

# STUDY OF THERMAL STABILITY BEHAVIOR OF MoN & WN THIN FILMS IN ULSI

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## ABSTRACT

*The thermal stability of amorphous MoN & WN thin films was investigated against Cu diffusion. The MoN & WN layers were reactively sputtered of 50 nm by using a different nitrogen flow rates. The phase identification was evaluated by X-ray diffractometer (XRD), four probe method and scanning electron microscope (SEM). Results indicate that the amorphous 50 nm MoN layer acts as a good diffusion barrier up to 600 °C for Cu metallization and on the other hand the amorphous WN layer acts as a good diffusion barrier up to 500 °C.*

## KEYWORDS

*Reactive dc sputtering, diffusion barrier, Cu metallization.*

## 1. INTRODUCTION

The fabrication of ultra large scale integrated (ULSI) devices requires the development of new interconnection technologies for deep submicron processes. Particularly, the technologies of the diffusion barrier between the interconnection materials and the silicon in the contact hole, or between interconnection materials play a critical role with the continued scaling down of devices. Among various materials, conventional MoN & WN thin films have intensively been investigated for the extended use in deep submicron regimes.

Multilevel metallization of ULSI circuits has become an area of intense research interest as devices are scaled down in size in order to increase circuit integration. Aluminum and its alloys have been commonly used as the main metallization materials. Aluminum, however suffer from major limitations such as electro migration and stress voiding induced open circuit failure. Hence copper is being evaluated for ULSI metallization. The current trend in shrinking integrated circuits to achieve greater logic density and chip performance places a great demand on the electronic materials used in the construction of silicon devices and packages [1]. In particular, thin-film wiring contacts and interconnections used in substrate and chip packaging are very important part of the reliability and performance characteristics of ultra-large scale integration (ULSI) devices [2]. Copper and its alloys are being used in thin-film wiring because they offer an attractive alternatives to Al based materials due to its lower electrical resistivity, superior resistance to electro migration and stress voiding [3]. However one of the major drawback of copper is its rapid diffusion in silicon, resulting in deterioration of devices at low temperatures. Therefore, provision of a diffusion barrier is necessary between Cu and Si [4-10].

In this work, MoN & WN thin films were deposited by d.c reactive magnetron sputtering from a Mo & W target. The crystalline and amorphous microstructure formation is studied. In addition, the thermal stability of amorphous MoN & WN barrier layer is investigated by using Si/MoN(50nm)/Cu and Si/WN(50nm)/Cu structures and vacuum annealing from 300 °C to 700 °C for 30 minutes.

## 2. EXPERIMENTAL DETAILS

In this we discuss about the procedure for sample preparation and characterization in explained in detail. The sample preparation includes two main steps; first one is of cleaning and after that deposition of thin film using sputtering technique.

### 2.1. Sample Preparation

**Cleaning of Samples**-The substrates used in this experiment were N-type (100)-oriented Si wafers and glass substrates. Mo/W and MoN/WN thin films were deposited by DC sputtering on silicon (100) wafer/glass plates. These substrates were cleaned by standard cleaning process. They were subjected to ultrasonication for 10 minutes in acetone solution. Following this, these substrates were cleaned with standard RCA1 ( $\text{NH}_4\text{OH}+\text{H}_2\text{O}_2+\text{H}_2\text{O}$ ; 1:1:5) and RCA2 ( $\text{HCl}+\text{H}_2\text{O}_2+\text{H}_2\text{O}$ ; 1:1:5) solution for 10 minute. After drying, wafers were ready for thin film deposition.

