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Low Temperature MOCVD-Processed Alumina Coatings

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Abstract. We first present a Review about the preparation of alumina as thin films by the technique of MOCVD at low temperature (550°C and below). Then we present our results about thin films prepared by the low pressure MOCVD technique, using aluminium tri-isopropoxide as a source, and characterized by elemental analysis (EMPA, EDS, ERDA, RBS), FTIR, XRD and TGA. The films were grown in a horizontal, hot-wall reactor, with N_2 as a carrier gas either pure or added with water vapour. The deposition temperature was varied in the range 350-550°C. The films are amorphous. Those prepared at 350°C without water added in the gas phase have a formula close to AlOOH. Those deposited above 415°C are made of pure alumina Al_2O_3. When water is added in the gas phase, the films are pure alumina whatever the deposition temperature.

Introduction

Thin films and coatings of alumina are materials of major technical interest for numerous applications: optical and microelectronics components, wear resistance, catalysis and catalyst support, protection against corrosion and high temperature oxidation. We have been interested in using the MOCVD technique (chemical vapour deposition from a metal organic source) to deposit alumina films both for catalyst supporting and for protection against corrosion and oxidation. Our first results [1] lead us to systematically study the MOCVD of alumina at moderate temperature, using aluminium tri-isopropoxide (ATI) as a source. Following Aboaf's pioneering works [2], most of the MOCVD preparations of alumina thin films mention aluminium alkoxides as sources. ATI has been the most used one [2,7,28-46,48-52] as it is easy to use and despite problems due to ageing.

In this paper we present a short Review about the MOCVD preparations of alumina from ATI. Most of the reported depositions have been made in the temperature range 200-500°C. To make the presentation clear, deposition with a hot-wall reactor and deposition with a cold-wall reactor will be presented separately. Then we will present recent results obtained in our group, using a hot wall reactor, with N_2 as a carrier gas and with or without water vapour as a gas reactant.

MOCVD of Alumina from ATI: a Review

ATI has an oligomer structure mainly consisting of tetramer molecules [3-6]. Once melt, it remains liquid far below the melting temperature, so that it can be used as a liquid precursor. However, it has the inconvenience to transform into less volatile species on ageing and on being heated. Jones et al. proposed to generate it in the reactor by reacting trimethylaluminium and isopropanol in the vapour phase [7]. Alternative complexes have also been proposed: aluminium tri-(2-ethylhexanoate) [8], aluminium tri-(sec-butoxide) [9-13], aluminium (ethylacetoacetate)-di-isopropoxide [14], aluminium triacetylacetonate [15-22], dialkyl aluminium acetylacetonate [23], dialkyl aluminium alkoxides [24-27].

The MOCVD of Al_2O_3 from ATI was first used to coat thermally fragile semiconductor materials with insolating films in transistor devices. Films of Al_2O_3 were claimed to be deposited at

420°C [2], 400-500°C [28-30], 400°C [31], 350-500°C [32], 350-400°C [33], below 330°C [34], 300°C [35], 250-420°C [36], 250-400°C [37]. Low-temperature deposition (325-500°C) from ATI was also used for preparing graded-index aluminium oxide thin films [38,39].

Hot-Wall Reactor MOCVD. Aboaf [2] used a hot-wall reactor to deposit alumina on silicon for passivating electronic devices. The carrier gas was O_2 or N_2, or a (90 N_2 + 10 H_2) mixture under atmospheric pressure. Films deposited at 420°C were transparent and non porous. Their electron diffraction pattern showed diffuse rings. At a growth rate higher than 100 Å/min the quality of the films deteriorated (hazy looking oxides with a very porous structure). The films prepared at 420°C were hard but covered with fine particles produced by gas phase reactions, while those prepared at 270°C were soft and smooth. Following the authors, for the films prepared at 420°C "*infrared measurements did not show any OH absorption band after deposition. No OH band was detected after treating Al_2O_3 specimens for 22 days, at 25°C, in a 100 % relative humidity ambient*". However the author noticed differences in electrical behaviours, optical indices and etching properties between films grown in N_2 and in O_2, what he tentatively explained "*in terms of an oxygen sorption mechanism. During deposition of Al_2O_3 in oxygen, incorporation of oxygen in the oxide takes place. The resulting film would be more "porous" in respect to Al_2O_3 deposited in nitrogen*".

