Ta_2O_5 thin films for integrated circuits applications elaborated by electrostatic spray deposition: SEM and TEM characterizations.

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Abstract – A new spray technique (Electrostatic Spray Deposition (ESD) has been used to elaborate Ta_2O_5 thin films. The density and continuity of such films are a key point for applications. Because of the nanoscale film thickness, high resolution characterizations have been carried out to check the deposit quality. We report here the results of SEM and TEM on cross sections of the as deposited and annealed thin obtained by ESD.

1. Introduction

Advances in microelectronics generate a growing demand for new insulator films for MOS capacitors. Because of its high dielectric constant, Ta_2O_5 appears as a promising gate dielectric material to replace SiO_2. The challenge in the synthesis of Ta_2O_5 thin films is to obtain dense and thin homogeneous layers at low deposition temperature. Besides Ta_2O_5 thin films are also expected to act as a diffusion barrier to copper diffusion in Si or SiC devices. Because of this large application potential, it is relevant to investigate the film elaborated by the new ESD technique. Actually, the ESD technique briefly described below is of high interest for several reasons. It is a low cost process, with good reproducibility, which allows a control of the film morphology and conformity. The ESD technique is a spray technique in which the thin film is formed by the spreading of droplets on the substrate. In the present work, thin films were deposited using a vertical ESD set-up (Figure 1) described in previous papers [1][2]. The precursor solutions were prepared by dissolving tantalum ethoxide in diethylene glycol monobutyl and ethanol. The experimental conditions (Table 1) were determined to obtain films with thickness below 30 nm [2].

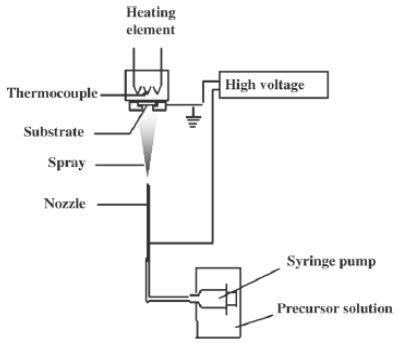

Figure 1 – Experimental ESD set-up.

Organometallic precursor	$Ta(OC_2H_5)_5$
Solvent composition	33 vol. % C_2H_5OH 67 vol.% $C_8H_{18}O_3$
Solution concentration	0.00625 M
Substrate temperature	110°C
Solution flow rate	0.49 ml/h
Nozzle to substrate distance	20 mm
Deposition time	5 minutes
Applied voltage	4 kV
Substrate	Si (100) treated with HF 10 vol.%

Table 1 – Experimental ESD conditions

However, because deposition occurs in liquid state, conformity for the as deposited states is expected but, the density, further stability of the thin film and reactivity with the substrate are questionable. Therefore to complement the SEM investigation, a TEM study of cross section has been carried out on samples as deposited and annealed at various temperatures in air and in helium atmospheres.

2. SEM and TEM characterizations

2.1. As deposited films

According to plane view (Figure 2a) and cross section (Figure 2b) examined by FE-SEM, the as deposited film by ESD was found dense and with a 25 nm thickness.

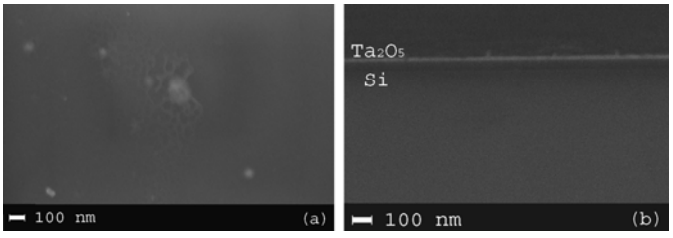

Figure 2 *– FE-SEM images of film deposited on Si substrate (a) plane view (b) cross section*

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TEM observation carried out on a cross section has confirmed that the as deposited film was continuous (Fig. 3a) but formed of very small crystals (Fig. 3c). An 2 nm amorphous silica layer was observed at the interface between the film and the substrate (Fig. 3b). This silica layer may be due to the fact that the deposition occured in air atmosphere.

