



## **Effect of tungsten sputtering current on structural and morphological properties of WC thin films**

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### **Abstract**

Nanostructured tungsten carbide (WC) thin films were deposited on Si wafer by DC magnetron co-sputtering using tungsten and graphite targets. The effect of tungsten sputtering current on the structural and morphology of thin film was investigated by X-ray diffraction (XRD), Raman scattering spectroscopy and field emission scanning electron microscopy (FESEM), respectively. The XRD results showed that all film formed nanocrystalline WC phase with preferred orientation along (100) plane. The crystallize size of WC was increase with increasing the tungsten sputtering current. In additional, WC<sub>1-x</sub> phase were observed when increasing the tungsten sputtering from 0.40 to 0.60 A.

**Keywords:** Tungsten carbide; Co-sputtering; thin films

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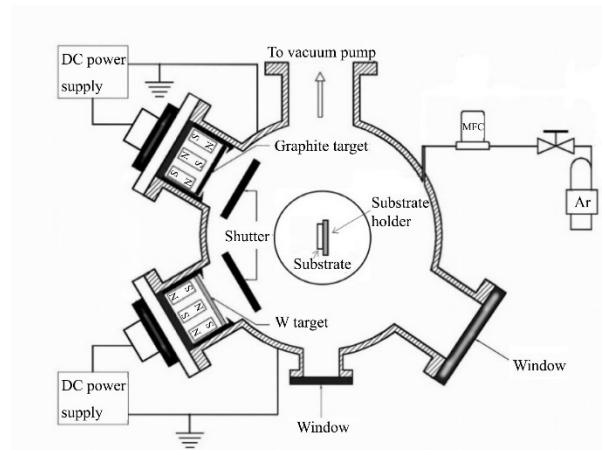
### **1. Introduction**

Tungsten carbide (WC) thin film has been widely using in the production of many applications due to their useful properties such as chemical resistance, high thermal stability, and mechanical hardness [1 – 3]. Many deposition techniques have been used to deposit the WC thin films such as chemical vapor deposition [4], plasma enhanced chemical vapor deposition (PE-CVD) [5], pulsed laser deposition [6], and sputtering methods [7 – 9]. Among these methods sputtering method is commercially used and has been established for industrial applications. The properties of WC thin films deposited by a sputtering technique were highly sensitive to many deposition parameters including, sputtering current, sputtering power, bias substrate voltage and substrate temperature. In this work, the WC thin films were prepared by a DC magnetron co-sputtering and this work was focused on the effect of tungsten sputtering current on structural and morphological properties of WC thin films.

### **2. Materials and methods**

WC thin film were deposited on silicon wafer substrates by a DC magnetron co-sputtering, whose schematic diagram was shown in Fig. 1. High purity of Tungsten (99.95% purity, Kurt J. Lesker) and graphite (99.99% purity, Kurt J. Lesker) disks with diameter of 3 inches were used as the sputtering target. Before deposition, the sputtering chamber was evacuated to the base pressure of  $5 \times 10^{-5}$  mbar by a diffusion pump accompanied with a rotary pump. Pure Ar (99.99%) sputtering gas was introduced into the vacuum chamber through a mass flow controller. The argon flow rates were fixed at constant at 3 sccm. Then, the targets were pre-sputtered to clean the target surface for 5 min. In the deposition process, The graphite sputtering current was fixed at 0.60 A for all of the deposition conditions whereas tungsten sputtering current were varied from 0.20 to 0.60 A in order to investigate the effect of tungsten

sputtering current on the structural and morphology of WC thin film. Table 1 presented the deposition parameters for depositing the WC thin films.