Aboaf's technique was taken up again by Ito and Sakai [35], Tanaka et al. [33], Okamura and Kobayashi [31,40] and Kobayashi et al. [34], still for applications in microelectronics (gate insulator in GaAs-MIS and InP-MISFET transistors). They all used no additional oxygen sources in the gas phase. They deposited films in the temperature range 250-400°C, with occasional annealing at 550 or 700°C with or without the presence of H_2. These works are more a pragmatic approach to amelioration of films electrical behaviour than a systematic investigation of correlations between experimental parameters and films composition and properties. The authors had to face the thermal fragility of the substrates (GaAs, InP) on the one hand, and electrical instability or dispersion for films deposited at a too low temperature on the other hand. "*When the Al_2O_3 film was deposited below 300°C, frequency dispersion of the film capacitance was observed*" [33]. *"In conclusion, a suitable deposition temperature was found to be 340-350°C for Al_2O_3-InP MISFET. A device prepared at a lower temperature was accompanied by an increasing current drift, while the effective mobility of the MISFET decreased when the insulator was deposited at a higher temperature*" [34]. These and other remarks suggest that the film composition might well not be Al_2O_3 at low deposition temperature.

Fournier et al. [32], Morssinkhof et al. [41-45], and Yoshikawa et al. [46] reported more systematic studies of the MOCVD process of Al_2O_3 thin films from ATI in a hot-wall reactor. Fournier et al. were the first to operate at low pressure (95-100 Pa). The carrier gas was Ar, and the substrates were silicon. From infrared transmittance spectroscopy, films prepared at 350°C did contain hydroxylated species while they did not when prepared at 500°C. The authors observed: "*The samples prepared at 350°C and 400°C showed shrinkages of 6.5 % and 5.0 %, respectively, after annealing in flowing oxygen for 4 hours at 350°C. Those prepared at 450 and 500°C showed less than 1 % shrinkage. The large shrinkage obtained for the 350°C film is consistent with the observed presence of hydrated species indicated by the IR data*.[…] *It appears from these results that decomposition of aluminium tri-isopropoxide must be carried out above 400°C in order to obtain films which are thermally stable*". Film refractive index varied from 1.580 to 1.629 with deposition temperature going from 350°C to 500°C.

Thorough investigations have been carried out at the University of Twente, with Morssinkhof's PhD thesis and the related papers. Both atmospheric pressure (AP) MOCVD and low pressure (LP) MOCVD were used to prepare alumina from ATI in a hot-wall reactor. Substrates consisted of stainless-steel, with the purpose to study protection against corrosion. From a study of the decomposition of ATI in the presence of N_2 at low pressure (466 Pa) Morssinkhof et al. [43]

