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Deposition and Characterization of Tungsten Nitride Thin Film

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ABSTRACT: In this paper Tungsten Nitride Film is deposited by reactive sputtering using DC magnetron sputtering system.The deposition parameters are optimized for the deposition of tungsten thin film.The effect of pressure on the deposition rate was studied.On the basis of obtained data optimized parameters are selected.After that we tried to deposit Tunsten Nitride film using reactive sputtering,for this purpose Nitrogen gas was supplied to the chamber along with the Argon gas.After that we have deposited samples at same thickness as a function of Nitrogen flow (Argon flow was constant) and studied the sheet resistance as a function of Nitrogen flow.

1. INTRODUCTION

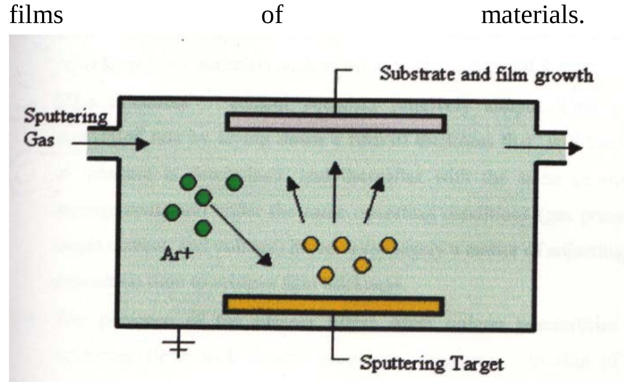
The reason for selecting Tungsten as a target material is its application that are obtained due to its important properties such as low coefficient of thermal expansion,good thermal and electrical conductivity,high melting point and most important property that it can withstand high temperature above 1100 C.A variety of thin film deposition techniques are employed to deposit Tungsten Nitride films such as thermal deposition ,evaporation,chemical vapour deposition,metal organic chemical vapour deposition,sputtering etc.Since electrical and optical properties of film depend srongly on their microstructure,stioctiometry and nature of impurities present,each deposition technique with its associated parametre yields films of different properties of the extensively used techniques are explained in following sections.

1.1 SSPUTTERING

Sputter deposition process is an old technique being used in modern semiconductor industries.Sputtering is a process that can deposit any material on any sustrate.The target is connected to negative high voltage,Argon gas is introduced into the chamber and is ionized to a positive charge.The positively charged Argon atoms are attracted and strike the negatively biased target as shown in fig. 1.1.The Argon ions “knock off” atoms and molecules from the target into the chamber.The sputtered atoms and molecules scatter in the chamber and out of which some comes into rest on the wafer.A principle feature of a sputtering process is that the target material is deposited on the wafer without chemical or compositional change.

The benefits of Sputter deposition technique over other deposition technique are as follows:

- SSputter deposition technique is useful for deposition of complicated materials such as stainless steel without change of composition provided target temperature is kept low.
- SSputtering can be accomplished from large area targets.This often simplifies the problem of film thickness uniformity and shadowing effect also becomes less pronounced.
- Cleaning of substrates is much simplified because the surface of substrates can be sputter cleaned by ion bombardment before sputter deposition.
- TThrough DC or RF sputtering of metals or semiconductors in reactive gases as oxygen and nitrogen,it is possible to form oxidized on Nitride films of materials.



1.2 FACTORS INFLUENCING THE DEPOSITION RATES

1. Current and Voltage: In the energy range employed for the deposition of the most films the sputtering yields increases relatively slowly with increasing ion energy or applied voltage on the other hand the number of particles striking the cathode is proportional to the current **d**ensity.Consequently,current is a significantly more

important parameter for determining the deposition rate rather than voltage.

2. Pressure: As a pressure in a sputtering is raised, the ion density and therefore,the sputtering current density increases linearly with pressure provided the latter is not so high.At higher pressure deposition rate becomes less.The reason for reduction in deposition is that material sputtered from the target may collide with gas atoms on its way to the substrate,at a rate,which will increase with increasing pressure.The result of the collision is to defeclect the sputtered atom,backward and hence deposition rate is decreased.The optimum pressure to use for sputtering may be lower than 130mtorr.This is because of the problem of backscattering and reduced size of crookes dark space at higher pressures makes effective shielding of the cathode increasingly more difficult.

