

# Metallo-organic low-pressure chemical vapor deposition of Ta<sub>2</sub>O<sub>5</sub> using TaC<sub>12</sub>H<sub>30</sub>O<sub>5</sub>N as precursor for batch fabrication of microsystems

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## Abstract

Ta<sub>2</sub>O<sub>5</sub> thin films were produced by metallo-organic low pressure chemical vapor deposition using Tantalum(V) Tetraethoxydimethylaminoethoxide (TaC<sub>12</sub>H<sub>30</sub>O<sub>5</sub>N) as precursor. This liquid precursor at room temperature makes it possible to deposit thin films of Ta<sub>2</sub>O<sub>5</sub> on wafer batches of up to 35 wafers. In this communication, we report on the processing and equipment development to achieve batch fabrication, and on the optimization of the deposited thin films properties for their application in microsystems. An evaporator was linked to a horizontal hot wall furnace. The deposition of Ta<sub>2</sub>O<sub>5</sub> was performed at 425 °C and the influence of a post-annealing at higher temperatures on the chemical, electrical and optical properties of the films was evaluated. Annealing treatments in oxygen were found to reduce the amount of residual carbon and hydrogen in the films. An annealing in oxygen followed by an annealing in forming gas was used to improve the charge levels and hysteresis. The optical properties of the amorphous Ta<sub>2</sub>O<sub>5</sub> films varied slightly with the annealing treatment. Annealing the films at a temperature of 700 °C and higher caused their crystallization, leading to a decrease of their optical bandgap. The processed films have found applications in microsystems as chemical resistant coatings, optical coatings for wave guides, and chemical sensitive layers for Ion-Sensitive Field-Effect Transistors.

*Keywords:* 91 Deposition process; 351 Organometallic vapor deposition; 431 Sensors; Extra suggestion: Tantalum oxide or tantalum pentoxide

## 1. Introduction

Over the years, many techniques have been proposed to prepare tantalum pentoxide thin films [1]. These techniques can be grouped in three main categories: oxidation techniques, chemical vapor deposition (CVD), and physical vapor deposition. In microelectronics, CVD is generally preferred for the deposition of insulating films. The main advantages of this technique is that it can be performed over a wide pressure range, gives uniform, conformal and adherent layers, and provides fast deposition rates [1]. Disadvantages can be the use of dangerous gases and high

temperatures. During the last years, efforts were focused on this technique to process functional Ta<sub>2</sub>O<sub>5</sub> films. Different CVD processes have been proposed to deposit Ta<sub>2</sub>O<sub>5</sub>: Atmospheric Pressure CVD, LPCVD (Low-pressure CVD), PECVD (Plasma Enhanced CVD), photo- or laser-assisted CVD among others [1–4].

Several gaseous reactants, made of vapors obtained from liquid or solid reagents, have been investigated for the deposition of Ta<sub>2</sub>O<sub>5</sub> by CVD. Two main approaches have been reported so far, the use of organometallic or of carbon and hydrogen free precursors, with tantalum pentaethoxide (Ta(OC<sub>2</sub>H<sub>5</sub>)<sub>5</sub>/TAETO), being the most commonly used [1,2]. The precursors used led to processes where the deposition of tantalum pentoxide is performed on a limited number of wafers at the same time [1]. In this paper, we report on the use of a precursor, namely Tantalum(V)

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Tetraethoxydimethylaminoethoxide ( $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$ /TAT-DMAE), and a CVD reactor design that allow depositing  $\text{Ta}_2\text{O}_5$  thin films on several wafers at the same time. Compared to  $\text{Ta}(\text{OC}_2\text{H}_5)_5$ , this precursor was especially developed to be used in a bubbling configuration due to its higher volatility and lower melting point. In our case, the lower melting point of  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$  made it easier to design a machine for its evaporation. The choice of  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$  vs.  $\text{Ta}(\text{OC}_2\text{H}_5)_5$  has therefore been primarily driven by its easier properties and the cheaper hardware that follows.