adopted the following sequence of mechanisms formerly proposed by Shulman et al. [47]: "T*he saturated gas stream is led into the furnace. At a temperature above 225°C ATI decomposes very fast in the gas phase to hydroxide. The reaction of hydroxide to alumina is slower, i.e. $Al(OH)_3$ can diffuse to the substrate surface. Once adsorbed on the surface (or on seeds in the gas phase) it decomposes to alumina and water. This water is then available for further hydrolysis of ATI*". From an Arrhenius plot built at 467 Pa, in the temperature range 250-450°C, they deduced: i) kinetics regime up to *ca.* 300-325°C, ii) activation energy of 30 to 40 kJ/mol, iii) predominance of gas phase reactions above 400°C. However, in his thesis Morssinkhof [41] proposed a different model for the decomposition mechanism of ATI which was later summarized as follows [44]: "*This model is based on a β-hydride elimination mechanism by the occurrence of a rather stable monomeric six-ring with intramolecular bond formation between Al-O group and the hydrogen attached to the β-carbon. During the first step propene and isopropanol will be formed, followed by a second step involving the elimination of the last propoxide group, resulting in a boehmite molecule and propene. In addition to these gas phase reactions,*[…] *the boehmite adsorbs on the substrate followed by a reaction forming alumina and water*". Kinetics study results were summarized as follows [45]: "*The deposition of Al_2O_3 is kinetically limited below 673 K* (400°C) *and is a first order reaction with an activation energy of 30 kJ.mole^{-1} at atmospheric pressure. The deposition of Al_2O_3 is kinetically limited below 623 K* (350°C) *and is a second order reaction at low pressure (3 torr)*(400 Pa) *with an activation energy of 30 kJ.mole^{-1}*". FTIR spectroscopy showed an OH band in the range 3000-3500 cm^{-1} for films deposited at 250 and 300°C; the films deposited in the range 300-500°C did not diffract X-rays; crystallization into γ-alumina was observed at 850°C [41,44]. In this set of extensive works, the postulated formation of $Al(OH)_3$ or AlOOH in the vapour phase is quite questionable since they are not molecular compounds. The words *boehmite molecule* and the formula O=Al-OH have no chemical signification. That Morssinkhoff retained this model is all the more surprising as his FTIR characterization of the gas phase during ATI thermal decomposition showed no formation of hydroxylated species (no band in the 3000-3600 cm^{-1} domain) [41].

Testing the CVD process to modify alumina composite membranes, Lee et al. [48] operated at low pressure (306 Pa) with N_2 as a carrier gas. From micro pore diffusion measurement and FTIR spectroscopy, they inferred that at 300°C $Al(OH)_X$ species deposited instead of Al_2O_3, and that the hydroxide species were removed by heating at 400°C.

Yoshikawa et al. [46] performed AP-MOCVD with He as a carrier and occasional addition of water in the gas phase. The Si substrate was placed in a temperature gradient. The deposits were found to be either $Al(OH)_3$ or Al_2O_3 depending on the deposition zone. The authors wrote: "*$Al(OH)_3$ molecules or clusters were generated in the gas phase either by pyrolytic reaction or hydrolytic reaction. They coagulated and formed particles which deposited on the substrate. The remaining molecular $Al(OH)_3$ migrated to the substrate by either convection or diffusion. Eventually, it reacted on the substrate surface and thin film was deposited via reaction $2Al(OH)_3 = Al_2O_3 + 3H_2O$*". Here again, 'molecular $Al(OH)_3$' is evoked.

Cold-Wall Reactor MOCVD. Most of the reported studies on alumina thin films prepared from ATI at low temperature in a MOCVD cold-wall reactor have concerned semiconductor device applications. In the early 1970's Duffy et al. [28-30] noticed that films deposited at 500°C contained excess oxygen with respect to formula Al_2O_3. This excess did not depend on whether O_2 was added or not to the carrier gas (He). Film refractive index varied from 1.60 to 1.68 with deposition temperature increasing from 425 to 500°C.

Cameron et al. [37] deposited alumina at 250-400°C, 100 Pa with N_2 as a carrier gas. From AES they observed very uniform film compositions. However they noticed the great sensitivity of growth rate and film quality to the state of the ATI source: "*the vapour pressure of aluminium isopropoxide is well known to be a complex function of its thermal history. The overall effect of these factors is a gradual decrease in average growth rate over a period of a few weeks. Over a*

period of a few hours, however, this system gives glassy films of reasonable quality with very reproducible growth rates". For a reactor geometry expected to produce a low surface concentration of reactants, the growth rate was diffusion-controlled between 250 and 400°C. For higher surface concentrations, the growth rate was kinetically controlled between 300 and 400°C.