3.Ground shield:In sputtering systems the target is surrounded by dark space shield,also known as ground shield.The purpose of this is to restrict ionbombardment and sputtering to the targets only.

THIN FILM

Thin films are thin material layers ranging from fractions of a nanometre to several micrometres and the act of applying a thin film to a surface is known as thin filn deposition.

- **MECHANICAL PROPERTIES OF THIN FILM**
  a) Adhesion: The Adhesion of deposited film has been of interest for a considerable time.The durability of coating is of prime omportance in many fields and one of the main factors that govern this durability is adhesion.
  b) Stress: It may be compressive so that in extreme cases it may buckle up on the substrate.alternatively it may be tensile and certain cases the force may high enough to exceed elastic limit of the film in order to breaks up.

- **ELECTRICAL PROPERTIES OF THIN FILM**
  Source of resistivity in metallic conductors : According to modern quantum electronic theory,electrical conduction in metals is due to electrons while electrical resistivity resuits from the scattering of these electrons by the lattice.
- Dielectric properties of thin film
  The electrical properties of thin insulators are employed in the fabrication of devices.These films are used in a wide variety of components and the films are usually amorphous or near amorphous.
- Sputtering material
  a)Tungsten: It has the highest melting point of all metals and low thermal expansion coefficient and has highest tensile strength. It does not oxidize in air and need no protection from oxidation at elevated temperatures.

Application of tungsten thin film :

1. Tungsten is used in microelectronics as a contact material for conductive layer and barrier layer between silicon and other metal.
2. Tungsten metal has been the most widely used diffusion barrier material for contact in integrated circuits .
3. This film has been proposed for the application of metallization and in nanotube preparation
4. Tungsten film can also be used in electrode for thin film capactor and field effect transistor.
5. Tungsten film are appropriate for ULSI Cu interconnections using electroless cu deposition .
6. In microelectronics,the dominant application of tungsten has been as a via metarial for multilevel metallization.
7. Its high melting point also makes tungsten suitable for areospace .
8. Tungsten is used in microchip technology and liquid crystal displays.and also used in jewellery because of its longevity and high durability.

b)Tungsten Nitride:it is an inorganic compound ; a nitride of tungesten. It is a ceramic material and is electrically conductive.it is very hard solid material and brown colour.

Property of tungesten nitride :

1) Tungsten and tungsten nitride thin film deposited by supttering system shows high resistivity properties and these high resistivity property were measured experimentally.
2) Tungsten nitride thin films grown by reactive DC sputtering shows mechanical properities like adhesion.the adhesion of the film was measured in dependence on two principal parameters: - the nitrogen partial pressure in the magetron discharge gas mixture of nitrogen and argon and the substrate temprature.
3) It exhibit good heat resistance ,refractroriness and wear resistance is potentially suitable for a widw range of high temprature aaplications.

4) Atomic layer deposition of tungsten nitride film usin sequential surface reactions.atomic layer deposition uses seqential surface reaction to achieve film growth in monolayer or submonolayer thickness.

Application of tungsten nitride thin film device :

1) Optoelectronic applications:-it is based on photo electronic effects which arries due to direct interation of the incident photons with electrons in material.The most widely used photo effect are those which involve conversion of incident optical energy to electrical energy.These are (a) Photoconductive (b)Photoelctric (c) Photovotaic.

EXPERIMENTAL WORK

The procedure for sample preparation and characterization includes following steps:

a) Cleaning of substrate
b) Deposition of thin films using sputtering
   After sample preparation,yhe characterization was carried out using following techniques.
   a) Thickness of the film using surface profiler
   b) Resistivity measurement using four probe.
   c) Phase identification using XRD.

PROCEDURE FOR CLEANING SAMPLES

The steps used for cleaning the samples are following

1) Take the samples of silicon wafer or glass slide.
2) Clean the silicon samples properly, load the wafers in beaker using non-PR tweezers and dip in DI water to remove any impurities deposited on the surface of the wafer.DI water is made by purification followed by reverse osmosis and deionization.
3) For removing grease and oil film boil the samples in TCE for 15 minutes. Then ultrasonic degreasing in acetone for 5 minutes.Rinse thoroughly in DI water 4 to 5 times.Spin dey the wafer.