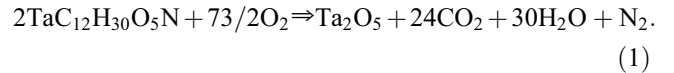
So far and to the best of our knowledge, the use of  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$  to deposit  $\text{Ta}_2\text{O}_5$  films was limited to PECVD and Ultraviolet Assisted Injection Liquid Source–CVD techniques, with the deposition made on a restricted number of wafers due to the vertical configuration of the CVD reactors and to the thermal budget limitation related to the application in microelectronics [3,4]. In our case, the process development was made on a horizontal LPCVD reactor linked to a system for the evaporation of the precursor,  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$ , to achieve wafer batch fabrication. This communication describes the development of the LPCVD process using  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$  as precursor to achieve batch fabrication and the optimization of the chemical, electrical, and optical properties of the deposited  $\text{Ta}_2\text{O}_5$  films for their application in microsystems.

## 2. Experimental details

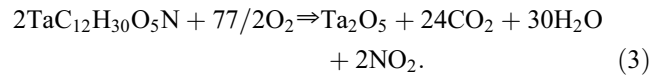
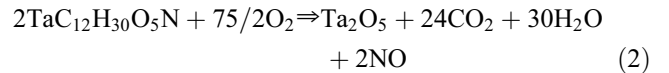
### 2.1. Deposition process

A horizontal LPCVD hot wall furnace was developed by Tempress Systems Inc. (Heerde, The Netherlands), for the deposition of  $\text{Ta}_2\text{O}_5$  by MOLPCVD using  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$  (TAT-DMAE, from Schumacher, Carlsbad, CA, USA) as a precursor. The system allows processing batches of up to 35 wafers. The complete deposition system consists of a precursor evaporation chamber, a 3-zone heated deposition

chamber, of three containers:  $\text{TaC}_{12}\text{H}_{30}\text{O}_5\text{N}$ , ethanol and waste, and of few gas lines: He,  $\text{O}_2$  and  $\text{N}_2$ . Fig. 1 shows a schematic drawing of the system. The liquid is pressurized using Helium (Fig. 1, V6). Then, it is carried out through a liquid mass flow meter to the evaporation chamber (Fig. 1, V5+evap), where it is evaporated at a temperature of 110 °C. From that point, the gaseous precursor is brought to the deposition chamber through a heated gas line (130 °C) using Helium (Fig. 1, V4) as the carrier gas. In the deposition chamber, the precursor reacts with oxygen at about 425 °C (Fig. 1, V2) to obtain a deposition of  $\text{Ta}_2\text{O}_5$  thin films on the silicon wafers. The following chemical reaction is proposed if  $\text{N}_2$  does not get oxidized:



Another assumption could be that  $\text{N}_2$  gets oxidized leading to different reactions with the formation of NO and/or  $\text{NO}_2$ :



Nitrogen is used both to purge the system (V1+V9) and to regulate the pressure in the deposition chamber by saturating the pumping system (Fig. 1, V15+V16). The ethanol (Fig. 1, V8) is necessary to clean the tubing carrying the precursor when the container has to be filled up.

### 2.2. Film preparation

The deposition was performed at a pressure of 26.66 Pa with a ratio of  $\text{O}_2/\text{He}$  of 1.75 (350 sccm  $\text{O}_2$ ). The flow rate of oxygen was adjusted to prevent the accumulation of carbon in the films (cf. 3.1). The temperature in the three heated zones varied from 415 to 435 °C. The temperature

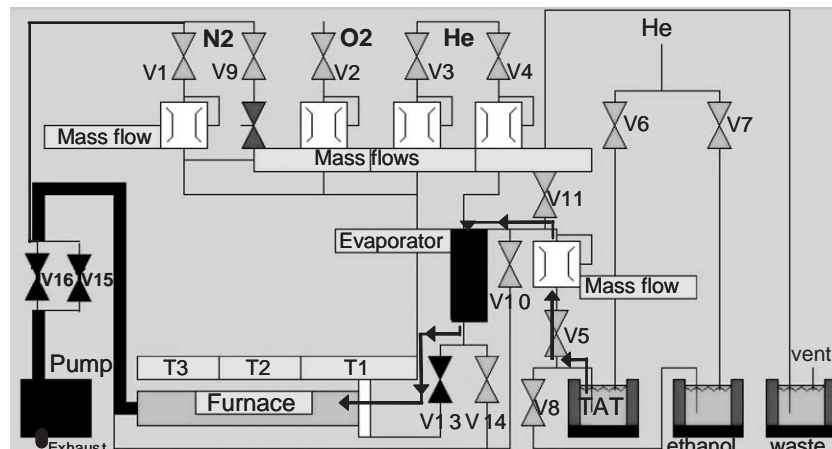


Fig. 1. Schematic representation of the CVD reactor developed by Tempress Systems Inc. to deposit thin films of  $\text{Ta}_2\text{O}_5$  using as precursor.