Saraie et al. [36] using a vertical reactor with a horizontal substrate holder and N_2 as a carrier gas measured E_a = 76.1 kJ/mol from a kinetics regime Arrhenius plot between 265 and 330°C. Using the same reactor, Saraie et al. [49] studied the influence of a reactive atmosphere (N_2, $N_2 + H_2$, $N_2 + O_2$, $N_2 + H_2O$) on the deposition rate and electrical properties. A ($N_2 + H_2O$) mixture allowed to grow films at low temperature, but resulted in increasing apparent deposition rate when decreasing the temperature from 265 to 200°C. On heating above 500°C the thickness of the films shrinked down to that of films deposited at higher temperatures. IR absorption measurements at ~ 3450 cm^{-1} showed that these low-temperature deposited films were hydroxilated all the more as the substrate temperature was low. Hydroxo groups were eliminated above 500°C. The authors concluded that *"the addition of water vapour is effective in fixing Al_2O_3 into a solid film at low temperatures (even 170°C), but the resulting films are swelled by incorporated OH"*.

Go and Sugimoto [50] used a horizontal cold-wall reactor with a slightly inclined substrate holder to prepare highly corrosion resistant Al_2O_3 films by LP-MOCVD (800 Pa) in the presence of O_2. Deposition temperatures were in the range 200-500°C. An Arrhenius plot showed kinetically controlled growth rate between 200 and 300°C with E_a = 47.7 $kJ.mol^{-1}$. Refractive index and corrosion resistance increased with increasing deposition temperature. In a study by *in-situ* ellipsometry with a vertical reactor and a vertical substrate holder, Go et al. found an Arrhenius linear plot in the temperature range 262-361°C with apparent activation energies of 98.0 $kJ.mol^{-1}$ and 113.7 $kJ.mol^{-1}$ depending on whether O_2 was added or not in the gas phase.

Yom et al. [51] obtained well crystallized heteroepitaxial films of γ-Al_2O_3 on Si(100) substrates at 740°C and 3 torr (400 Pa), using a vertical reactor, Ar as a carrier gas and N_2O as a reactive gas. Dhanavantri and Karekar [38,39] have prepared graded-index thin films by varying the substrate temperature between 300°C (n = 1.562) and 450°C (n = 1.622).

Hara et al. [52] used LP-MOCVD in a cold-wall reactor to grow mixed (Al_2O_3, Ta_2O_5) thin films at 350°C, in the presence of O_2. From O_{1s} XPS spectrum they deduced the presence of OH groups in the films. The ratio of the peak area for OH^- to that of O^{2-} was maximum (S_{OH}/S_O = 0.76) for the films not containing tantalum oxide. By comparison with the results of Go and Sugimoto [50] they concluded that *"hydroxyl groups detected by XPS may arise from adsorbed OH^- species on the surface of the films, which is formed by water adsorption after the film formation"*.

From this Review it appears that ATI is a suitable precursor for the MOCVD of alumina films. Numerous investigations of this process have been performed for various applications. However several points remain to be made more precise or even to elucidate: i) the thermal degradation process of ATI in the MOCVD process and in the precursor delivery system is not precisely known; ii) studies concerned a temperature range of several hundred degrees, but kinetics investigations have given contrasted results; iii) the films prepared at low temperature do not diffract X-rays, hence are difficult to characterize; iv) the composition of films prepared at low temperature is not clear. For all these reasons, we have undertaken a systematic study of the MOCVD of alumina from ATI in the range 350-550°C to tentatively clear up some imprecise or even contradictory points in former studies. Results presented here concern the composition of the films mainly.

Experimental Part

MOCVD Experimental Parameters. A custom-made, horizontal hot-wall reactor [1] was used. The precursor was heated at 110°C. Because of structural evolution upon ageing, the precursor was renewed in the bubbler after nearly 12 hours of use. Depositions were carried out with N_2 both as a carrier gas and a dilution gas. A third line delivered water saturated N_2 occasionally. For deposition

with dry gas phase fixed experimental parameters were: total pressure = 667 Pa; N_2 flow rate over ATI = 53 sccm; dilution N_2 flow rate = 590 sccm. Variable parameters were: deposition temperature between 350 and 700°C; deposition time between 10 and 110 minutes. The films were grown on thermally grown SiO_2 on Si and on (100) Si wafers. For deposition with H_2O in the gas phase, the total pressure was 2 kPa. Humid N_2 was produced by bubbling N_2 (35 sccm) in water maintained at 1°C.

