Scanning Magnetron Sputtered Tin Coatings as Diffusion Barrier for Silicon Devices

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Abstract: ***Scanning magnetron target (274mmx174mm) is used to deposit TiN film on to Si (<100> p type 10 ohm cm resistivity) substrate for microelectronic application. To deposit best quality TiN films with low resistivity[1], parameters. Such as deposition rate, scanning speed, target substrate distance (T.S.D.) and ion current ma, Nitrogen partial pressure, were optimized. Total nitrogen and argon pressure was kept at $5x10^{-3}$ m bar. TiN films were sputter deposited on to Si with different substrate bias. Cu film is sputter deposited on to as deposited TiN films since it is found as best substitute for diffusion barrier. Thickness of the TiN film is determined keeping in consideration diffusion length[2] of Cu in TiN at different temperature. Stylus Dektak is used to obtain desired thickness of TiN and copper films. Cu /TiN /Si Structure thus formed were annealed at different temperature to study microelectronic application for diffusion barrier. Resistivity fig7of the Cu/TiN /Si structure were obtained using four probe method, low resistivity Cu/TiN/Si structure were preferred. Inter diffusion of silicon and or top metal through the TiN film was investigated. XRD fig 8 SEM fig 9, 10, 11 EDAX fig 12 of Cu/TiN /Si is obtained to determine its feasibility for microelectronic application.***

Keywords: diffusion resistivity

1. Introduction

TiN is used as a diffusion barrier in advanced integrated circuit devices due to its chemical stability and low resistivity. Ideal diffusion barrier for a contact metallization is defined as a chemically and mechanically stable and electrically conductive layer that inhibits inter diffusion between its bordering media TiN has been reported as diffusion barrier material for silicon devices which acts as an impermeable barrier to silicon atoms [1].

The diffusion length of Cu in TiN at temperature between 600 and 700 ^{0}C has been repoted about 400 $A^{0,}$ the diffusion length [2]of Cu in TiN at 800^{0}C was observed 800$A^{0.}$

There has been considerable interest in TiN as diffusion barrier in multilevel metallization schemes for integrated circuits[3, 4-7] in Al/TiN /Ti/Si system it has been shown that TiN films as thin as 1000A^{0} thick can prevent the silicon aluminum inter diffusion at 600^{0} c for 30 min, as observed by RBS. But it was found that that the electrical devices fabricated using these metallization schemes failed during a much lower thermal annealing cycle[5, 8-11]

Suni et al[12] found that there were two failure mechanism for the breakdown of TiN diffusion barriers. First fracture of barrier layer caused by very large compressive stress in the film due to negative bias applied to the substrate, second inter diffusion of the contact metal and silicon through defects such as pinholes and voids. In TiN /Ti/ Thick SiO_2 [13]structure peeling was found creating problem.

With the continuing progress of integrated circuits technology the requirements to increase the device density necessitates the improvements of multilevel metallization. Copper (Cu) has been extensively studied as a potential substitute for Al and Al alloys in multilevel metallization of semiconductor devices and integrated circuits[14-15]. Cu metallization for multilevel inter connects has advantages such as high melting point[15-16], high electro migration resistance[17], low bulk resistivity, low reaction tendency with commonly used diffusion barrier materials[14, 16-18].

Experimental procedure: The present system is a sophistication of a D.C. sputtering unit having a combination of 300 lit /sec diffusion pump with a 200lit /min rotary vacuum pump the system gives an ultimate vacuum of $5x10^{-6}$ milli bar. The pressure monitoring is done by using pirani and penning gauge combination. The vacuum chamber is a 300mm diameter S.S cylinder opened at both ends. The target (274mm × 174mm) is mounted on the cathode plate which is scanning magnetron with optimized speed and deposition is carried out in sputter down mode. The substrate holder with silicon substrate (<100> p type 10 ohm cm receptivity) fixed to the base plate Iolar- 2 grade argon (99.999%) has been used as sputtering gas flow of which is controlled by combination of two needle valve in series. A high voltage type, with15 Kv and 10A rating was used. TiN films were deposited on to Si in reactive environment nitrogen argon is taken as sputtering gas. Parameters such as deposition rate745A^{0}, scanning speed14cm/sec, target substrate distance (T.S.D.) 11cm and ion current 200ma, Nitrogen partial pressure at $3x10^{-5}$m bar, total nitrogen and argon pressure at $5x10^{-3}$ m bar were optimized.

