# MOLPCVD of $Ta_2O_5$ using $TaC_{12}H_{30}O_5N$ as precursor for batch fabrication

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#### INTRODUCTION

 $Ta_2O_5$  thin films for Microsystems (MEMS) applications were produced by MOLPCVD using  $TaC_{12}H_{30}O_5N$  (TAT\_DMAE) as precursor. This precursor allowed to produce thin films of  $Ta_2O_5$  on wafer batches of up to 25 wafers. These films found applications in Microsystems as chemical resistant coatings, optical layers for wave guides, and chemical sensitive layer for Ion-Sensitive FETs (ISFETs). In this communication, we will report on the processing and equipment development, and on the chemical, electrical, and optical characterization of the films processed.

### FILM PREPARATION

A horizontal LPCVD reactor was developed by Tempress Systems Inc, The Netherlands, for the deposition of  $Ta_2O_5$  by MOCVD using  $TaC_{12}H_{30}O_5N$ (TAT\_DMAE, from Schumacher, U.S.A.) as precursor. The system allows processing wafers in batches of up to 25 wafers. The reactor is made of a precursor evaporation chamber, a 3-zone heated deposition chamber, of three containers: TAT-DMAE, ethanol and waste, and of three gas lines: He,  $O_2$  and  $N_2$ .

The liquid TAT\_DMAE is pressurized using Helium and carried to the evaporation chamber where it is evaporated at a temperature of  $110^{\circ}$ C. From that point, the gaseous precursor is brought to the deposition chamber through a heated gas line ( $130^{\circ}$ C) using Helium as the carrier gas. In the deposition chamber, the precursor reacts with oxygen at about 425°C to obtain a deposition of Ta<sub>2</sub>O<sub>5</sub> thin films on the silicon wafers. Nitrogen is used to purge the system and to regulate the pressure in the deposition chamber. The ethanol serves when the TAT\_DMAE container has to be filled up.

The Ta<sub>2</sub>O<sub>5</sub> thin films were deposited on silicon or on silicon wafers with a dry thermal oxide. After their deposition, different heat treatments were performed on the films. They could be annealed under oxygen from 425°C to 800°C or in H<sub>2</sub>/N<sub>2</sub> at 450°C or successively in oxygen and in H<sub>2</sub>/N<sub>2</sub>. Amorphous or polycrystalline Ta<sub>2</sub>O<sub>5</sub> films, with different electrical and chemical characteristics, were obtained.

# CHARACTERISATION

The composition, the microstructure, and the chemical and electrical properties of the  $Ta_2O_5$  films were characterized. WDS, RBS, XRD, C-V, and ellipsometric measurements were performed with the aim of optimizing the quality of the films for the different applications mentioned above. The chemical resistance of the  $Ta_2O_5$  films to KOH (40%, 60°C) and HF (50%) was also

evaluated as a function of the annealing treatment. Finally, the films were integrated in ISFET devices for pH detection.

## RESULTS

The characteristics of the  $Ta_2O_5$  films depended on the nature of the annealing treatment. Stoechiometry, permitivity, fixed charges, C-V hysterisis, crystallinity were evaluated each type of thermal annealing performed. O/Ta ratios varying from 2.4 to 2.5 were found by RBS and WDS. Some carbon, less than 3%, was also present in the as-deposited films. The amount was reduced after the annealing in oxygen. Increasing the partial pressure of oxygen during the deposition also helped to decrease the quantity of carbon in the films. The permitivity for the asdeposited films was 24 and could increase to 40-50 for films annealed at 700 to 800°C. The transition from an amorphous to a polycrystalline film occured when the films were annealed at temperatures higher than 625°C.

The chemical resistance of the films to HF and KOH improved with the annealing temperature increasing. The films were used as a protective coating for glass and silicon etching. The optical properties of the amorphous films varied slightly with the annealing treatment (n=2.15-2.2, Eg=3.8-4.2 eV), which make them suitable for integrating waveguides in Optical-MEMS.

The electrical and chemical sensitive properties of the  $Ta_2O_5$  films are of importance in Microsystems mainly for applications in ISFETs 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  $Ta_2O_5$ . The films electrical characterisitics were then optimized for a gate dielectric made of  $Ta_2O_5$ and SiO<sub>2</sub>. An optimum annealing in oxygen showed to reduce the hysterisis. Fixed charges could also be reduced by annealing the films in  $H_2/N_2$ . ISFET devices made using this optimized gate insulator showed a good pH sensitivity of 59 mV/pH.

## CONCLUSION

The combination of a LPCVD reactor with TAT\_DMAE as precursor allows deposition of  $Ta_2O_5$  thin films using a batch fabrication process. The influence of the deposition parameters and annealing treatments on the  $Ta_2O_5$  films characteristics was investigated with the aim of optimizing for applications in the field of Microsystems.

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