

## Tungsten Silicide Thin Films Preparation by Magnetron Sputtering

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**Abstract:** Tungsten silicide thin films were deposited on a Si substrate by magnetron RF sputtering of a composite target. In the first experiment, sputtering of a  $WSi_2$  target was performed in a silane-argon atmosphere (reactive sputtering). Thin films with ratio  $Si/W = 1.07$  were obtained at the largest partial silane pressure allowed for in the sputtering apparatus. In the second experiment, Si richer thin films were obtained by non-reactive sputtering of a  $WSi_{2.7}$  target where a  $Si/W$  ratio of 1.7 was found. The ratio  $Si/W$  was measured by the Rutherford backscattering technique. After annealing for 30 minutes in a  $N_2$  atmosphere at  $1000^\circ C$  X-ray diffraction shows the tetragonal structure of  $WSi_2$ . The resistivity value of these thin films is found to be about  $60\mu\Omega\text{-cm}$ .

**Key words:** Tungsten silicide, sputtering, thin films, stoichiometry, resistivity

### INTRODUCTION

Polycrystalline silicon (Poly-Si) has been intensively used in the fabrication of integrated circuits in MOS technology where it is encountered as a gate or interconnection material. Poly-Si has attractive properties such as good stability at high temperatures as well as good adherence. The relatively high resistance (about  $20\text{-}30\mu\Omega\text{-cm}$  for a  $5000\text{\AA}$  thickness film of Poly-Si)<sup>[1]</sup> is a major limitation in circuit performance for very/ultra large scale integration (VLSI/ULSI) technology. As the device lines become narrower and longer as the density of the device becomes higher, which results in a large signal delays in interconnection<sup>[2]</sup>. Alternative materials to Poly-Si must have good resistance to chemical reagents and to the oxidized ambient during the integrated circuits fabrication process<sup>[3]</sup>. Some of such materials are refractory metal silicides among which the most studied ones are  $WSi_2$ ,  $MoSi_2$ ,  $TiSi_2$  and  $TaSi_2$ <sup>[4]</sup>.

In the present article we summarize our work carried out on thin films deposited by cathodic sputtering of cold-pressed targets made of tungsten silicide  $WSi_x$ . We used two different targets, one with  $x = 2$  (reactive sputtering only, the other with  $x = 2.7$  and with the resistivity of the various  $WSi_x$  films being measured before and after annealing.

### MATERIALS AND METHODS

No particular treatment of the substrate surface was made before deposit process. The average growth speed was  $36\text{\AA min}^{-1}$  in all experiments. The tungsten silicide thin films were deposited in three different ways on a low phosphorus-doped monocrystalline Si(110) substrate:

**Reactive sputtering:** Silane is first diluted in argon plasma ( $SiH_4$  (5%), argon (95%)), this gas being very flammable the maximum partial pressure used for it was  $10^{-4}$  mbar while the incident power was 450W for all deposited films. According to the reaction  $SiH_4 \rightarrow Si + 2H_2$ , the atomic ratio  $Si/W$  will increase with partial silane pressure. Figure 1 shows an RBS spectra of as-deposited thin film at  $10 \times 10^{-3}$  mbar of partial  $SiH_4$  reactive gas pressure. The calculated ratio  $Si/W$  is found to be 1.07 which is different from the initial target ( $WSi_2$ ) stoichiometry, the films are tungsten rich.

**Non-reactive sputtering:** To increase this ratio another composite target  $WSi_{2.7}$  was sputtered with the same incident power. The RBS spectra of our deposited films are shown on Fig. 2 and the calculated ratio is 1.7. The deposited films are tungsten rich too.

**Non-reactive sputtering on heated substrates:** The substrates were heated up to  $500^\circ C$  during the deposit process, this heating can help the desorption of impurities such as hydrocarbons,  $H_2O$ ... out of the surface substrate.

We also studied the surface morphology of the films using a scanning electron microscope and measured their resistivity using a four-point probe method, last their thickness was measured using an Alpha-step profiler.

