasma were already partially crystallised. This difference was attributed to the additional energy provided by the plasma species, the flux of which is more intense in the direct plasma configuration than in the remote plasma.

The following gives examples where certain PEALD films are crystalline, while comparable films grown by thermal ALD (or other comparable PEALD processes) are amorphous. The deposition of V_2O_5 from vanadyl-tri-isopropoxide and O_2-plasma at 150 °C is one such example. When H_2O was used as reactant and the growth was by ALD or PEALD, amorphous films resulted, but when non-metal reactant was O_2-plasma, the PEALD-grown film was crystalline.[1010] This difference was related to the fact that carbon impurities prevented crystallization of the films grown using H_2O: the ALD and PEALD-grown H_2O-based V_2O_5 films contained 6.5 and 22 at. % C, respectively, while the PE O_2-grown film was free from carbon impurities according to

XPS.[1010] A second example is the $Ti(NMe_2)$-based process to deposit Ti(C)N by PEALD. While all other investigations referred to in Table V using alkylamide reactants by ALD or PEALD resulted in amorphous films, in one investigation the authors had managed to tune the PEALD process so that crystalline, dense, and conductive film was obtained.[995] A third example is the growth of TiO_2 at 110 °C using the $TiCl_4$ reactant. A PEALD process using $TiCl_4$ and O_2 plasma resulted in a crystalline TiO_2 film,[818] while a corresponding thermal ALD process with H_2O resulted in amorphous films at such low temperature.[228,727,814]

The case studies presented in this work contained two examples, where the use of PEALD has resulted in amorphous thin films, similarly to the corresponding thermal ALD processes. The $AlMe_3$–O_2 PEALD processes to deposit Al_2O_3 resulted in amorphous films at $25-300$ °C.[432] To obtain crystalline Al_2O_3 films by thermal ALD, about 700 °C is required (Table III). Obviously, the additional energy provided by the plasma process does not correspond to several hundreds of degrees temperature increase in thermal ALD. Another example is the $Ti(NMe_2)_4$-based processes to deposit TiN, where PEALD processes most often are reported to deposit amorphous material, similarly as the thermal processes.[988,989] This process is somewhat special, though, because the decomposition of the metal reactant makes the process to deviate from the principles of ALD.[976]

E. Effect of substrate

The outermost surface of the substrate has a strong influence on ALD growth, since it determines how the growth starts. Suitable adsorption sites are needed for growth to initiate—if there are none, no growth occurs. The GPC does not need to be constant during the first cycles or tens of cycles of ALD. The way the GPC varies with the number of cycles in the beginning of the growth reveals details of the substrate–reactant interaction, and division of processes to linear growth, substrate-enhanced growth, substrate-inhibited growth of type 1 and substrate-inhibited growth of type 2 has been proposed.[2,163] Type 2 substrate-inhibited growth has been related with island growth, where growth starts from initial nuclei scattered on the surface.[163,2380] Island growth and difficult nucleation are common in ALD processes, especially when the substrate and film are of different types (metal on oxide, nitride on oxide, oxide on H-terminated Si, etc.). An extreme case of nucleation problems is used in area-selective ALD growth, where pre-determined parts of the surface have been passivated with a non-reactive surface termination to block the growth from those parts completely.

The effect of surface groups and their amount on substrate surface on the beginning of ALD growth has been widely studied when depositing high-κ oxides (ZrO_2, HfO_2) on silicon and/or oxidized silicon surfaces. The number of reactive OH groups differs significantly on H-terminated and fully hydroxylated silicon oxide surfaces. For reaction between $HfCl_4$ and H_2O, it has been shown that at 300 °C the hafnium amount deposited in one cycle is 0.3–0.4 Hf/nm^2 on H-terminated Si and 3.7–3.9 Hf/nm^2 on fully hydroxylated SiO_2 surface.[1961] The difference in nucleation is reflected in the structure and properties of the HfO_2 films. Compared to good quality continuous films deposited on SiO_2 surface, HfO_2 films deposited on H-terminated Si surfaces show a non-uniform, island-like morphology and poor electrical properties due to poor nucleation.[1892]