PRECAUTIONS

The glass beakers kept on the cleaning bench should not be mixed or mixed with those meant for PR related work.

For thoroughly rinsing in DI water use atleast 5 changes baths with sufficient agitation in water.

PROCEDURE FOR DEPOSITION OF THIN FILM

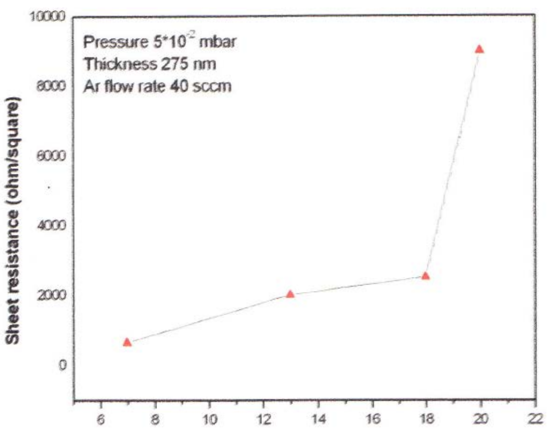
THE EXPERIMENTAL set up for deposition of tungsten nitride is shown in fig.The silicon wafer acts as the anode and tungsten target acts as the cathode.The gases used are argon and nitrogen.Once the sample was cleaned it was attached to substrate holder of sputtering system.In sputtering system vacuum of about $3*10^{-5}$ mbar was created by using rotary and turbo pumps.After creating vacuum,gate valve was closed and gases were supplied and flow rates of argon and nitrogen as well as pressure was adjusted.Once adjusted power was supplied for deposition time.

OBSERVATION AND RESULTS

DIFFERENT SAMPLES OF TUNSTEN NITRIDE WERE DEPOSITED for constant pressure and argon flow and varying nitrogen flow rate.The samples were deposited for different deposition times(2min,5min,7min).The calculated average deposition rates were shown in table 1.On the basis of these deposited samples we have drawn a graph between average deposition rate as the function of nitrogen flow rate and observed that average deposition rate decreases with increase in nitrogen flow rate as shown in fig.

Table-1 Variation in Average deposition rate with N_2 flow rate

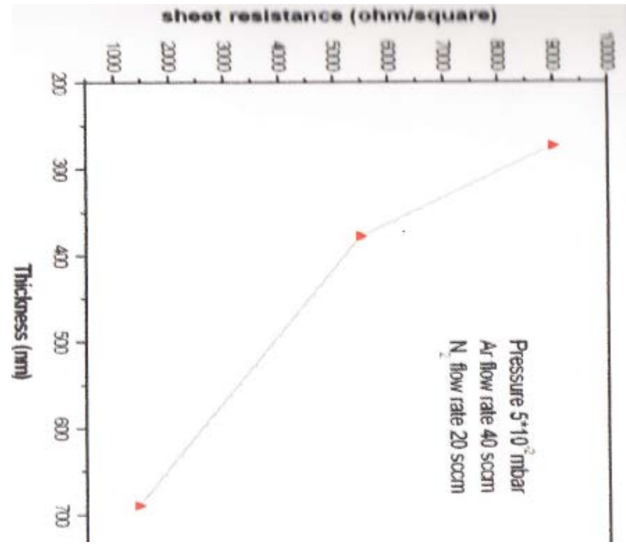
Nitrogen flow rate(sccm)	Deposition rate (nm/min) DT(2min)	Deposition rate (nm/min) DT(5min	Deposition rate (nm/min) DT(7min	Average depositon rate(nm/min)
10	170.5	104	94.57	123.02
13	140	128.6	117	128.53
15	137.5	126.2	115	125.73
18	134	140	129.85	134.61
20	130	98.57	75.8	103.95



The samples of same thickness(275 nm) of tungsten nitride films were deposited for different nitrogen flow rate.The pressure 5*10-2 mbar and argon flow rate was maintained constant.the obtained data is summarized in table2. On the basis of above data we have plotted a graph of sheet resistance as the function of nitrogen flow and observed that sheet resistance increases with increase in nitrogen flow as shown in fig.\