Films Characterization. Films composition and thickness were determined by scanning electron microscopy with a LEO 435 VP apparatus equipped with a PGT IMIX-PC EDS analyzer. EPMA, Elastic Recoil Detection Analysis (ERDA) and Rutherford back-scattering (RBS) were also used. EPMA was run at 10 and 15 kV with a CAMECA SX-50 apparatus. For ERDA and RBS analyses (Centre d'Etudes Nucléaires de Gradignan), the samples were irradiated with a 2.3 MeV He^+ beam under a tilt angle of 10°. A Si barrier detector counted the particles backscattered at 160° thus giving the Al/O ratio. A second one located at 20° forwards, masked by a 7 µm Al foil, detected the H atoms ejected from the sample, allowing their quantification up to 0.4µm deep in the sample. Grazing incidence X-ray diffraction (XRD) was run with a Seifert 3000 TT diffractometer. Transmittance IR spectroscopy was performed with a FTIR BIORAD QS 300 apparatus on 500 µm thick films deposited on 0.4 mm thick (100) Si wafers. TGA analyses were made with a SETARAM TAG 24s thermo balance (sensitivity = 0.5 µg). Samples were ~ 1.2 µm thick films on square SiO_2/Si platelets of ~ 1.5-2 cm^2. For each TGA analysis, two such platelets were heated under flowing dry air (0.2 l/min.). Heating rate was 5°C/min between RT and 150°C, and 2°C/min up to 600°C.

Results and discussion

XRD. Deposited films are transparent. Those prepared in the temperature range 350-550°C show no X-ray diffraction lines. Films grown at 700°C show wide diffraction peaks that fit with the pattern reported for γ-Al_2O_3 in the JCPDS file #10-425. Well-crystallized γ-Al_2O_3 formed upon annealing for 1 hour at 800°C under flowing nitrogen [1]. These results are in agreement with previously reported XRD studies in this system [2,28,41].

FTIR. The infrared spectroscopy study of the films revealed an amazing difference between coatings prepared in the absence and in the presence of water in the gas phase. Fig. 1 presents a part of the transmittance infrared spectra for films deposited on silicon at temperatures ranging from 350 to 480°C (623-753 K), without water in the gas phase. The spectra have been normalized so that the absorbance bands below 1000 cm^{-1} have nearly the same height.

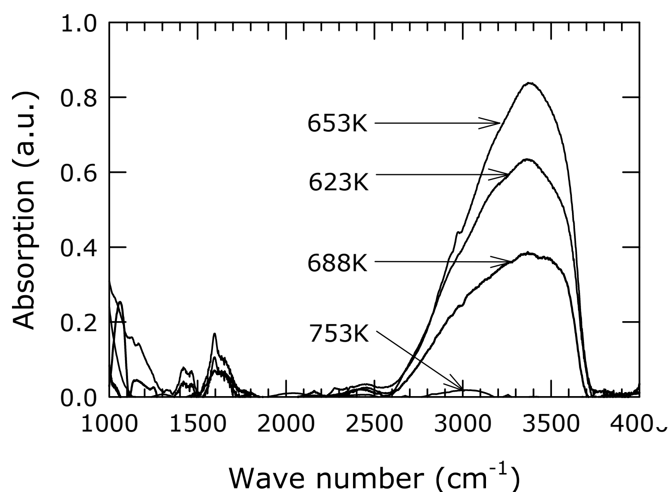


Figure 1. FTIR transmittance spectra for films prepared without water in the gas phase

Films prepared at 350, 380 and 415°C adsorb in the range 2600-3800 cm^{-1}. The strongest absorbance is observed for 380°C and the weakest one for 415°C. Thus the films are more or less hydroxylated. Water absorbance at about 1600 cm^{-1} is also observed with the same decreasing peak intensity. The films prepared at 480°C (and 550°C not shown) show no absorbance above 1000 cm^{-1}.