In some cases, the substrate determines whether film grows in an amorphous or crystalline form. The deposition of HfO_2 films from $HfCl_4$ and H_2O at 300 °C is an example of this. Crystalline HfO_2 films of ~20 nm thickness with columnar grains of ~8 nm diameter were observed on Si when it was terminated with 0.5 nm of thermally grown SiO_2, while termination with 0.55 nm of chemically grown SiO_2 (O_3-based aqueous process) led to an amorphous film.[1893] Another example is the deposition of $TiO_2-Al_2O_3$ laminates using the $TiCl_4-H_2O$ process at 200 °C. The laminate stack consisted of 20 nm films of TiO_2 and Al_2O_3 films, five of each, starting with TiO_2.[207] The first TiO_2 layer grown directly on Radio Corporation of America (RCA)-cleaned Si was partly crystalline, while all the TiO_2 layers grown on Al_2O_3 in the process sequence were amorphous.[207] In cases where a certain choice of substrate leads to amorphous growth, it is likely that the film thickness also plays a role: For significantly thicker films, the amorphous films might turn to crystalline.

For growth of crystalline films, the substrate will often guide the size of the grains. Crystalline ALD films frequently show columnar grains that extend through the whole film thickness. The first cycles of the ALD growth define how the initial nuclei are located, and this will affect the characteristics of the crystalline film even after thousands of cycles. The denser the initial nuclei are located, the smaller the grains will be, while fewer initial nuclei lead to larger grains, as the columnar crystals need to grow larger before their coalescence. Obviously, there are possibilities for tuning the surface preparations to obtain the desired grain size of a crystalline film. As an example, the Pt nanoparticle size from $MeCpPtMe_3$ and O_2 at 270 °C could be controlled by choosing the substrate (SiO_2 vs. Al_2O_3) and other process parameters (cycle numbers, exposure time).[2268] In that work it was also shown that the initially obtained Pt nanoparticle distribution and grain size were stable during annealing, unless the temperature needed to initiate diffusion of Pt particles (1000 °C) was exceeded.[2268] Another example is the growth of ZnO on sapphire, where the initial crystal size was controlled by the number of ALD cycles and annealing conditions, and growth was continued on pre-formed nuclei.[2316] In the deposition of ZnS, glass substrate resulted in better nucleation and, according to AFM, smoother films than mica substrate.[1298] In XRD, the films on mica showed dramatically more intense reflections, which was most probably due to their larger and more strongly oriented grains.

In some cases, the substrate dictates which crystalline phase is formed. This may lead to the growth of a certain phase at a temperature lower than generally expected, and is often related to epitaxy. The growth of TiO_2 on SnO_2 is an example of this: The SnO_2 substrate guided the growth from $Ti(^iOPr)_4$ and H_2O_2 to result in rutile at 250 °C,[883] while anatase is commonly observed at this temperature. The growth

of Al_2O_3 from $AlCl_3$ and O_2 is another example: Epitaxial growth of α-Al_2O_3 was observed on sapphire at 600 °C and on single-crystal Nb at 450–500 °C,[100,101] while amorphous alumina would be expected at these temperatures in the absence of epitaxy.

Many examples can be found from the literature of the effect of substrate on the preferred orientation of polycrystalline films. For example, the $TiCl_4 - H_2O$ process to deposit TiO_2 resulted at 250–350 °C in randomly oriented polycrystalline anatase on HF-etched Si, KBr and RCA-cleaned Si,[207,751] while strongly preferred orientation and partial epitaxy were observed on MgO(100).[757] Zinc oxide grows in general in crystalline form, and with $ZnCl_2$ and O_2, epitaxial growth of zinc oxide was found on both sapphire[1153,1154] and GaN[1155] at temperature range 450 − 550 °C. The deposition of Ru is yet another example of the effect of substrate on the orientation of polycrystalline films: Hexagonal Ru films deposited with PEALD from $Ru(EtCp)_2$ and NH_3 plasma had a random orientation on silicon but were [001] oriented on PEALD TiN film.[1670]