was set at an average of 425 °C following the recommendation of the precursor supplier. The deposition rate was of 2.5 nm/min. The uniformity of the film thickness was in between 5% and 8% on a single wafer and of 5% from wafer to wafer, over a complete batch of wafers. The film uniformity meets the requirements for different applications in the field of microsystems. A reduction of the pressure in the deposition chamber (13.33 Pa) improved the uniformity but decreased significantly the deposition rate. In this work, for practical reasons related to production, the optimization of the film properties was performed on films deposited at a pressure of 26.66 Pa.

Ta<sub>2</sub>O<sub>5</sub> thin films with a thickness of 150 nm were deposited on pre-cleaned p-type silicon wafers (100 mm wide, 525 μm thick, 5–25 Ω cm) covered with only a native oxide (~2.5 nm) or with a 80 nm thick dry thermal oxide. After deposition, different heat treatments were performed on the films. They were annealed during 30 min in oxygen at atmospheric pressure between 425 °C and 800 °C (425, 525, 625, 700 or 800 °C) or in 10% H<sub>2</sub>/N<sub>2</sub> at 450 °C, or successively in O<sub>2</sub> and H<sub>2</sub>/N<sub>2</sub>. Amorphous and polycrystalline Ta<sub>2</sub>O<sub>5</sub> films with different electrical and chemical characteristics were obtained.

### 2.3. Characterization

The composition, microstructure, and the chemical and electrical properties of the Ta<sub>2</sub>O<sub>5</sub> films were characterized as a function of the annealing treatments. Rutherford Back-Scattering (RBS: 1.7 MeV He<sup>+</sup>, detector at 170°, ±4% precision on the O/Ta ratio and ±1% on the carbon content), X-ray Diffraction (XRD: Philips MPD 1880 diffractometer, λCuK<sub>α</sub>=0.15406 nm), Capacitance–Voltage (*C–V*: MDC CSM/Win system, frequency of 100 MHz), and ellipsometry (UVISEL TM 460, Jobin-Yvon-HORIBA, incidence angle of 70.9°, energy range of 0.75–4.5 eV) measurements were performed.

The electrical properties of the Ta<sub>2</sub>O<sub>5</sub> films and Ta<sub>2</sub>O<sub>5</sub>/SiO<sub>2</sub> were studied using metal-oxide-semiconductor capacitors structures made of the dielectric layer covered with an aluminum gate (circular with a diameter of 1 mm, 300 nm thick). The Nss calculation compared the measured flatband voltage, *V*<sub>fbm</sub>, with the expected flatband voltage, *V*<sub>fbe</sub>. The measured flatband voltage was found from the flatband capacitance value (*C*<sub>fb</sub>) on the *C–V* curve. The expected flatband voltage was the metal-semiconductor work function difference (*φ*<sub>ms</sub>). The difference between these flatband voltage values was attributed to the non-ideal charges (*Q*): fixed oxide charge, mobile oxide charge, oxide trapped charge, and interface trapped charge. Nss, the effective number of charges per unit area (number/cm<sup>2</sup>), is equal to  $|Q/q|$ , where *Q* is the net effective charge per unit area (C/cm<sup>2</sup>) and *q* the elementary charge.

The chemical resistance of the Ta<sub>2</sub>O<sub>5</sub> films to KOH (40%, 60 °C) and HF (50%), chemicals typically used in bulk and surface silicon micromachining processes, was also eval-

uated. The results obtained were used to optimize the quality of the films for the different applications mentioned before.

## 3. Results and discussion

The characteristics of the Ta<sub>2</sub>O<sub>5</sub> films depended on the nature of the annealing treatment as previously mentioned by other research groups in the literature [1,2,4]. Stoichiometry, permittivity, fixed charges, *C–V* hysteresis, crystallinity were evaluated for the different thermal annealing performed.