### 2.2 Thin Film Deposition Process

A reactive DC magnetron sputter deposition unit was used as the sputter system in this Experiment. The Silicon wafer acts as the anode and Molybdenum target acts as the cathode. The gases used are argon and nitrogen. The magnetron was generated by a ring shape permanent magnet placed in the gun behind the target attachment surface. The size of the Mo/W target was 76.2mm (3 inches) and the purity of the target is 99.99%. Vacuum was drawn in the chamber by a mechanical rotary pump. The background pressure in the chamber was less than  $5.5 \times 10^{-6}$  torr prior to deposition. After reaching the desired base vacuum, the ultrahigh purity (99.999%) argon gas or the mixture of argon and nitrogen gas was introduced into the chamber through the activated throttle valve. The nitrogen gas flow was varied between from 0 to 20 Sccm. During depositions, the sputtering power, argon gas flow and total gas ( $\text{Ar} + \text{N}_2$ ) pressure were kept constant at 80 W, 40 Sccm and  $3 \times 10^{-2}$  torr, respectively; the substrate holder was placed at 40 mm far from the target. During Pre-sputtering of the target was done before the deposition to remove any possible nitrides and oxides from the target surface. One hour pre-sputtering was carried out prior to the deposition of MoN/WN coatings. The power of pre sputtering was set at just the same as the deposition condition i.e. 80W. A shutter above the substrates was applied in pre sputtering to prevent any deposition failing down on the substrate during pre sputtering. After pre-sputtering, the shutter above the substrates was removed and the deposition started.

### 2.3 High temperature vacuum annealing

All deposited samples were annealed in Hind High Vacuum Coating Unit at 300-700 °C, the annealing time at each temperature was 30 minute. The system is equipped with rotary and diffusion pump to achieve pressure in the range of  $5 \times 10^{-5}$  Torr.



Figure1. Vacuum coating unit used for the samples annealing

The different techniques were used to characterization the as-deposited and annealed films. A four point probe was used for measuring the sheet resistance of the deposited films. Thin films X-ray diffraction

spectra of MoN & WN are recorded with X-ray diffractometer (XRD, Panalytical's X'Pert Pro) operating in the  $\theta$ -2 $\theta$  Bragg configuration using Cu K $\alpha$  radiation. Data were collected at a scan rate of 0.075  $^\circ$  min<sup>-1</sup> and sampling interval of 0.002  $^\circ$ .

To test the thermal stability of the MoN & WN barriers, copper films of 100 nm in thickness were sputtered over the 50 nm thick MoN & WN layers. The Si/MoN/Cu and Si/WN/Cu samples were then annealed in a vacuum furnace for 30 minutes. Interfacial behavior and phase formation of the samples after annealing were characterized by JEOL JSM-6390 LV (INCA from Oxford instruments) Scanning Electron Microscope (SEM) and XRD, respectively. Variation in sheet resistance of samples before and after annealing was measured using a four point probe method.

### 3. INTRODUCTION TO DIFFUSION BARRIER

Diffusion barrier layers are integral part of many metallization systems for on-chip interconnects applications. These are located between metal lines or contacts to semiconductors, insulators, and other metals. The issue of barrier layers emerged at the early stage of metallization in microelectronics since the reactivity of most interconnect metals results in many types of direct or indirect failure modes. However, the research and development of barrier for copper metallization is still under way, as the copper metallization technology is being evolved. The concept of use of barrier layers in metallization system is simple: two materials that have unfavorable chemical interaction are kept separate by an intermediate layer.

Diffusion describes the details of atomic migration associated with mass transport through materials. The resulting atom movements reflect the marginal properties of materials in that only a very small fraction of the total number of lattice sites, namely, those that are unoccupied, interstitial, or on surfaces, is involved [11]. Surface diffusion is a very important transport mechanism of thin films because of the large ratio of the number of surface-to-bulk atoms. This mechanism plays an active and vital role in film nucleation and growth processes [12].

Grain-boundary diffusion is an important concern while we are talking about the thin film deposition because film usually has small grain size and high grain boundaries densities. Because of the low diffusion activation energies, grain boundary diffusion is the dominate diffusion mechanism for thin films at low temperature [12]. Hence the critical issue for effective diffusion barrier is to either stuff or eliminate the grain boundaries.