On the basis of the examples in the paragraphs above, it is clear that the substrate can influence the evolution of crystallinity in ALD films in many ways. A natural consequence of such influence is that the steady-regime GPC obtained for an ALD process—defined by the reactants and the ALD temperature—may differ for two substrates, even when all experimental growth parameters are identical. Experimentally, such differences in GPC have been observed, for example, for the $TiCl_4 - H_2O$ and $TiF_4 - H_2O$ processes on various glass substrates.[687,724] Also theoretical modelling has shown that the GPC can differ on two substrates at least because of surface topography differences (and consequently differences in the effective surface area) originating from differences in film nucleation density,[2380] differences in the shape of the growing crystallites,[38] and differences in the GPC on the various facets of crystalline grains.[39,40]

F. Effect of film thickness

In the growth of crystalline films, film thickness affects the crystalline properties of the films. The effect of film thickness on crystallinity is often interlinked to other factors, such as temperature and substrate.

Thinner films of a given material may appear amorphous and turn to crystalline after a given thickness. As discussed in detail in Sec. III A 3, ZrO_2 is one of the materials whose deposition starts in an amorphous form, while crystalline ZrO_2 is observed after a certain thickness. This thickness depends on the reactants and temperature, and maybe in some cases also the substrate. Aarik *et al.*[1544] showed that for $ZrCl_4$ and H_2O reactants, the thickness where ZrO_2 crystallinity is observed is 100 nm at 185 °C, and it decreases with increasing deposition temperature to about 1 nm at 600 °C (Figure 11). In the TiO_2 deposition from $TiCl_4$ and H_2O, it has been shown that on RCA-cleaned Si the films are originally deposited in amorphous form, but when the films are made thicker, the films crystallize throughout, leading to grains an order of magnitude larger than the film thickness.[207] Similar through-crystallization of an amorphous film operates on an Al_2O_3-coated surface, and grains with diameter of approximately 1 μm have been observed for a TiO_2 film thickness of about 20 nm.[814]

Increasing thickness may also transform a crystalline material from one phase to another. The growth of ZrO_2 from $ZrCl_4$ and H_2O is an example of such behavior. According to the experimental details reviewed in Sec. III A 3, the first phase to appear is typically cubic or tetragonal; with increasing film thickness, the tetragonal phase starts to dominate; and for still thicker films, the monoclinic phase appears. This behaviour is due to a competition of surface and bulk energy contributions.

G. Grain size of ALD films

A general rule of thumb says that grain size of crystalline thin films should not exceed the film thickness. Most ALD processes probably obey this general rule. Of the case studies of this work, the ZnO process (Figure 12), TiN process (Figure 14), and ZnS process (Figure 16) are examples, where columnar grains are observed, where the width of the columns is smaller than the film thickness. In a separate work, the deposition of ZrO_2 and HfO_2 from alkylamides and water was investigated, concluding for thicknesses up to about 100 nm that the crystal diameter was slightly less than the film thickness, and the average crystal diameter was less than half of the film thickness.[1602] These experimental observations closely resemble the simulation results by Nilsen *et al.*[37–40] for the development of grains from crystalline seed objects during ALD, a summary of which is shown in Figure 19.

In some cases, the grain size of polycrystalline ALD films has slightly exceeded the film thickness. In Figure 10, ZrO_2 crystallites are seen that are somewhat wider than the film thickness. Another example of the grain size modestly exceeding the film thickness is a recent TiO_2 study, where for films up to 20 nm thickness grown on RCA-cleaned Si and thermal SiO_2, the grain size measured by AFM exceeded the average film thickness by about 20 nm.[814] Island growth[162,163,2380] might be one candidate to explain such behaviour, although this should be looked into in detail and there may be other explanations, too.