### 3.1. Chemical composition and microstructure

RBS measurements were performed on amorphous Ta<sub>2</sub>O<sub>5</sub> films since these are the films we would like to take advantage of their electrical and optical properties. A O/Ta ratio of 2.4±0.096 was found by RBS for the as-deposited Ta<sub>2</sub>O<sub>5</sub> films. An annealing at 625 °C during 30 min in oxygen had the effect to increase the oxygen content in the films, with a O/Ta ratio of 2.46±0.098. Carbon and hydrogen, in concentrations of about 3%±1, were also present in the as-deposited films. However, based on the results presented in the literature [1,4], a post-anneal in oxygen (625 °C, 30 min) was performed to reduce the amount of carbon to a value less than 2%±1. Increasing the flow rate of oxygen (from 200 to 350 sccm) in the deposition chamber helped also to decrease the quantity of carbon in the films. This post-anneal also decreased the amount of hydrogen to a value of about 1%. Despite the relatively low accuracy on the measured O/Ta ratios, the RBS measurements demonstrated that a thermal treatment in oxygen (in the range of temperature studied) improves the quality of the films by eliminating impurities (C, H) and increasing the oxygen contents. Some investigations still need to be performed to determine the concentration of the He atoms that might have been trapped in the films during the deposition process.

Finally, the as-deposited films were amorphous and their crystallization started to occur when the films were annealed at temperatures *T* ≥ 700 °C (Fig. 2). The diffraction peaks

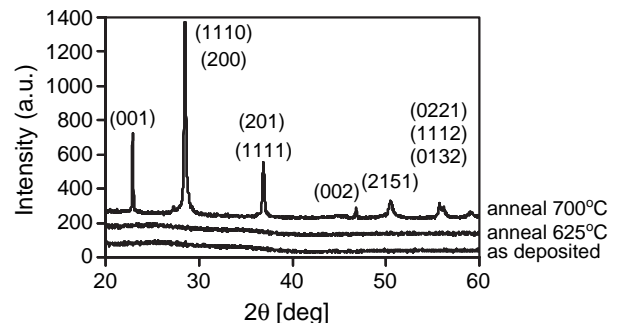


Fig. 2. XRD spectra of Ta<sub>2</sub>O<sub>5</sub> films as deposited, and annealed at 625 °C and 700 °C in oxygen.

corresponded to  $\beta$ - $\text{Ta}_2\text{O}_5$  with an orthorhombic structure as observed in Ref. [5].

### 3.2. Electrical properties

The permittivity of the amorphous films was  $24 \pm 2$  and is in the range (22 to 28) of the results previously reported [1]. After being annealed at 700 and at 800 °C, the dielectric constant increased to reach a value of  $44 \pm 2$ . The growing permittivity corresponds to the transition from the amorphous to the polycrystalline phase [1] (see Fig. 2).

The effect of the annealing in oxygen and in forming gas on the capacitance–voltage characteristic of the tantalum oxide film deposited directly on silicon or on a silicon dioxide film was also investigated. Working in the field of microsystems and since, as described below, the electrical properties of the tantalum oxide films deposited on silicon were far from being optimized compared to films deposited on silicon dioxide, efforts were dedicated to optimize the electrical properties of tantalum oxide films deposited on silicon dioxide for applications in Ion-Sensitive Field-Effect Transistors (ISFETs). The use of  $\text{Ta}_2\text{O}_5$  as ion-sensitive material instead of other gate dielectrics, such as  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$ ,  $\text{Al}_2\text{O}_3$ , was motivated by the advantages of an improved pH response and a better chemical resistance [6].

The as-deposited amorphous tantalum oxide film on silicon had a charge level,  $N_{\text{ss}}$ , of  $5 \times 10^{11}/\text{cm}^2$ , which increased to a value in the order of 1 to  $4 \times 10^{12}/\text{cm}^2$  when annealed in oxygen. Moreover, as-deposited and annealed  $\text{Ta}_2\text{O}_5$  films on silicon exhibited a large hysteresis varying from 0.5 to 2.0 V depending on the annealing treatment.