Transition metal nitrides constitute an important class of interstitial compounds with exceptional chemical stability, high strength, high hardness, high melting point and good electrical conductivity. For example, molybdenum and tungsten nitrides are good barriers against diffusion of copper in microelectronic circuits. They can also be used in electrodes for thin film capacitors and field effect transistors. With a lower electric resistivity and better resistance to electro migration than those of aluminum, Cu is highly attractive for interconnections for ultra-large scale integration device applications. However, Cu is mobile, creates a deep trap level in Si and causes degradation of device reliability. It is necessary to use a diffusion barrier between Si and Cu to retard the inter diffusion between Cu and Si. MoN & WN thin film is one of the promising materials for diffusion barrier.

Here we will discuss about the three main types of diffusion barrier such as stuffed diffusion barrier, sacrificial diffusion barrier and amorphous diffusion barrier [12, 13]. Stuffed diffusion barriers concern on the segregation of impurities along otherwise rapid diffusion paths such as grain boundaries. Sacrificial diffusion barriers maintain the separation of metal and substrate only for a period of time. Sacrificial barriers exploit the fact that reactions between adjacent films in turn produce uniform layered compounds that continue to be separated by a narrowing barrier film. So long as the barrier remains and compounds possess adequate conductivity, this barrier is effective. The application of Ti as a barrier material which reacts with Si to form TiSi<sub>2</sub> and with Al to form TiAl<sub>3</sub> is a good example. On the other hand, amorphous barriers have no grain boundaries by which the rapid diffusion occurs. During deposition process, some of the deposited alloys intermixing physically with individual elements shows

different long range order crystalline structures and with limited terminal solubility, indicating the positive entropy of formation for solid solution.

#### 4. LITERATURE SURVEY

A large number of refractory metals such as Mo, W, Cr, Co, Pd, Ti, Ta and Nb and their alloys either in polycrystalline or amorphous phase have been investigated for their diffusion properties in terms of their high thermal stability and low resistivity [14, 15].

Transition metals, transition metal alloys, transition metal nitrides, oxides, or borides act as a diffusion barrier in an effective manner. The following is the different types of diffusion barriers reported by literatures such as Nitride Types: MoN, WN, AlN, Si<sub>3</sub>N<sub>4</sub>, TiN<sub>x</sub>, ZrN, TaN etc. [13] [16-24]. Silicate Types: MoSi<sub>2</sub>, HfSi<sub>2</sub>, Ta<sub>56</sub>Si<sub>44</sub>, WSi<sub>2</sub> etc. [25]. Carbide Types: TiC, SiC, WC, Mo-C etc. [26] [27]. Miscellaneous ternary alloys: Ta<sub>36</sub>Si<sub>14</sub>N<sub>50</sub>, Ta-Si-N, Mo<sub>16</sub>Si<sub>34</sub>N<sub>50</sub>, Ti-Si-N etc. [28, 29].

Basically, amorphous structure is a best one structure for diffusion barrier due to lack of fast diffusion path, e.g., grain boundaries. In this research, we present the formation of an amorphous MoN & WN diffusion barrier by DC reactive magnetron sputter deposition. The phase structures and sheet resistivity of the MoN thin film deposited under different nitrogen contents has been reported by Yimin et al [11]. The stability of the amorphous diffusion barrier has been also discussed. It was noticed that as the total sputtering gas pressure increased, the intensity of  $\gamma$ -Mo<sub>2</sub>N peak decreased, indicating a decrease in the crystallinity of the molybdenum nitride. The decrease in film crystallinity with the increasing total sputtering gas pressure may result from the difference in deposition rate at different total sputtering gas pressure. At high total sputtering pressure, the deposition rate is higher, thus the atoms have less time to diffuse on the substrate surface to rearrange themselves in a more stable low energy lattice site, before another layer of atoms arrive. So the degree of film crystallinity deposited at a higher total sputtering gas pressure is lower. Yimin et al [11] study on the barrier properties and failure mechanism and it was observed that the Mo film is a poor barrier against Si diffusion. On the other hand, when the N/Mo ratio increases, the diffusion of Si atoms becomes less significant. As a result, the MoN<sub>x</sub> film with higher N content prohibits a better barrier property. After vacuum annealing at 600°C, 700°C, and 800°C for 60 minutes, it was observed that the crystallinity increases when the annealing temperature increases.