### RESULTS AND DISCUSSION

Our RBS measurements showed that the deposited thin films are tungsten rich compared to the sputtered stoichiometric target  $WSi_2$ . It is known that sputtering of a composite target produces generally thin films with

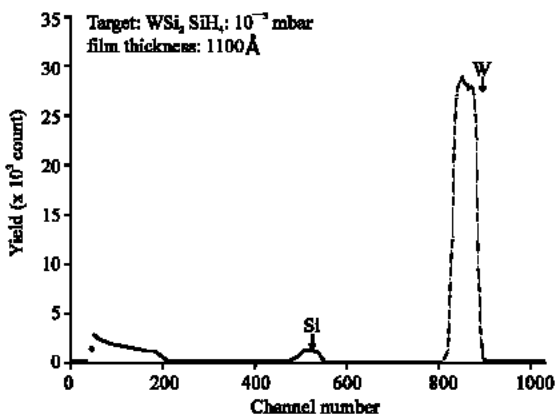


Fig. 1: RBS spectra of reactive sputtered film from a  $WSi_2$  composite target

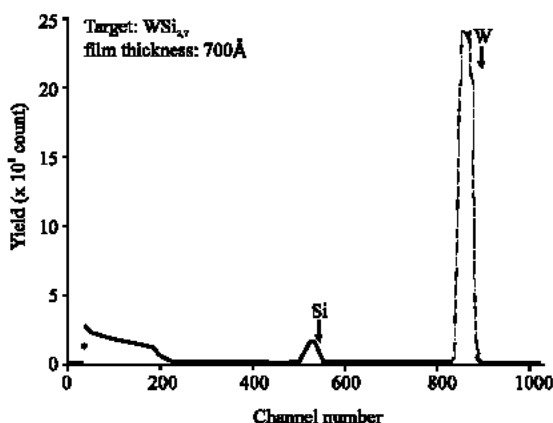


Fig. 2: RBS spectra of sputtered film from a  $WSi_{2.7}$  composite target

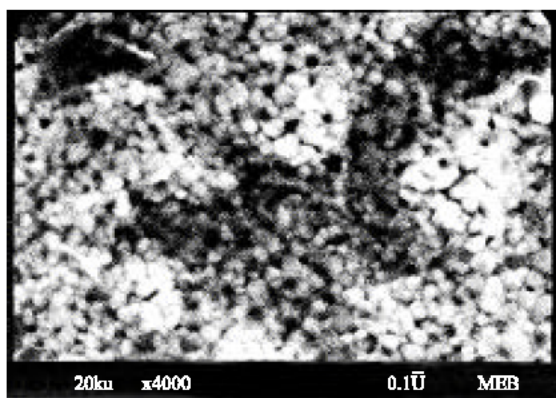


Fig. 3: SEM image of annealed thin film surface

stoichiometry different from that of the target<sup>[5,6]</sup> especially in sputtering of a metal silicide where the ratio of silicon-to-metal of the film is different of that the sputtered target. This difference in film composition can be attributed to two factors<sup>[7]</sup>: A difference in

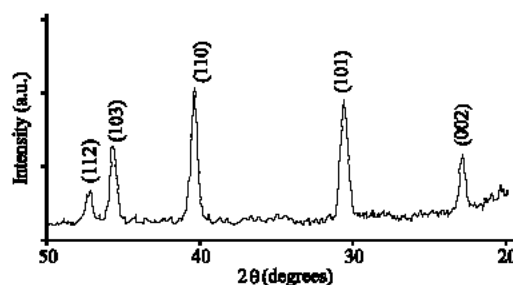


Fig. 4: X-ray diffraction spectra of annealed film

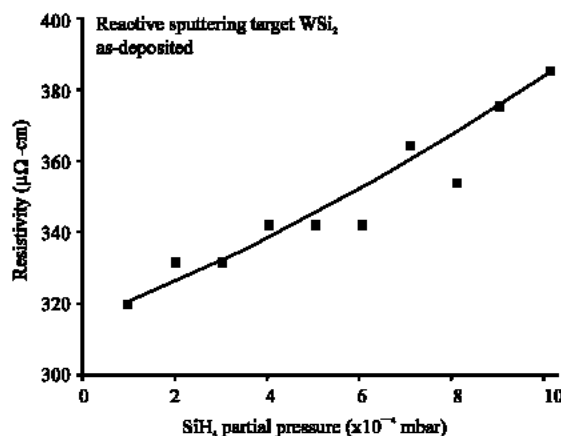


Fig. 5: Resistivity of as-deposited thin film as function of partial silane pressure

angular distribution of the sputtered species and the sticking coefficient of metal and silicon. The ratio of silicon-to-metal affects film properties such as resistivity and crystallisation during annealing<sup>[8]</sup>. On the other hands these measurements showed a 2% presence of argon. The thickness of the thin films increased about 100-200Å after annealing because of the diffusion of silicon from the Si-substrate into the thin films during the formation of  $WSi_2$ . This diffusion can be overcome by deposition of silicon rich silicide thin films.