Lower charge levels and hysteresis were obtained for gate dielectric stacks made of amorphous  $\text{Ta}_2\text{O}_5$  and  $\text{SiO}_2$  films. Non annealed  $\text{Ta}_2\text{O}_5/\text{SiO}_2$  stack had a  $N_{\text{ss}}$  of  $2 \times 10^{11}/\text{cm}^2$  and exhibited a hysteresis of 0.3 V. When annealed in oxygen at 625 °C, the charge level of the  $\text{Ta}_2\text{O}_5/\text{SiO}_2$  stack remained approximately the same but was however slightly reduced for annealing performed at 425 ( $7 \times 10^{10}/\text{cm}^2$ ) or 525 °C ( $9 \times 10^{10}$ ). The hysteresis of  $\text{Ta}_2\text{O}_5/\text{SiO}_2$  stack annealed in oxygen decreased to reach a value of 45 mV when annealed at 625 °C, of 20 mV when annealed at 525 °C and of 50 mV when annealed at 425 °C. Fixed charges and the hysteresis also decreased when as-deposited  $\text{Ta}_2\text{O}_5/\text{SiO}_2$  films or  $\text{Ta}_2\text{O}_5/\text{SiO}_2$  films which had a thermal treatment in oxygen at 425 or 525 °C were further annealed in  $\text{H}_2/\text{N}_2$ . This was for instance not observed when an annealed in forming gas was performed on films previously annealed in oxygen at 625 °C for which the fixed charges remained unchanged and the hysteresis increased to reach a value of 200 mV. Fig. 3 shows typical  $C-V$  measurement curves obtained for  $\text{Ta}_2\text{O}_5$  films deposited on  $\text{SiO}_2$  which were successively annealed in  $\text{O}_2$  and in  $\text{H}_2/\text{N}_2$ . In the fabrication of ISFETs, the annealing in  $\text{H}_2/\text{N}_2$  is the last step used to establish activation of the ohmic contacts between aluminum and silicon and in the same time it improves the electrical properties of the gate dielectric stack.

Crystallised (annealed at 700 or 800 °C)  $\text{Ta}_2\text{O}_5$  films deposited directly on silicon had a charge level,  $N_{\text{ss}}$ , slightly higher than  $10^{12}/\text{cm}^2$ . A large hysteresis of  $\sim 1$  V was also observed in the  $C-V$  measurement curves. As for the amorphous films, the electrical properties of the crystallized tantalum oxide films that were deposited on silicon oxide