Stylus dektak is used to determine the thickness of thin film. 800 A^{0} thick TiN film is sputter deposited on to Si then 600 A^{O} thick Cu film is sputter deposited on to as deposited TiN film. CU/TiN /Si structure thus formed were annealed for 1 h at 650, 750 800 and 900 ^{0}c in the vacuum at $3x10^{-5}$ m bar.

Four probe methods are used to determine the resistivity at different temperature. Philips Analytical X-Ray diffrectometre (pw 3710) using Xpert software was used to study X.R.D of the prepared samples at different temperature. Jeol scanning Microscope (JSM-840) was used to study SEM photograph of the prepared samples at

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different temperature. Chemical analysis is done by X-ray analysis EDAX method of the surface layer.

Preparation of the TiN films: In the present study TiN film have been deposited on to silicon substrate to study their feasibility for microelectronic application, Using Scanning magnetron, titanium target and Ar as sputtering gas N_2 as reactive gas TiN films were deposited onto silicon (<100>, p-type, 1-10 ohm resistitivity) substrate under varying N_2/Ar pressures, varying currents, varying target substrate distances (T.S.D.) varying bias conditions and varying annealing temperatures N_2 Pressure for given sputtering ion current 200 MA at 11 cm T.S.D.was determined empirically by visualizing the golden color of the deposited films. The gold like appearance of the film was presumed to be TiN according to other reports[19-22]Stoichiometric TiN film is obtained only within a narrow range of N_2 partial pressure, N deficient films are obtained below the optimal N_2 partial pressure and N rich films at excess N_2 partial pressure[22 -23].The TSD was kept quite large (11cm) to achieve homogeneous composition of the deposited films by scanning magnetron sputtering while the ultimate pressure was achieved $5X10^{-6}$ mbar good golden color TiN films were obtained at nitrogen pressure$3x10^{-5}$mbar for sputtering ion currents 180 and 200 ma. The effect of ultimate vacuum on mechanical properties of sputtered coatings is significant. In the coatings deposited by sputtering techniques the contamination of the deposition atmosphere can influence the properties owing to different phenomena which can arise during substrate cleaning and for coating growth [24-26] .Total nitrogen and argon pressure was kept at $5x10^{-3}$ mbar.

Stresses are low at $5x10^{-3}$ mbar Ar pressure [27] below this Ar pressure compressive stresses increase with decreasing pressure almost linearly and at higher pressure this turns to be tensile stress around $6x10^{-3}$ mbar. As Ar pressure is increased further the internal stress seems to decrease only slightly becoming essentially Zero [27] .Presently it was observed that the films, deposited at lower than $5x10^{-3}$ mbar Ar pressure, Peel out after few hours which indicated high stresses in the films deposited at lower pressures. The crystal structure of TiN film is also affected by total gas pressure- [28] .Films were deposited at different substrate biasing voltages viz...o, -20, -10, -60-80, -100, and -120 v.

X.R.D. Study of the Deposited TiN Films: **Figure 1, 2**

Philips Analytical X-Ray diffrectometre (pw 3710) using Xpert software was used to study X.R.D of the prepared samples. The observed XRD patterns of the golden TiN films deposited at different substrate bias are given in figure 1and 2.The scanning magnetron sputtered TiN films deposited at 180 ma ion current shows amorphous structure fig.1 and films deposited at 200ma ion current show crystalline structure with a single (220) face orientation figure 2, which indicates the strong dependence of the film structure on the ion current.

X-ray diffraction patterns of the TiN films deposited by Arc ion plating show strong dependence on bias voltage. At zero volt biasing a strong (220) peak, at -80 and -100 V strong (111) peaks and at -30v random orientation of film have been observed by Matsue[16] et al. The preferential orientations is controlled by certain growth conditions, such as bias voltage of the substrate[17]and the arrival ratio[18-19] of the number of sputtered titanium and nitrogen atoms.Xiao[20] et al have observed that the films deposited by R.F. magnetron sputtering at the deposition rate $480A^0$ /Min show preferred orientation of (200).Where as those deposited by the arc plating at the deposition rate $3000A^0$ indicated strong (111) preferred orientation, this indicates the film orientation depends on the deposition rate.

TiN films grows along the (100) plane in R.F. sputtering because the atoms on the substrate has more time to migrate due to low deposition rates.

Assuming the total strain energy of the TiN film pellag[21] et al have proposed a model of correlation between surface energy (Shkl) and strain energy (Uhkl) of TiN film, to explain the mechanism resulting in preferred orientation. They showed that both surface energy and strain energy have directional properties and correlation of surface energy and strain energy are described as Whkl =Shkl +Uhkl were Whkl represents the total free energy of the system, which should minimal in thermodynamic equilibrium. A preferred (100) orientation can be considered as one for which the surface energy is dominant over the strain energy on the contrary (111) orientation should be preferred when the strain energy is large and dominant. The X.R.D. pattern patterns of the present crystalline films show (220) orientation indicating the equal dominance of surface and strain energies for the scanning magnetron sputtering deposited TiN films.