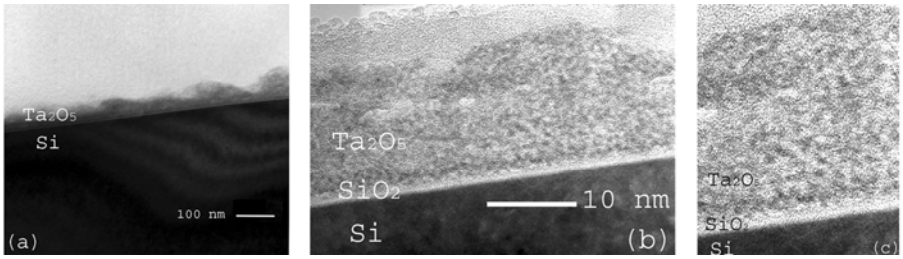

Figure 3 *–TEM images of the as deposited Ta_2O_5 film in cross section: (a) continuous deposit with a variable thickness (b) at the interface with the substrate, note a 2 nm silica layer (c) black dots contrast revealing nanocrystallite within the as deposited film.*

2.2 Influence of thermal treatment and atmosphere

2.2.1. In air atmosphere

The as deposited film has been annealed in air at different temperatures (750, 800 and 850°C) for 1 hour. After annealing at 750°C (Fig. 4a) the film was still amorphous and started to crystallize about the temperature above 800°C (Fig. 4b and 4c). The film thickness decreased by increasing the temperature from 750 to 850°C. Owing to the Ta strong absorption contrast, we observed an increase in density compared to the as deposited film. However simultaneously the silica layer at the interface with the Si substrate has grown during the annealing and reached a 15 nm thickness after annealing at 850°C.

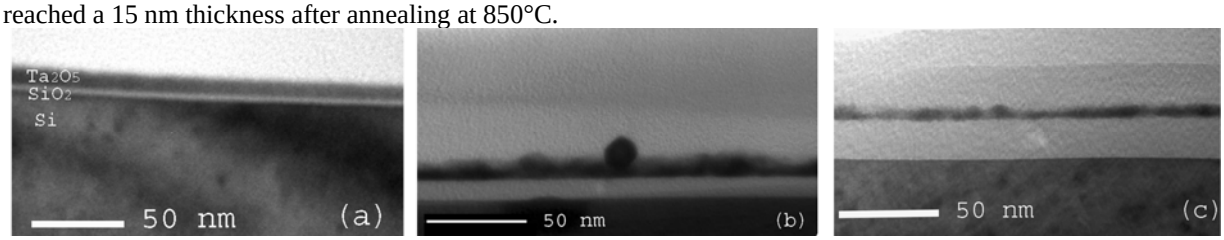

Figure 4 – TEM images in cross section of a Ta_2O_5 film annealed in air at: (a) 750°C (b) 800°C (c) 850°C.

2.2.2. in helium atmosphere

This annealing aimed at testing the influence of atmosphere on the SiO_2 layer growth. As illustrated in Figure 5, after thermal post treatment in helium at 850°C, the silica SiO_2 layer was approximately 5 nm thick (Fig. 5b). Though still forming a continuous layer (Fig. 5a), the Ta_2O_5 film developped well defined crystals (Fig. 5c).

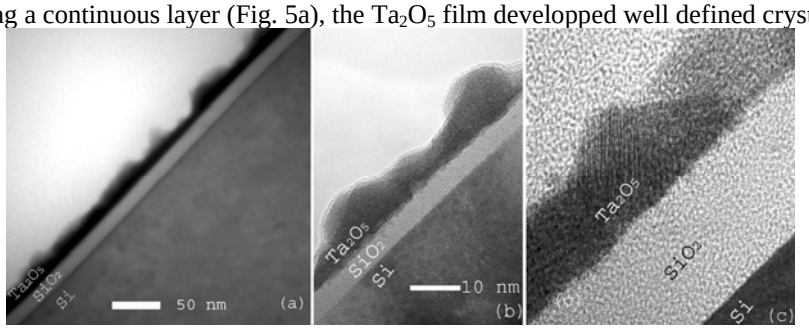

Figure 5 – TEM images in cross section of a Ta_2O_5 film annealed in helium at 850°C.

3. Conclusion

The present nanostructural characterizations aim at testing the quality of Ta_2O_5 films elaborated by a new spray technique. Though the thickness and continuity of the deposit are quite satisfactory, it remains to better control the elaboration atmosphere and reactivity at the interface with the substrate. Annealing in He atmosphere already gives encouraging results.

4. References

[1] A. Lintanf, R. Neagu, E. Djurado, *Nanocrystalline Pt thin films prepared by electrostatic spray Deposition for automotive exhaust gas treatment*, Solid State Ionics **177** (2006) 3491-3499.

[2] A. Lintanf, A. Mantoux, E. Blanquet, E. Djurado, *Elaboration of Ta_2O_5 Thin films using Electrostatic Spray Deposition For Microelectronic Applications*, J. Phys. Chem. (2007) in press.