**Fig. 1** Schematic diagram of DC magnetron co-sputtering system

**Table 1** Deposition parameters.

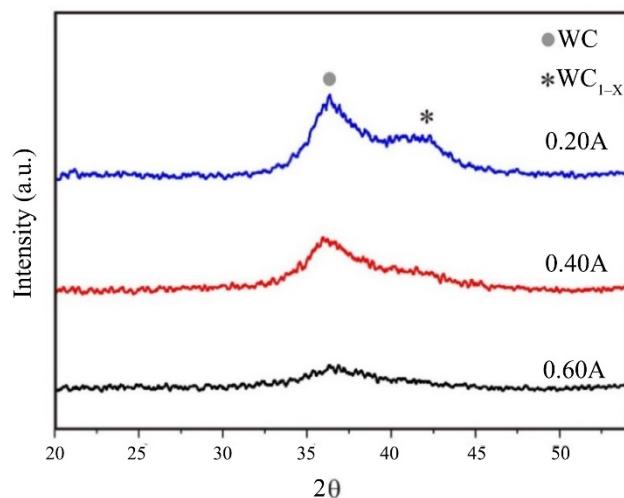
Parameters	Values
Target	Tungsten, graphite
Substrate	Si wafer
Base Pressure	$5 \times 10^{-5}$ mbar
Working Pressure	$3.30 \times 10^{-3}$ mbar
Ar flow rate	3 sccm
Deposition time	10 min
Tungsten sputtering voltage	390 V
Graphite sputtering voltage	400 V
Graphite sputtering current	0.60 A
Tungsten sputtering current	0.20 – 0.60 A

The structural and microstructural properties of the thin films were examined by X-ray diffractometer (XRD, Bruker D8 ADVANCE) with CuK $\alpha$  radiation as X-ray source and measured in a low angle mode and Raman spectroscopy (NT-MDTNTEGRA spectra) with a 632.80 nm laser beam. The surface morphology and thickness were evaluated by Field-Emission Scanning Electron Microscope (FE-SEM, Hitachi, S-4700).

### 3. Results and Discussion

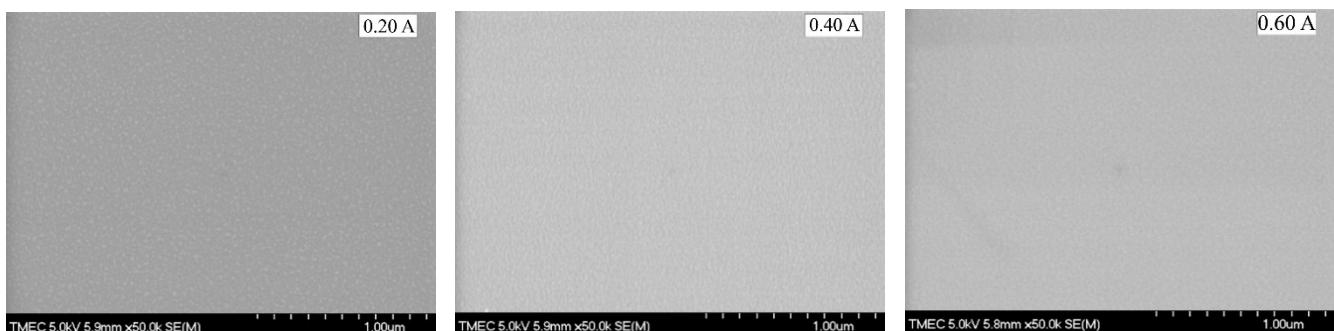
The grazing angle X-ray diffraction patterns of WC thin films deposited on silicon wafer are shown in Fig. 2. It was observed that all samples showed the broad diffraction peaks corresponding to the WC (100) at  $2\theta = 36.4^\circ$ , however a diffraction peak of graphitic carbon phase or tungsten metal phase was not observed. As the tungsten sputtering current increases, the intensity of the WC (100) diffraction peak become decrease and broader. In additional, it was also found that the broad shoulder diffraction peak occurred at  $2\theta = 42.6^\circ$  when increasing the tungsten sputtering current from 0.40 to 0.60 A. This peak was attributed to the WC<sub>1-x</sub> phase (JCPDS card: 20-1316). This result suggested that when the tungsten sputtering current was increasing, the energy and quantity of tungsten ions form tungsten target was increased and bonded carbon ion to form as WC or WC<sub>1-x</sub> phase. The average crystallite size of

WC were calculated from the full width at half maximum (FWHM) of the WC (100) diffraction peak by using the Scherrer formula [10]. It was found that the average crystallite size of WC were nanocrystalline and the crystallite size increased with increasing the tungsten sputtering current. The obtained values were 1.90, 2.30 and 2.70 nm for the tungsten sputtering current of 0.20, 0.40, and 0.60 A, respectively.