Grain Size and Stress: Fig-3

The stress in the film and grain size is calculated from the XRD data. For polycrystalline film with isotropic biaxial stress, the stress T is given by [22]

$$T=E\,(a-a_0)/\,2va_0$$

Where E is the young' s modulus (590 KN/mm^{-2}); v the Poisson ratio (o.3) and a_0 the lattice constant ($4.240A^0$) of the face centered cubic TiN a is the measured lattice constant. From the X.R.D. patterns, Figure 2 the lattice constant is obtained 4.249967 A^0 for the films prepared at room temperature without any bias voltage the lattice constant reduces to $4.248297A^0$ for the film prepared at room temperature with -40 bias voltage. The change in the lattice constant can be attributed to the stress in thin films.

Stress in the film can be due to the presence of oxygen and also in the low pressure sputtering regime, ion bombardment during film growth results in compressive stress due to atomic peening mechanism. Calculated from these figure 3, shows the variation of the stress with

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substrate biasing voltage. The films deposited at room temperature and with biasing voltage up to -40 volt show compressive stress.

Films deposited at higher substrate biasing show transformation to tensile mode intrinsic stress and resistivity of TiN films depends on various deposition parameters and on substrate position. They appeared mainly to be controlled by the effect of oblique incidence of sputtered particles and by the bombardment of energetic species on growing films[23].

Also the nitrogen partial pressure and the argon partial pressure were found influencing the internal stress crystallographic structure and resistivity. The internal stress of the deposited TiN Films changes as a function of both argon and nitrogen partial pressure and films deposited at low argon pressure tends to have high compressive stress. The internal stress is also related to preferred orientation of the film. The internal stress of the TiN films depends on its microstructure. Films deposited with (111) preferred orientation have smaller internal stress then that for the films with (100) preferential orientation[24, 25]. The grain size (t)of the deposited crystalline TiN films was determined from the X ray line broadening Using the scherrer's formula[26] $t = 0.9\ \lambda / B\cos Ø$

Where λ is the wave length of x ray used, B the full width at half maximum corresponding to 2 ə. The grain size was observed changing from 371.7 A^0 to 385 A^0 as the substrate biasing voltage changes from zero to -40 volt grain size decreases with further increase in the negative biasing voltage.

Dependence of resistivity on annealing temperature: Figure 4, 5

Variation of the observed resistivity of the scanning magnetron sputtering deposited TiN films with annealing temperature has been shown in figs. 4 and 5. It has been observed that resistivity decreases with increasing annealing temperature, showing a minimum at 750^0c, for all the samples. For the amorphous TiN film, deposited with bias voltage -80 volt resistivity was observed decreasing from 737 μΩcm (room temperature value) to 304 μΩ-cm (at 750^0c). Also for the samples deposited with bias voltage -40 v resistivity of the crystalline samples decreases from 320 μΩcm to 132μΩcm (at 750 ^{0} c)for temperatures around 800^0c the resistivity increases to a maximum value and then decreases at higher temperature, for all the prepared TiN thin films samples. The decrease of resistivity with increasing annealing temperature may be due formation of oxide layer (due to oxygen migration across the film) and decrease of the resistivity above 800^0c may be due to higher grain size[40].

SEM results: figure 6 (a), (b)

Jeol scanning Microscope (JSM-840) was used for SEM study of the prepared samples.SEM results of the surface morphology of the crystalline TiN films deposited with -40V substrate bias and annealed at different temperatures are shown in Fig.6 (a) (b) Surfaces of the films were observed smooth. flat and compact and there was no sign of holes or degradation of TiN Surface up to 900 ^{o}c in the present study, . Poitovin et al[41] have observed many failures in the TiN structure, after annealing at 1000 ^{o}c they observed some big holes (around 10 micro m. in diameter) and many little holes (around 1micro m. in diameter)