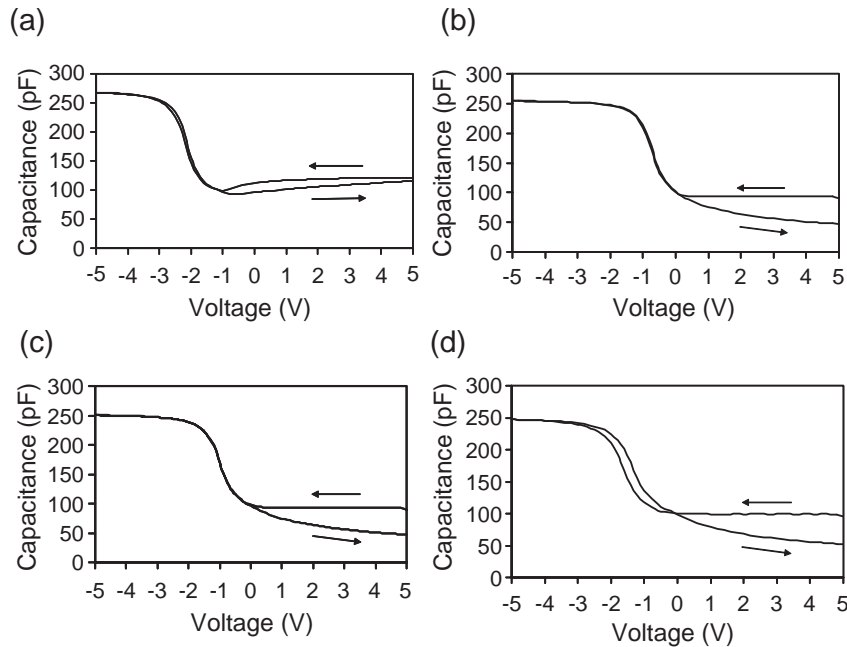


Fig. 3. Capacitance–voltage curves (retrace mode) obtained for a  $\text{Ta}_2\text{O}_5$  film deposited on  $\text{SiO}_2$  (a) as-deposited and an annealing in  $\text{H}_2/\text{N}_2$  at 450 °C and (b) annealed successively in  $\text{O}_2$  at 425 °C and in  $\text{H}_2/\text{N}_2$  at 450 °C and (c) annealed successively in  $\text{O}_2$  at 525 °C and in  $\text{H}_2/\text{N}_2$  at 450 °C and (d) annealed successively in  $\text{O}_2$  at 625 °C and in  $\text{H}_2/\text{N}_2$  at 450 °C.

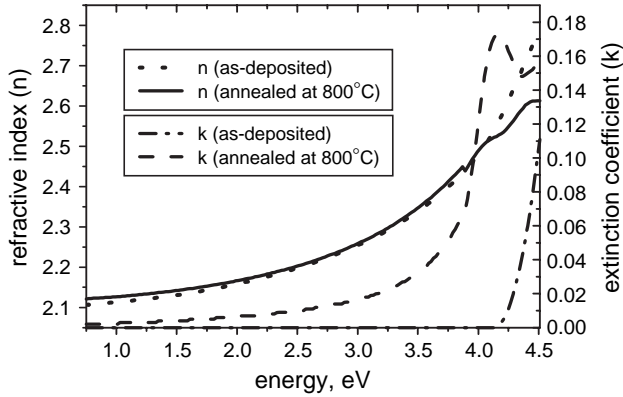


Fig. 4. Dispersion of optical parameters ( $n$ ,  $k$ ) for a  $\text{Ta}_2\text{O}_5$  film: as-deposited (red curves) and annealed at  $800\text{ }^\circ\text{C}$  (black curves). A distinct shift in the band gap to the lower energy as well as an absorption tail below energy gap is observed for the annealed (polycrystalline) film.

were improved. After an annealing in oxygen at  $700\text{ }^\circ\text{C}$  of  $\text{Ta}_2\text{O}_5$  films deposited directly on silicon dioxide, the charge level,  $N_{\text{ss}}$ , decreased to a value of  $5 \times 10^{11}/\text{cm}^2$  and the hysteresis was reduced to a value of  $200\text{ mV}$ . The crystallization at  $800\text{ }^\circ\text{C}$  of  $\text{Ta}_2\text{O}_5$  films on  $\text{SiO}_2$  did not change significantly the level of charges but had a similar effect on the hysteresis value, which decreased to  $100\text{ mV}$ . The best electrical properties were obtained when  $\text{Ta}_2\text{O}_5$  films on  $\text{SiO}_2$  crystallized at  $700\text{ }^\circ\text{C}$  were annealed in forming gas. The charge level remained the same at  $5 \times 10^{11}/\text{cm}^2$  but a hysteresis of less  $10\text{ mV}$  was obtained after being annealed in forming gas.

### 3.3. Optical properties

The optical functions of as-deposited  $\text{Ta}_2\text{O}_5$  films were modelled using the Tauc–Lorentz dispersion relation developed by Jellison and Modine for amorphous semiconductors [7]. This dispersion relation was also used for  $\text{Ta}_2\text{O}_5$  films annealed at  $T \leq 625\text{ }^\circ\text{C}$  since those stay amorphous. Values of the refractive index ( $n$ ) between 2.15 and 2.2 (at  $633\text{ nm}$ ) were measured. The optical band gap ( $E_g$ ) varied from 3.8 to 4.2 eV. Similar values are reported in the literature for films deposited by CVD and pulsed laser techniques [8,9]. Annealing temperatures  $700\text{ }^\circ\text{C}$  and above caused crystallization. In the case of polycrystalline films  $\text{Ta}_2\text{O}_5$ , the Tauc–Lorentz dispersion model is no longer valid and a classical Lorentz model was applied. Fig. 4 shows the optical parameters ( $n$  and  $k$ ) as a function of energy of two  $\text{Ta}_2\text{O}_5$  films: amorphous (as-deposited) and polycrystalline (annealed at  $800\text{ }^\circ\text{C}$ ). Two Lorentz oscillators were necessary to fit the ellipsometric data of the polycrystalline  $\text{Ta}_2\text{O}_5$  film. They give rise to two characteristic features in the extinction coefficient dispersion curve. These features, absent in amorphous material, arise from interband electronic transitions. The first absorption peak is situated at about  $4.2\text{ eV}$  and the second one lies outside the measured energy range (at about  $4.6\text{ eV}$ ).