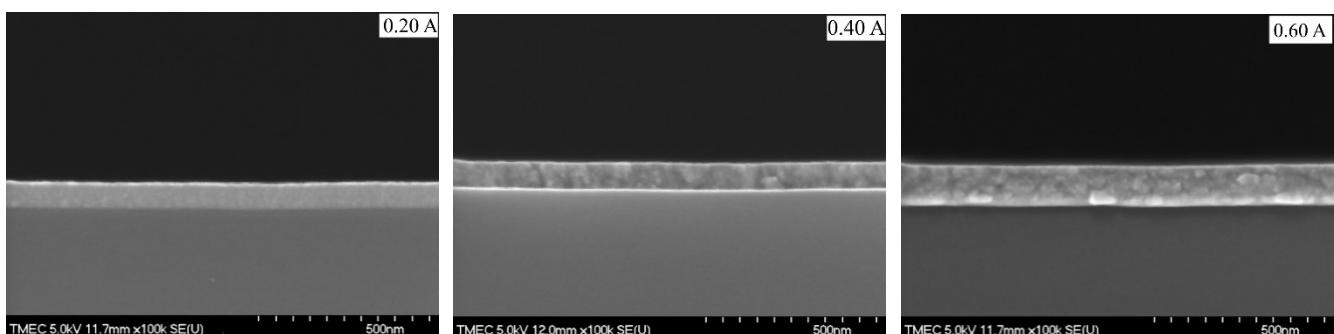


**Fig. 2** XRD patterns of WC thin films deposited at different tungsten sputtering current.

Fig. 3 shown the FESEM photographs of the surface morphologies of WC thin films. All thin film exhibit a uniform nanocrystalline grains (grain size less than 10 nm) and a very smooth surface. The FESEM photographs of the cross-section morphologies of WC thin films are shown in Fig. 4. It was clearly seen that the thin film thickness and the grain size were increased with increasing tungsten sputtering current. This evident was corresponded to the crystallite size which determined from XRD pattern.

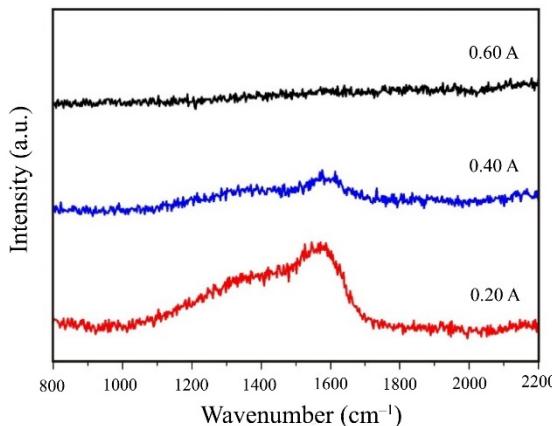


**Fig. 3** FESEM image of surface morphologies of WC thin films deposited at different tungsten sputtering Current



**Fig. 4** FESEM image of cross-section morphologies of WC thin films deposited at different tungsten sputtering current

In order to investigate the carbon phase in WC thin films, All Raman spectra in this work were recorded in range of 800 – 2,200 cm<sup>-1</sup>. The Raman spectra of WC thin films deposited at different tungsten sputtering current are shown in Fig. 5. For the film deposited at tungsten sputtering current 0.20 A, The raman results shown a strong band at around 1,581 cm<sup>-1</sup> (G band) and a shoulder weak band at around 1,340 cm<sup>-1</sup> (D band). The G and D bands are attributed to the first-order scattering from the E<sub>2g</sub> phonon of sp<sup>2</sup> carbon bonding and structural defects carbon structure, respectively. These bands indicate the presence of amorphous phases of carbon in the WC films which not detected by XRD analysis. In addition, The intensity of D band and G band decreased with increasing tungsten sputtering current which indicate that increasing of W content inside the deposited films which from as WC and WC<sub>1-x</sub> phases. This result confirm the XRD results indicating increasing of the crystallinity of WC films when the tungsten sputtering current increased.



**Fig. 5** The Raman signals of WC thin films deposited at different tungsten sputtering current.

#### 4. Conclusion

In summary, the effect of tungsten sputtering current on the structural and morphology of WC thin films were investigated. XRD analysis showed the film exhibited nanocrystalline WC (100) phase at tungsten sputtering current 0.20 A. With increasing the tungsten sputtering current, the thin films consist of two phases, WC phase and WC<sub>1-x</sub> phase. For the FESEM results showed the film thickness and the grain size were increased with increasing tungsten sputtering current. The Raman spectra revealed that the increasing of tungsten sputtering current had significantly influenced on amount content of carbon in the WC thin film. The presence of amorphous carbon phases in the WC thin film are promising candidates for use in tribological applications.

#### 5. Acknowledgement

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