Resistivity of Cu/TiN/ Si Structure: Figure 7

Annealing of Cu/TiN/ Si structure did not change color of the copper films up to 750 ^{0}c.However color of the film annealed at 800 and 900 ^{0} c was found changed. Showing formation of new materials, the variation of the resistivity of the copper films with annealing temperature is shown in figure 7 as observed from figure 7 the resistivity did not significantly change up to 750^0c. However a small decrease in resistivity with increasing annealing temperature was observed, which can be attributed to the change in grain size and reducing defects. But this decrease in resistivity of cu is not as high as Deenamma et al observed [38]. Since in the present study Cu was deposited on the as deposited TiN films, the grain size of underlying TiN layer may also be increasing causing the stress in copper films. The fact that the copper resistivity did not change significantly for the samples annealed up to 750^0c indicating that there is no inter diffusion of Cu and /or Si through TiN films up to 750 ^{0}c. But at 800 ^{0}c resistivity was found increasing indicating the starting of diffusion beyond 800^0c, resistivity was found anomalously increasing showing significantly rise in diffusion.

XRD of CU /TIN/SI Structure: Figure 8

Figure 8 shows that X.R.D. patterns of the CU/TiN /Si samples annealed at different temperatures. The increase in the intensity of the copper lines with increasing annealing temperature is due to the larger grain size and better orientation resulting from annealing. Also there are no extra peaks corresponding to any compound of Si, Ti and Cu up to 750 ^{0}c. These results confirms the absence of any inter diffusion of Cu and Ti through TiN film. For the samples annealed at 800 and 900 ^{0}c, some extra peaks of Cu and Si through TiN barrier are observed. The resistivity fig 7 is supported by X-RAY diffraction results.

The resistivity and XRD results suggest that up to 750^0 c the scanning magnetron TiN film is a good diffusion barrier for silicon devices.

SEM results: Figure 9, 10, 11

The inter - diffusion of the border media across the TiN film was studied by SEM. The SEM photographs of Cu/TiN/Si structure, annealed at different temperatures, are shown in fig 9 - 11. Room temperature deposited samples show flat and smooth surface, and clusters of Cu grains were seen due to grain growth in the samples annealed at higher temperatures. Up to 750 ^{0}C. annealing temperature no inter-diffusion through the TiN film was

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observed. fig. 9. No holes were observed in the samples annealed up to 750 ^{0}C. Also. SEM photographs of the cross section of Cu/TiN/Si structure, annealed at 650 ^{0}C, show three different layers and no inter-diffusion, fig. 10 (a). In the samples, annealed at 800 ^{0}C, some traces of diffusion were observed on the surface. Holes of different diameters were observed in the SEM photographs of the samples annealed at 800^{0}C, fig. 11 (a) and, significantly high diffusion was observed in the SEM results of the samples annealed at 900 ^{0}C, surfaces show islands of different structures, fig. 11 (b). Also SEM of the cross section of Cu/TiN/Si structure shows breaking of the TiN layer and inter-diffusion of Cu and Si, in the samples annealed at 900 ^{0}C, fig. 10 (b)

Chemical Analysis: **Figure 12**

Chemical analysis by energy dispersive X -ray analysis EDAX methods of the surface layer of the annealed samples was carried out using jeol scanning microscope (J.S.M.840) to observe any change in surface chemistry due to annealing. The observe results are given in figure 12.

Sample annealed up to 750 ^{0}c show only copper peaks indicating no inter diffusion of copper and silicon through the TiN barrier. Chemical analysis of the holes observed in the SEM of the sample annealed at 800 ^{0}c give the Si and Ti peaks with Cu peaks.

Indicating the inter diffusion of Cu and Si through TiN layer. Chemical analysis of the islands observed in the SEM results of the samples annealed at 900 ^{0}c show Si, Cu and Ti peaks indicating high inter diffusion of Cu and /or Si through the barrier.

Resistivity XRD, SEM and EDAX results are consistent with each other for the scanning magnetron sputtered TiN samples investigated as diffusion barrier for silicon devices. The diffusion length [2]of Cu in TiN at temperature 600 and 700 ^{0} c has been reported about 400 A ^{o}. The thickness of the TiN films used in the present study was 800 A ^{0} which becomes comparable to the diffusion length of cu in TiN at 800 ^{0} C. Deenamma et al have reported diffusion through TiN films at 750 ^{0}c which may be due to smaller thickness and high stresses in their samples. Films deposited in the present study show lower stresses than those reported [38]earlier.

2. Results

Results: TiN films were deposited under varying currents varying target substrate distance (TSD) varying bias conditions and varying annealing temperatures. Good golden color film was obtained at nitrogen partial pressure of 3x10^{-5} mbar ion currents were 180 and 200 ma. Total argon and nitrogen pressure was kept at 5x10^{-3}m bar.