There are also cases where the grain size of polycrystalline ALD films has exceeded the average film thickness significantly, being several times or even order(s) of magnitude larger. The ruthenium case study gives one example of such behavior. The average grain size was on the order of 100 nm for a film whose average thickness was about 20 nm, the largest grains exceeding the lateral size of 200 nm (Figure 17). The film topography differs from those of the simulations for ALD (Figure 19). One particular choice made in the simulations was that surface diffusion was not allowed.[37–40] While this choice most likely is valid for most processes, for the elemental Ru films it will not be, and the large grain size in the Ru case probably is explained by the surface diffusion of the metallic species. Another example where the crystallite size significantly exceeds the film thickness is the growth of TiO_2. For example, the deposition of ~20 nm TiO_2 from $TiCl_4$ and H_2O on RCA-cleaned Si at 250 − 300 °C resulted

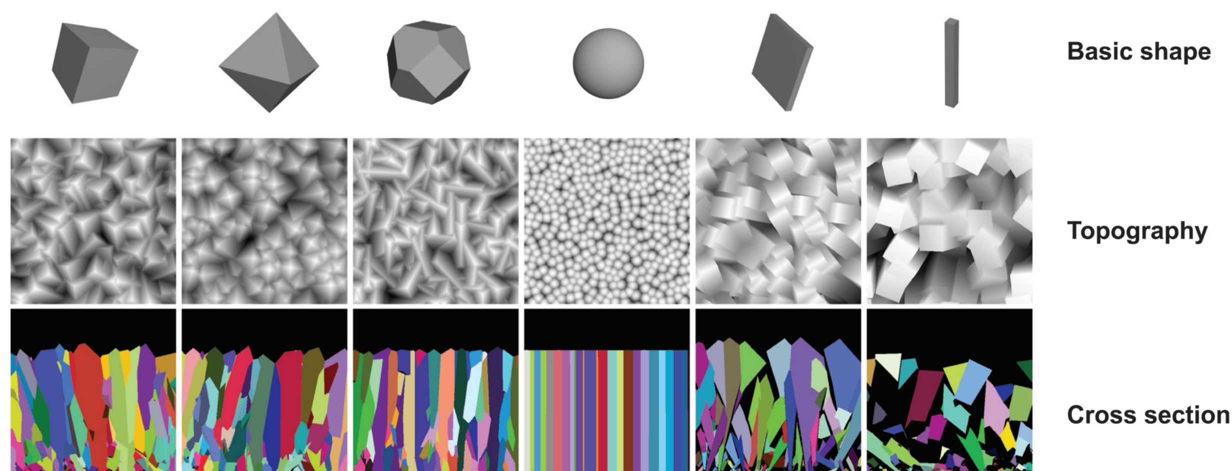

FIG. 19. Pictorial summary of the simulation results by Nilsen *et al.*[37–40] for the growth of crystalline ALD films from seed objects of various kinds, with respect to the topography and cross-section, for films of a similar simulated thickness. The porosity indicated for films made of platelets and needles is exaggerated. Reprinted with permission from O. Nilsen, O. B. Karlsen, A. Kjekshus, and H. Fjellvåg, Thin Solid Films **515**, 4550 (2007). Copyright 2007 Elsevier.

in anatase crystals >200 nm in diameter.[207,228,814] In the same process on an Al_2O_3 surface, the crystallites were still larger,[814] an example of which is shown in Figure 20. Also the corresponding ethoxide-based TiO_2 process resulted in large grains.[837] In the TiO_2 case, the large crystallites are explained to result from the crystallization of the originally amorphous film during the growth of a thicker film.[207,814] A further speciality regarding $TiCl_4$-based processes is the fact that rutile TiO_2 crystallites up to more than a micrometer in diameter have been observed after one single $TiCl_4$ reaction on amorphous high-surface-area silica.[723] The generation of gaseous short-lived OH-containing intermediate species such as $TiCl_{4-x}OH_x$ has been proposed to explain the observation.[723,2381,2382]