**XRD and surface morphology:** The results of XRD analysis are presented in Fig. 3. After annealing, the diffraction pattern contains lines characteristic of the tetragonal structure of  $WSi_2$ . The surfaces of the various films are smooth and shiny before and after annealing. Figure 4 shows a Scanning Electronic Microscope (SEM) photograph of the annealed thin film.

The same Fig. 5 indicates an average grain size of about 1600Å which size influences significantly the films resistivity<sup>[9]</sup>.

**Resistivity measurements:** The resistivity of the films deposited by reactive sputtering increases slightly from

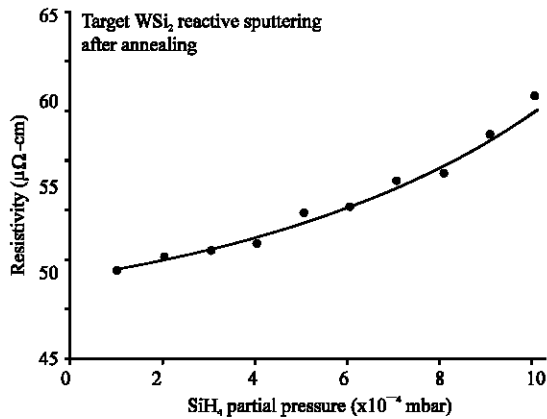


Fig. 6: Resistivity of annealed thin film as function of partial silane pressure

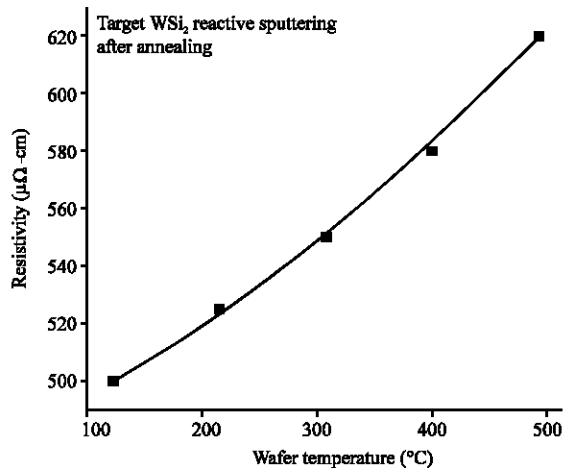


Fig. 7: Resistivity of thin film as substrate temperature

320 to 390 μΩ-cm with the partial silane pressure as shown Fig. 4. After annealing, this resistivity decreases and varies from 50 to 60 μΩ-cm as shown Fig. 6. Films obtained after sputtering of a WSi<sub>2.7</sub> target and as deposited had a resistivity of about 480 μΩ-cm and 60 μΩ-cm after annealing. This value is slightly larger than was reported before<sup>[10,11]</sup>.

For heated substrates the value of resistivity was higher. Figure 7 shows the resistivity of as-deposited films as a function of substrate temperature during film growth. This higher value of resistivity is related to the change in the crystalline phase of the deposited films with temperature<sup>[10]</sup> because the conduction mechanism<sup>[7,12]</sup> is different resulting in lower carrier mobility compared to the amorphous structure. After annealing the resistivity of our thin films was about 60-65 μΩ-cm.

## CONCLUSION

Tungsten silicide films were prepared by magnetron RF sputtering in different conditions by sputtering of WSi<sub>2</sub> and WSi<sub>2.7</sub> targets. The films thus obtained were tungsten rich before annealing. One can say that it is difficult to control the stoichiometry of the obtained films with this deposition technique. Moreover, the RBS measurements indicate the presence of argon within the thin films. More detailed investigations are however needed to show evidence of other impurities in these films. The resistivity of the different WSi<sub>x</sub> films varies from 320 to 390 μΩ-cm before annealing and the XRD shows that their structure is tetragonal after annealing with a resistivity of about 60 μΩ-cm. This value is slightly larger than that obtained with other tungsten silicide deposition techniques.

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