In the last decade, thin layers of Molybdenum nitride have been investigated as materials with a wide variety of potential applications that include catalysis, superconductivity, diffusion barriers for copper interconnects gate electrode materials for high-k gate dielectrics, and as corrosion- and wear-resistant coatings. The Mo-N system was reported to crystallize in three possible phases, namely, face-centered cubic Mo<sub>2</sub>N, tetragonal Mo<sub>2</sub>N, and hexagonal MoN. In many integrated circuits, depositing a metal layer on a substrate can be performed by thermal evaporation, electron beam evaporation, or sputtering. Molybdenum has a high melting point (2896 K) and a low vapour pressure (3.47 Pa at 3000 K) which makes Molybdenum ideal for sputtering. For this reason, sputtered Molybdenum sticks well to glass substrates, and the use of molybdenum can be commonly found on liquid crystal TFT displays as the gate metal on the TFT.

Song et al [30] studied the diffusion barrier performances of Mo, MoN and Mo/MoN metallization layers deposited by sputtering Mo in Ar/N<sub>2</sub> atmospheres, respectively.

In order to find appropriate diffusion barrier materials for Cu, the diffusion of Cu into Si through various barrier metals M (M=Cr, Ti, Nb, MO, Ta, W) was investigated by Ono et al [31]. The difference in the barrier properties of the transition metals appeared to be related to the metal-Cu binary phase diagrams and their self-diffusion coefficients. Such a difference in the diffusion barrier effect of the various transition metals might be explained on the basis of the following two physical properties. The first is a difference in the Cu-metal binary phase diagrams. Li et al [32] have reported that reactions of Cu-metal binary systems proceed in various manners with variation of the binary phase diagrams. It is considered that the formation of Cu-metal binary compounds disrupts the multilayer structure, which leads to the silicidation of Cu. The second relevant physical property is the difference in the self-diffusion coefficients of the transition metals. Since increases of resistivity were caused by silicidation of Cu, the diffusion coefficients of Cu and Si through the barrier layer are important. The order of self-diffusion coefficients

in the transition metals at 400-600 °C may be expected as  $Ti > Cr > Nb > Mo > Ta > W$ . However, a precise estimate of the respective barrier effects using self-diffusion coefficients may be difficult because the effects related to their crystal structures and barrier materials easily diffuse to the surface in some cases. Although a qualitative discussion cannot be made here, it is interesting to note that the diffusion barrier effect appeared to be related to the self diffusion coefficient of the barrier materials. At least under relatively low temperature annealing, when the multilayer structure is maintained completely, the self-diffusion coefficient is the most important factor in the barrier effect.

Hence after detailed review we can conclude that we can say that Most of the diffusion barriers fail by grain boundary diffusion. Transition metals fail as barrier at lower temperatures than their nitrides. Transition metal silicides fail due to the reaction of the Si with the Cu. Amorphous barriers offer very high reaction temperature; however, they are tending to crystallization at high temperature and after long time.

## 5. RESULTS AND DISCUSSION:

MoN and WN thin films as diffusion barrier (Si/MoN/Cu and Si/WN/Cu)-Amorphous 50 nm MoN & WN films use a diffusion barrier between the Cu layer and Si substrate to compare the barrier properties. Single crystalline materials are expected to function as the best diffusion barriers because at low temperatures bulk diffusion is extremely small, and there are no grain boundaries for fast diffusion. However, deposition of single crystalline thin films cannot yet be technologically realized. Consequently, the preferred microstructure of thin films for diffusion barrier applications is amorphous as it eliminates the fast diffusion paths along the grain boundaries.