None of the films prepared with water in the gas phase showed OH absorption bands. To our knowledge, only Yoshikawa et al. have reported about the influence of water addition in the gas phase on films deposited from ATI by MOCVD in a hot-wall reactor [46]. These authors performed deposition in a gradient of temperature, the hottest zone being at 400°C. They observed no dramatic change in film composition on gas phase humidification. They suggested that water would help forming hydroxo-organic species $Al(OH)_n(OC_3H_7)_{3-n}$ in the low-temperature zone of the deposit, following Xu [53]. Saraie et al. [49] did also perform MOCVD with ATI and a humidified gas phase, but they used a cold-wall reactor. As mentioned in the above Review, they observed that the use of water facilitated deposition of oxohydroxo aluminium species at temperatures as low as 170°C and that the amount of hydroxo species increased with decreasing deposition temperature.

Chemical analyses. Fig. 2 and Fig. 3 show film composition determined by EDS, EPMA, RBS and ERDA techniques for different deposition temperatures. RBS analyses referred to both the bulk of the coating and its surface layer. Fig. 2 shows composition expressed as the normalized ratio R = $[O/Al]_{sample}/[O/Al]_{alumina}$, where the numerator and the denominator are the O/Al weight ratios for the sample and for a standard sample of α-Al_2O_3 analyzed in the same conditions respectively. The samples prepared at 350°C have R values corresponding to AlO(OH). At 415°C and above, average R values correspond to Al_2O_3. Transition from oxygen-rich to Al_2O_3-type films occurs between 380°C and 415°C. The mean H concentration (Fig. 3) is close to 30 at% for films deposited at 350°C. At 415°C and above, it is stabilized at less than 5 at%. EPMA revealed C traces for deposition at 350 and 380°C and no C within the detection limit of the technique above 415°C.

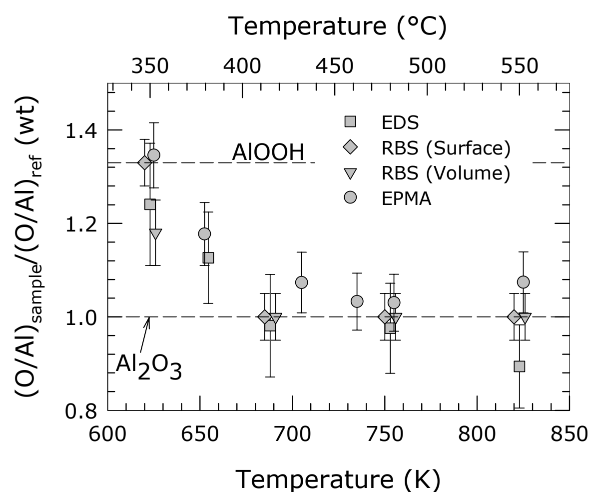


Figure 2. EDS, EPMA and RBS analyses of the O/Al ratio for films prepared without water added in the gas phase.

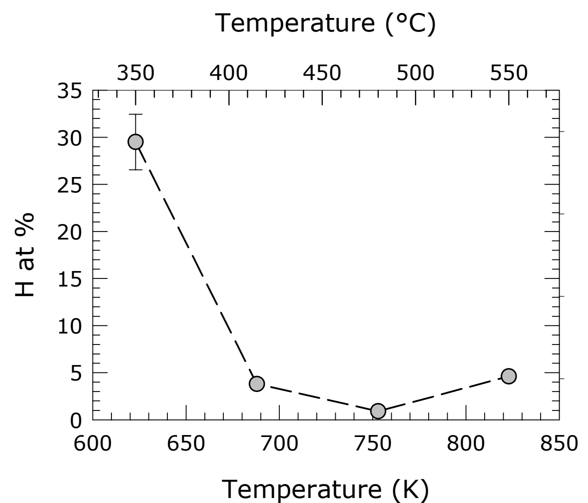


Figure 3. ERDA analyses of the H content for films prepared without water added in the gas phase.