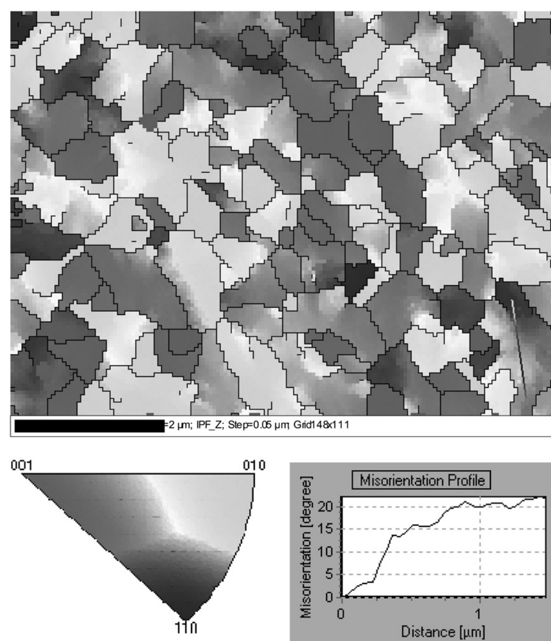

FIG. 20. Electron back-scattered diffraction inverse pole figure map for TiO_2 deposited on Al_2O_3 at 250 °C in 500 cycles. A misorientation profile is shown for a grain at the bottom right of the image; the scale bar is 2 μm. The film thickness is about 20 nm and the average grain size about 0.9 μm. Reprinted with permission from R. L. Puurunen, T. Sajavaara, E. Santala, V. Miikkulainen, T. Saukkonen, M. Laitinen, and M. Leskelä, J. Nanosci. Nanotechnol. **11**, 8101 (2011). Copyright 2011 American Scientific Publishers.

H. Schemes for the initial growth of crystalline grains in ALD

At least three general schemes can be identified from the ALD literature which proposed to describe the initial stages of growth of crystalline nuclei in ALD. These schemes are summarized in Figure 21. All schemes have their specific features regarding the size of the resulting crystalline grains, extent of crystallinity of the resulting film, and the related growth curve characteristics.

Scheme I refers to a growth of an initially amorphous film, in which crystalline nuclei form randomly. Crystalline grains grow when more material is deposited. The material landing on the crystalline nuclei adopts the existing crystalline structure, and the material landing on amorphous regions remains amorphous. This scheme was visually proposed by Hausmann and Gordon.[1602] In this scheme, the size of the resulting crystalline grains increases steadily with the film

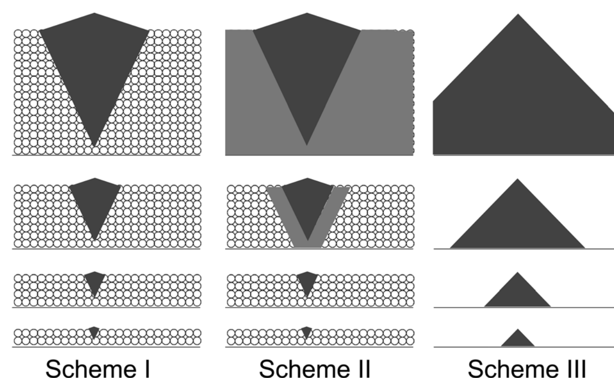

FIG. 21. Schematic illustration of different schemes for growth of crystalline grains in ALD. Scheme I: growth of crystalline grains from initial crystalline nuclei embedded in an amorphous film. Scheme II: growth of crystalline grains from initial crystalline nuclei embedded in an amorphous film, and crystallization of the amorphous regions next to the grains. Scheme III: growth of ALD film directly in crystalline form, starting from separate crystalline nuclei.