### 5.1 Sheet resistance of Si/MoN/Cu and Si/WN/Cu

The variation of sheet resistance as a function of annealing temperature is shown in figure. The difference of the sheet resistance between the annealed and as deposited samples, divided by the sheet resistance of as-deposited samples, is called the variation percentage of sheet resistance ( $\Delta R_s/R_s$  %) and is defined [33].

The variation of sheet resistance as a function of the annealing temperature is commonly used to examine the capability of diffusion barrier against Cu diffusion. It is well known that Cu diffuses fast in Si form Cu-Si compounds, and the formation of  $Cu_3Si$  compounds results in the increase of sheet resistance of the Cu films. Figure 2 and Figure 3 presents the variation percentage of the sheet resistance versus annealing temperature for the Si/MoN/Cu and Si/WN/Cu. It was observed that the sheet resistance remained constant up to 600 °C, further annealing at 700 °C the dramatic increase in the sheet resistance is observed attributed to the formation of high resistivity  $Cu_3Si$  precipitates and confirmed by the XRD measurement shown in figure .4 and figure.5 The increase in sheet resistance beyond 700 °C may be due to deterioration in the quality of top copper film as inward diffusion of copper has started and results in the formation of copper silicide which have high resistance as compared to copper film. For Si/WN/Cu structure the continuous increase in sheet resistance was observed at higher annealing temperatures. The sheet resistance increases up to annealing temperature 700 °C.

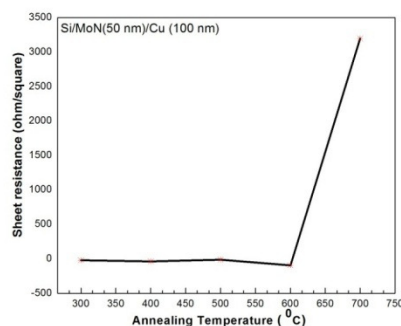


Figure2. Variation of sheet resistance with annealing temperature for Si/MoN/Cu

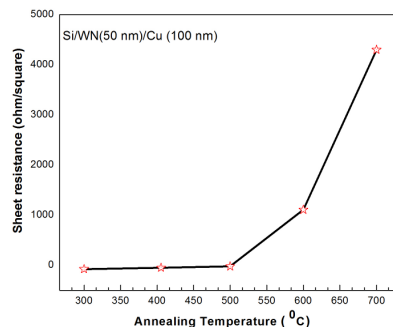


Figure 3. Variation of sheet resistance with annealing temperature for Si/WN/Cu

## 5.2 Phase analysis

The XRD spectrum of the Si/MoN(50 nm)/Cu(100 nm) & Si/WN(50 nm)/Cu(100 nm) structure is shown in the figure 4 and figure 5. In case of as deposited films, two peaks Cu (111) and Cu (200) are visible. Annealing of the samples up to 600 °C have not resulted in any change in the intensities of Cu (111) and Cu (200) peaks. The formation of copper silicide started at temperature 700 °C as evident by the appearance of two small peaks corresponding to  $\text{Cu}_3\text{Si}$  phases. The intensity of  $\text{Cu}_3\text{Si}$  peaks increases was observed. It was indicated that the silicidation start at 700 °C and at that particular point there was barrier failure. Annealing at 700°C indicated complete disappearance of copper 100 peaks. The subsequent annealing at 700 °C resulted in more interactions between copper and silicon as indicated by appearance of new phases of copper silicides.

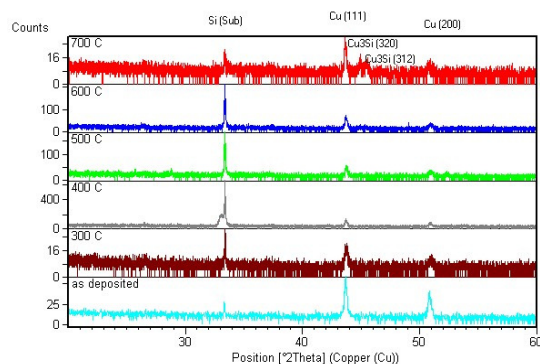


Figure 4. XRD spectrum of the Si/MoN(50 nm)/Cu(100 nm) structure annealed for 30 min at various temperatures