The negligible C content rules out the possibility that the deposit consisted of partially hydrolyzed precursor $Al(OR)_{3-x}(OH)_x$, even at the lowest deposition temperatures investigated. From measured O/Al ratios and H concentrations, samples prepared at 350°C have a composition compatible with the formula AlO(OH). Above 415°C, the composition is that of alumina Al_2O_3.

From EDS and EPMA analyses, the films grown in the presence of water vapour have R ratios corresponding to Al_2O_3 within the experimental error range, whatever growth temperature in the range 350-550°C, in accordance with FTIR observations.

TGA. Fig. 4 presents the weight loss per volume unit as a function of temperature during TG analyses for films prepared without water in the gas phase. No such investigation has ever been reported for thin films of alumina. TGA curve profiles strongly depend upon deposition temperature. Samples processed at 480°C (and 550°C, not shown here) do not lose weight on heating, thus confirming the composition Al_2O_3. The films processed at 350°C and 380°C lose weight. Weight loss takes place far below the preparation temperature. The film prepared at 623 K (350°C) shows two weight losses: about 30 % of the total loss occurs between 343 K and 533 K, and the remaining 70 % above 533 K. Differential analysis (DTG) shows two broad peaks at 388 K and 555 K.

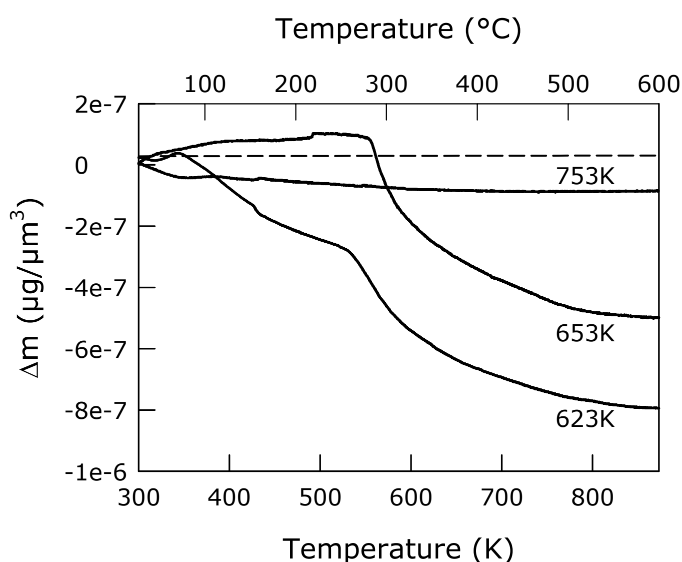
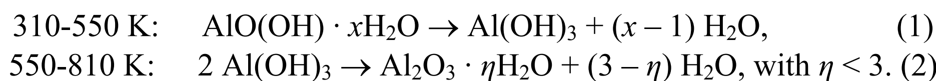


Figure 4. TG thermograms for films prepared without water added in the gas phase.

Since the film prepared at 350°C has a composition corresponding to AlO(OH), it is interesting to review thermal studies run on boehmite (γ-AlOOH), though they do not concern thin films but gel-derived solids [54-59], hydrothermally treated powders [60-64], synthetic products derived from $Al(OH)_3$ or natural compounds [60,63,65]. The literature survey shows contrasted results. For either gel derived or hydrothermally treated boehmite, most of the authors observed weight loss in two steps between 300 K and approximately 800 K: i) the first one starts short above 300 K and is completed at about 473 K; ii) the second one starts at about 473 K and is completed at 700-800 K [54,56-59,61,62]. Perić et al. [60] reported that boehmite prepared at 523 K from gibbsite $Al(OH)_3$ started to decompose between 639 K and 701 K depending on the heating rate in the range 5-40 K/minute. Decomposition occurs in two steps: the second weight loss starts about 90 degrees after the first one, when about 11% of the total weight is lost. Paglia et al. [64] observed a nearly similar process for boehmite prepared at the Alumina & Ceramics Laboratory. The decomposition scheme observed in the present study for the film grown at 350°C corresponds to none of these schemes.