thickness until the neighboring crystals touch each other. The crystallite size should not exceed the film thickness.[1602] Close to the substrate, the ALD film is mostly amorphous, and the film crystallinity increases with increasing thickness. Since in this scheme the growth of the film occurs everywhere on the surface, the ALD growth curve (material deposited vs. cycles) is approximately linear, except perhaps for slight variations due to difference in GPC on amorphous and crystalline regions of the film. This scheme is in accord with the experimental results obtained for the growth of ZrO_2 and HfO_2 films from the alkylamide reactants.[1602]

Scheme II can be considered as an extension of Scheme I. In addition to the crystal growth of Scheme I, crystallization of the previously deposited amorphous film next to the grains occurs. This scheme eventually leads to a fully crystalline film, although growth starts as amorphous. The grain size is not limited by the film thickness, but rather dictated by the distance between the randomly formed crystalline nuclei. This scheme was recently proposed by Puurunen *et al.*[814] Similarly as in Scheme I, the growth curve should be rather linear, with minor deviations from linearity caused by differences in GPC on amorphous and crystalline material. This scheme is in accord with the experimental results for the growth of TiO_2 from $TiCl_4$ and H_2O on various substrates.[207,228,727,814] TiO_2 crystallites with lateral size of even a micrometer have been observed for Al_2O_3 substrate for TiO_2 film thicknesses in the range of tens of nanometers.[814] The crystallization of as-deposited amorphous films through high-temperature annealing shares characteristics with this scheme, as there also, crystallites order(s) of magnitude larger than the film thickness is(are) sometimes observed.[1670,2383]

Scheme III assumes that growth starts only at specific sites on the surface as crystalline nuclei. With increasing deposition, the grains grow from the initial nuclei. During the initial phase of the growth, only part of the substrate is covered with ALD material. With increasing deposition, neighbouring grains touch each other, coalesce, and finally form a continuous film. The film is fully crystalline at all phases of growth. The crystallite size increases with the number of cycles, being typically larger than the average film thickness. Here, the film is not uniform, and the maximum film thickness measured from the top of the crystallites is significantly higher than the average film thickness calculated from the amount of material deposited per unit surface area. This scheme formed the basis of the simulations by Nilsen *et al.*[37–40] In the beginning of the growth, where the nuclei are freely expanding and have not touched each other yet, the growth curve is strongly non-linear. The growth in this case corresponds to island growth, where the average film thickness has a third-order relationship on the number of cycles in the initial growth regime before coalescence.[163,2380] After coalescence, the growth curve should be roughly linear, similarly as in the other schemes.

Growth of crystalline film through the Schemes I–III results in characteristically different crystallites and ALD growth characteristics (growth curve). While none of these simple schemes will be able to explain all the observations encountered in experimental ALD investigations, they will be useful in analyzing the type of growth in question. The real grain growth may then occur through one of these schemes, a combination of them, or completely another mechanism.

V. CONCLUSION

In this article, we have overviewed the existing ALD processes for growth of inorganic materials, reviewed the crystallinity of such films grown by ALD, explored the trends in crystallinity for illustrative material cases, and discussed general trends in the evolution of the crystallinity of ALD films. Most of the material has been tabulated for easy reference.

To simplify, the following trends can be identified regarding the crystallinity of inorganic ALD films. The higher the ALD temperature, the thicker the film, and the purer the resulting material, the more likely the resulting film will be crystalline. The use of plasma enhancement increases the probability of depositing a crystalline film, but does not guarantee it. Polycrystalline ALD films consisting of columnar grains are typical, the extent of randomness of the orientation depending on the substrate and temperature. The crystalline grain size is most typically related to the film thickness, although in some cases it can be order(s) of magnitude larger than the film thickness. Each ALD process is unique, however, and particular ALD processes may contradict some, several or even all of these simplified trends.

ACKNOWLEDGMENTS

Dr. Kai-Erik Elers, VTT, is thanked for helpful discussions related to TiN growth. Funding for writing this review came from the ALEBOND Tekes project (Dnro 3544/31/07) and from the Finnish Center of Excellence on Atomic Layer Deposition by the Academy of Finland.

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