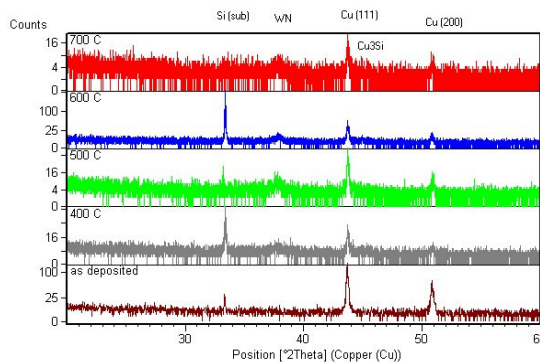


Figure 5. XRD spectrum of the Si/WN (50 nm)/Cu (100 nm) structure annealed for 30 min at various temperature

### 5.3 SEM Results:

It is important to know the microstructure of the thin films as it plays an important role in understanding of physical/chemical changes taking place as a result of annealing. It can be seen that as-deposited copper films on MoN diffusion barrier films is very smooth. It was confirmed by taking images at high magnifications as well. Up to 600 °C, there was no morphological change observed in the Si/MoN/Cu system. But a change in surface micrograph of the top copper layer is seen in the SEM images recorded after annealing at temperature 700°C. For Si/MoN/Cu system, the rupture of Cu films appeared at 700 °C, on the other hand, Si/WN/Cu system survive only up to 500°C.

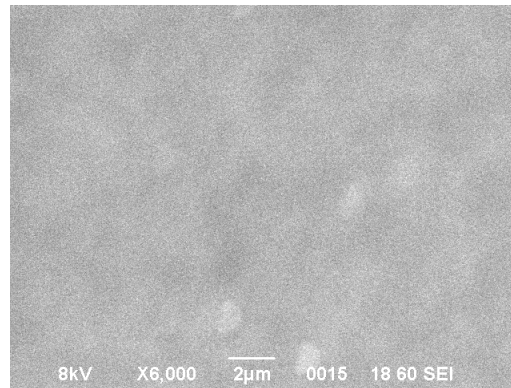


Figure 6 SEM image of Si/MoN/Cu as deposited structure

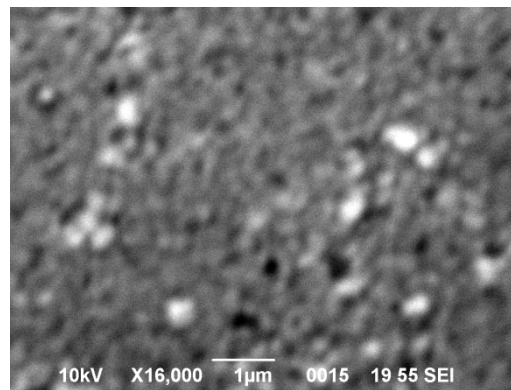


Figure7. SEM surface morphology of Si/MoN/Cu structure, 600 °C annealed samples for 30 min.

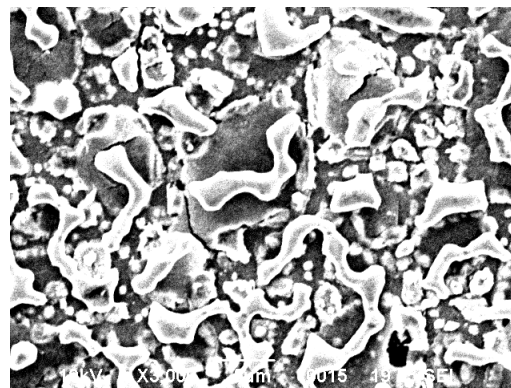


Figure 8. SEM surface morphology of Si/MoN/Cu structure, 700 °C annealed samples for 30 min  
deteriorated film clearly observed