From Ram et al. [55] the thermal behaviour of amorphous boehmite does depend upon the number of hydration water molecules in the formula $AlO(OH) \cdot xH_2O$. Particularly, for $x > 1$ TGA reveals two nearly equal weight losses (18 % each), the one between 310 K and 550 K, the other between 550 K and 810 K. Corresponding DTA endotherm maxima are at 388 K and 715 K. The authors have attributed the high temperature endotherm to dehydroxylation of $Al(OH)_3$, which would have formed during the low temperature weight loss. Corresponding reactions are:



310-550 K: $AlO(OH) \cdot xH_2O \rightarrow Al(OH)_3 + (x - 1)\, H_2O$, (1)

550-810 K: $2\, Al(OH)_3 \rightarrow Al_2O_3 \cdot \eta H_2O + (3 - \eta)\, H_2O$, with $\eta < 3$. (2)

This process might well correspond to what occurs in our film deposited at 623 K. The two weight losses start at temperatures (343 K and 533 K) rather close to those reported for starting reactions (1) (310 K) and (2) (550 K). The formation of $Al(OH)_3$ upon heating at low temperature and its subsequent decomposition at higher temperature may explain why the film decomposes below its temperature of formation. The x H_2O molecules necessary to the formation of $Al(OH)_3$ according to reaction (1) may come from the film being kept several weeks without preventing against the absorption of atmospheric water.

For the film prepared at 653 K the TGA curve shows one weight loss starting at 517 K, with a slope maximum at 562 K. This suggests the film was a mixture of Al_2O_3 and AlOOH (as the main component from the O/Al ratio shown in Fig. 3) with enough adsorbed water to allow the conversion of AlOOH into $Al(OH)_3$ during the low temperature heating period.

Yoshikawa et al. [46] have performed DTA analyses for thin films grown from ATI in a cold-wall reactor. One of the DTA profiles shows maxima at 380 K, 550 K and ~ 580 K (shoulder). These values of 380 K and 550-580 K compare quite well the DTG maxima observed in the present study for the films prepared at 623 K (388 K & 555 K) and at 653 K (562 K). By comparison with the DTA profile for anhydrous $Al(OH)_3$ powder that they showed to present a strong endotherm at 550 K, the authors concluded that their film consisted of hydrated $Al(OH)_3$. On heating, the film first dehydrates (endotherm at 380 K) and then decomposes into Al_2O_3 (endotherms at 550-580 K). As another literature data about $Al(OH)_3$, Perić et al. [60] observed that gibbsite from Bayer started to decompose between 457 K and 503 K depending on the heating rate.

Conclusions

A Review about MOCVD preparation of alumina thin films from ATI, the most popular precursor of alumina, has evidenced that most studies have privileged urgent application rather than fine investigation and optimization of the process. Therefore several points remain to elucidate. In the present study, films were prepared in a hot-wall reactor, in the temperature range 350-550°C, and characterized by combining elemental analyses (EMPA, EDS, ERDA, RBS), FTIR and TGA. They do not diffract X-rays. When N_2 alone is used as a carrier gas, the films prepared at about 415°C and above have the formula Al_2O_3. Prepared below 415°C, they contain hydroxo species. At 350°C, the composition is close to AlOOH. When water is added in the gas phase, the films have the composition Al_2O_3 over the entire temperature range 350-550°C. The TGA of a film of AlOOH suggests that the film transforms into $Al(OH)_3$ on being heated due to reaction with water adsorbed from the atmosphere. Results about growth kinetics and film morphology are going to be published.

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