Table-2 Variation in Sheet resistance with N_2 flow rate

SNO.	Nitrogen flow(sccm)	Sheet Resistance(Ω/sq.)
1	7	663.03
2	13	2012
3	18	2521
4	20	9003



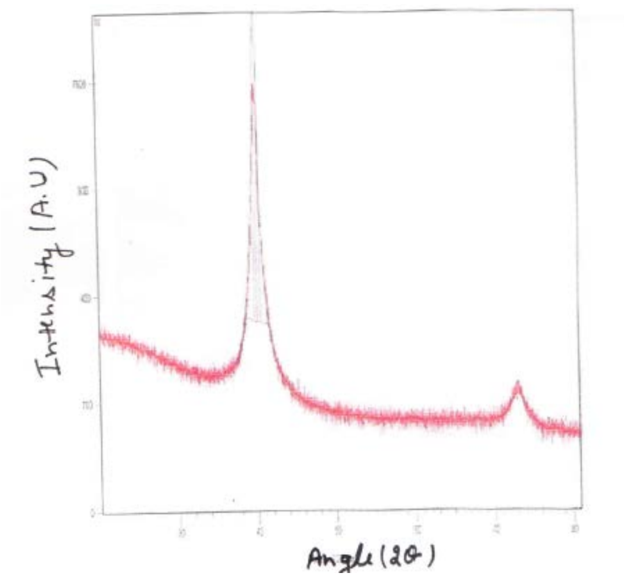
Different samples of tunsten nitride were deposited for constant argon gas flow 40sccm and pressure 5*10-2 mbar and by varying time,which are minimized in table 3.On the basis of these deposited samples we have drawn a graph of sheet resistance as the function of thickness and observed that sheet resistance decreases with increase in thickness as shown in figure.

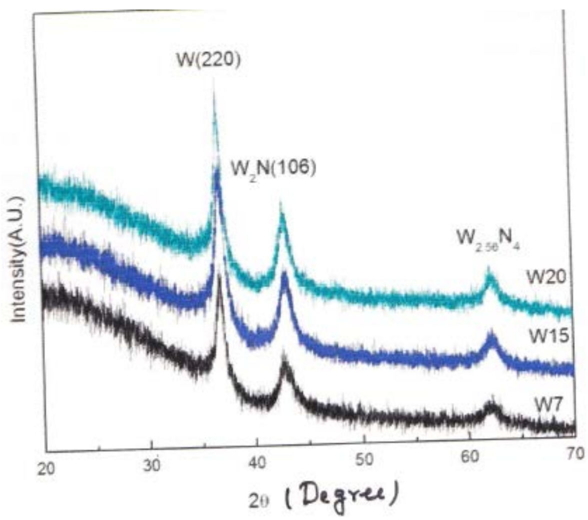
Table-3 Variation in Sheet resistance thickness

Sno.	Deposition time(minute)	Thickness(nm)	Sheet Resistance(Ω/sq.)
1	2	275	9003.27
2	5	379	5501.39
3	7	690	1456.72

XRD RESULTS

The XRD result of as deposited W thin film is given in fig,200 nm thin film is deposited at the pressure of 5*10-2 mbar with the Ar flow rate of 40sccm.The W peak observed at the 2Θ = 40.15 ° With the d spacingof crystal 2.243 A. The XRD result of tungsten nitride thin film is given in fig. W_2N thin film is deposited at the various nitrogen flow rate at 7,15,20 sccm at the constant pressure of 5*10-2 mbar.The XRD spectrum indicates the presence of peaks corresponding to tungsten nitride phases; W(220) at 2Θ = 36.9 , W_2N(106) at 2Θ =43 and the $W_{2.56}N_4$ at the angle of 2Θ =62.2





CONCLUSION

We have studied DC sputtering technique for the deposition of tungsten nitride thin film. The presence of WN peaks in the XRD spectrum indicate the useful formation of nitride phase. The variation of sheet resistance as a function of nitrogen flow rate was also studied. It was observed that sheet resistance of the WN films increases with increase in nitrogen flow rate. The deposition rate of the deposited films decreases with increase in flow rate on nitrogen. The sheet resistance decreases with increase in thickness of tungsten nitride.

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