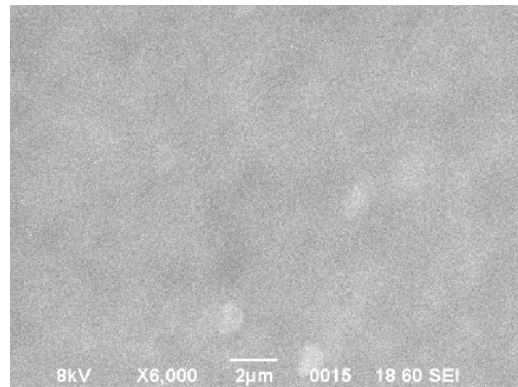


Figure 9. SEM image of Si/WN/Cu as deposited structure

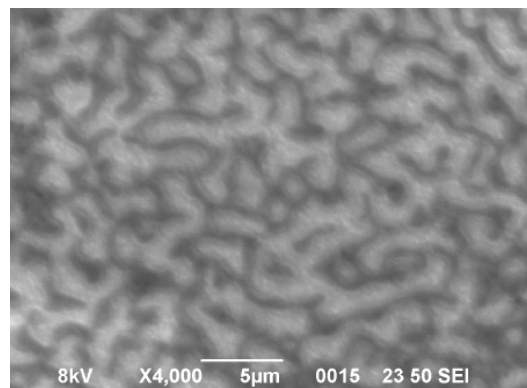


Figure.10 SEM surface morphology of Si/WN/Cu structure, 500 °C annealed samples for 30 min.

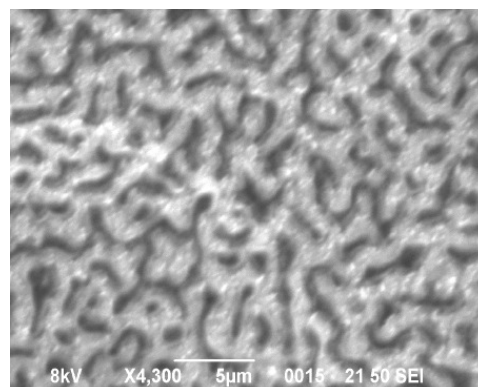


Figure 11. SEM surface morphology of Si/WN/Cu structure, 700 °C annealed samples for 30 min.

The SEM study shows that as a result of annealing above 700 °C, the blackish region (Cu denuded zone) i.e. depleted of surface copper atoms increases, therefore, increased sheet resistance at higher temperature as observed in electrical measurement are in agreement with each other.

From SEM images it appear that at 600 °C, small pinholes or cracks are developed due to rupture of the top copper layer most likely due to increased thermal stress in the barrier/copper interface or bulk of the films. These pinholes may act as pinning source of interface interaction and inter-diffusion of metals. It has been observed that when Si/WN/Cu structure annealed at 600 °C, a large number of whitish grains

were initiated at these pinholes. A large number of bright particles have been observed. The SEM study shows that as a result of annealing above 600 °C the blackish region (Cu denuded zone) i.e depleted of surface copper atoms increases therefore increased sheet resistance at higher temperature as observed in electrical measurement are in agreement with each other.

## **6. CONCLUSION**

The deposition of MoN & WN thin films for diffusion barrier applications by DC magnetron reactive sputtering technique has been carried out successfully. The presence of MoN & WN peaks in the XRD spectrum indicates the successful formation of nitride phase. The thermal stability of barrier thin films against Cu diffusion was determined. The thermal stability was evaluated by X-ray diffractometer (XRD), four probe method and SEM techniques. It was observed that the MoN acts as a good diffusion barrier up to 600 °C on the other hand WN was stable up to 500 °C. The annealing at higher temperature resulted in appearance of various copper silicide phases. It was confirmed that the degree of amorphization increases with increasing Nitrogen content. Sputtered deposited MoN thin films have been found to be suitable for copper metallization and are stable up to the 600 °C as compared to WN thin films.

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