Nguyen et al. [8] also observed these two features in the dielectric function spectra of films annealed at high temperatures ( $T > 700\text{ }^\circ\text{C}$ ), but with positions situated at higher energy:  $4.7$  and  $5.3\text{ eV}$ .

The extinction coefficient of the pc- $\text{Ta}_2\text{O}_5$  film shows a monotonous increase already well below the band gap. In addition, a shift of the optical band gap from  $4.04\text{ eV}$  for as-deposited film to  $3.56\text{ eV}$  for annealed at  $800\text{ }^\circ\text{C}$  was observed. A strong increase of the absorption in the pc- $\text{Ta}_2\text{O}_5$  is already evidenced in the raw data by a strong attenuation of the ellipsometric angle  $\Psi$  above  $\sim 3.5\text{ eV}$ . Such an absorption tail was also observed in pc- $\text{Ta}_2\text{O}_5$  films obtained by atomic layer deposition at high temperatures ( $400\text{--}500\text{ }^\circ\text{C}$ ) [10]. This tail was explained by the presence of agglomerations due to crystallization.

Fig. 5 presents the evolution of the optical band gap with annealing temperature for the  $\text{Ta}_2\text{O}_5$  films deposited on silicon. The values of the optical band gap were derived from the Tauc plot  $(\alpha h\nu)^{1/2}$  vs.  $h\nu$ , assuming an indirect transition [11]. The  $E_g$  is about  $4\text{ eV}$  for the as-deposited films. It then increases with annealing temperature up to  $525\text{ }^\circ\text{C}$ . This increase can be related to the decrease of the oxygen vacancies in  $\text{Ta}_2\text{O}_5$  during annealing in the  $\text{O}_2$  atmosphere. At higher annealing temperatures ( $625\text{--}800\text{ }^\circ\text{C}$ )  $E_g$  decreases to attain  $\sim 3.6\text{ eV}$  at  $800\text{ }^\circ\text{C}$ . A similar decrease of the band gap upon crystallization was reported previously [5,8,12]. Burte and Rausch [12] measured the values of  $E_g$  of  $4.4\text{ eV}$  for as-deposited a- $\text{Ta}_2\text{O}_5$  films, and  $E_g$  of  $4.2\text{ eV}$  for pc- $\text{Ta}_2\text{O}_5$  films annealed at  $900\text{ }^\circ\text{C}$ . Franke et al. [5] observed a sharp onset of the interband transition at about  $4\text{ eV}$  in a- $\text{Ta}_2\text{O}_5$ , but a smooth absorption onset starting at lower energies for pc- $\text{Ta}_2\text{O}_5$  films. The origin of the lowering of  $E_g$  upon annealing was not evidenced in [5,8,11,12]. For our films, there is no clear explanation either on the decrease of  $E_g$  due to a lack of the chemical composition and structure of the annealed films. However, one may speculate about chemical and structural factors affecting  $E_g$ .

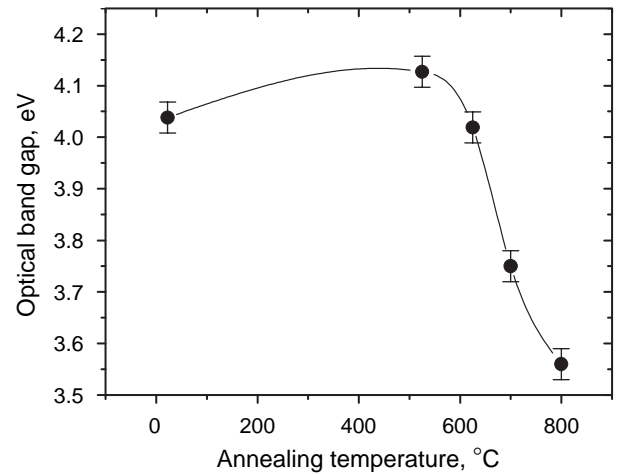


Fig. 5. Values of the optical band gap ( $E_g$ ) of  $\text{Ta}_2\text{O}_5$  films on silicon annealed at different temperatures in oxygen.

### 3.4. Chemical resistance

The chemical resistance to HF or KOH of Ta<sub>2</sub>O<sub>5</sub> amorphous films deposited on silicon improved slightly with the annealing temperature increasing to 625 °C. The etch rate for the as-deposited film was of about 37 nm/min in HF and 36 nm/h in the KOH solution (40%, 60 °C). After being annealed at 625 °C, the etch rate in HF (50%) stayed almost the same, about 35 nm/min, and decreased to 23 nm/h in KOH. In the case of polycrystalline films, the etch rate in HF and KOH was much slower than for amorphous films. The Ta<sub>2</sub>O<sub>5</sub> film thickness remained the same after some minutes of exposition to HF or some hours to KOH. However, the crystallization of the films deposited directly on silicon induced defects, such as pinholes and cracks, which limit the chemical resistance of films, especially in KOH. Investigations are under progress to find a buffer layer to minimize the stress in the tantalum oxide film during its crystallization with the aim of avoiding micro-cracks formation.

A summary of the results obtained is presented in Table 1.

### 4. Applications

Due to their chemical, optical and electrical properties, the Ta<sub>2</sub>O<sub>5</sub> films could be applied in the field of microsystems as protective coatings, waveguides and sensitive layers in chemical sensors [13–17]. Due to their good chemical resistance, the films were used as a protective coating for glass and silicon etching in HF and KOH, respectively. They could be used to completely protect one face of the wafer or could be patterned by reactive ion etching, using a mixture of CF<sub>4</sub> and O<sub>2</sub>, to define the areas to be etched [18]. The refractive index of the amorphous Ta<sub>2</sub>O<sub>5</sub> films is of interest for the integration of optical waveguides in microsystems. However, the precise control of the refractive index is essential for their proper operation. After a further optimization of the deposition process to obtain films with improved optical properties, the amorphous films could find applications in the field of Micro-Optical–Electrical–Mechanical Systems. Finally, the electrical and chemical properties of the Ta<sub>2</sub>O<sub>5</sub> films are of importance in microsystems mainly for applications in ISFET devices. Fixed charges and hysteresis were found

to be influenced by the annealing treatment and the presence of SiO<sub>2</sub> in between the silicon and Ta<sub>2</sub>O<sub>5</sub>. The best electrical characteristics were obtained for a gate dielectric made of a Ta<sub>2</sub>O<sub>5</sub> film on top of a dry SiO<sub>2</sub>, which were successively annealed at 525°C in oxygen and 450 °C in forming gas. N-type channel ISFETs made using this optimized gate insulator showed a good pH sensitivity of 59 mV/pH [19].

### 5. Conclusion

The combination of a hot wall horizontal LPCVD reactor with TaC<sub>12</sub>H<sub>30</sub>O<sub>5</sub>N used as precursor allows the deposition of Ta<sub>2</sub>O<sub>5</sub> thin films using a batch fabrication process. The influence of the deposition parameters and annealing treatments on the Ta<sub>2</sub>O<sub>5</sub> films' chemical, electrical and optical characteristics was investigated. Annealing treatments in oxygen reduced the amount of residual carbon and hydrogen in the films. An annealing in oxygen followed by an annealing in forming gas was used to improve the charge levels and hysteresis. Annealing at a temperature of 700 °C and higher caused the films to crystallize. The optical properties of the amorphous Ta<sub>2</sub>O<sub>5</sub> films varied slightly with the annealing treatment. The optical bandgap values measured were smaller for the polycrystalline films. Finally, the Ta<sub>2</sub>O<sub>5</sub> films were found to have different possible applications in the field of microsystems: such as chemical resistant coatings, optical wave guides and ion-sensitive films.

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Table 1  
Etch in HF and in KOH of Ta<sub>2</sub>O<sub>5</sub> thin films after different annealing treatments

	HF (50%) nm/min	KOH (40%, 60 °C) nm/h
As deposited	37	36
Annealed 625 °C O <sub>2</sub> 30 min	35	23

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