XRD study of the deposited TiN films reveals that TiN films deposited at 180 ma ion current have amorphous film structure and film deposited at 200 ma ion current exhibits crystalline structure. At 200 ma ion current film is crystalline having (220) face orientation, which indicates strong dependence of the film structure on the ion current.

The grain size with zero volt substrate bias is observed 371.7 A^0 which increases and attains shape of 374.7 A^0 around -35 volt which decreases further and grain size is 200 A^0 at -80 volt substrate bias.

Stress was found 2.4 G..p.a. at 0 volt substrate bias which decreases sharply after -35 volt and attains minimum at -52volt which is 1.3 G.P.a

From the observation it is clear that stress decreases with grain size sharply.

It has been observed that resistivity decreases with increasing annealing temperature showing a minimum at 750^{0}c for all samples. For the samples deposited with bias voltage -40 volt resistivity of the crystalline samples decreases from 320 micro ohm (at room temperature) to 132 micro ohm cm (at 750^{0}c), for temperature around 800 ^{0}c the resistivity increases to a maximum value and then decreases at higher temperatures.

SEM analysis reveals that crystalline film surface were smooth flat and compact and there were no sign of degradation up to 900^{0}c, in the present study.

(1) In figure 7

At 750^{0}c with substrate bias -40 volt, resistivity was found to decrease which is abut6X 10^{-4} Ω cm which occurs due to increase in grain size and reducing defects. But at 800^{0}c resistivity was found increasing indicating the starting of diffusion.

(2) In figure 8

XRD reveals that there were no extra peaks corresponding to any compound of Si, Ti and Cu up 750^{0}c. For the samples annealed at 800^{0}c and 900^{0}c, some extra peaks of Cu and Si compounds were observed indicating inter diffusion of Cu and / or Si through the TiN barrier. The resistivity measurements are supported by X ray diffraction results.

(3) In figure 9, 10, 11,

Up to 750^0 c annealing temperature no inter diffusion through the TiN films was observed, no holes were observed in the samples annealed up to 750^{0}c. Also SEM photographs of the cross section of Cu/TiN /Si structure annealed at 650^{0}c show three different layers and no inter diffusion.

(4) In figure 12

Up to 750^{0}c there was no indication of inter diffusion only copper peaks are observable. EDAX of the TiN films used in the present study was 800^{0}c give the Si and Ti peaks with copper peaks indicating the inter diffusion of Cu and Si through TiN layer.

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The thickness of the film used in present study was 800 A^0 which becomes comparable to the diffusion length of Cu in TiN at 800^0c. The diffusion length of Cu in TiN at temperature 600 and 700 ^{0} c has been reported[2] about 400 A ^{o}

All the results and discussion reveals that TiN film act as good diffusion barrier up to temperature 750^0c.

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Figure File

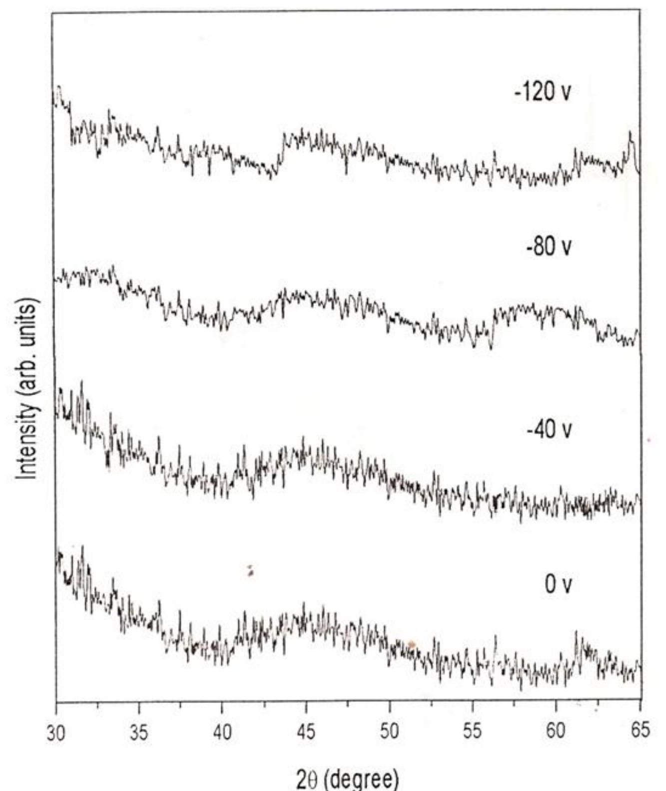

Figure 1: XRD patterns for TiN films deposited at different substrate biasing (ion current was kept 180 ma)

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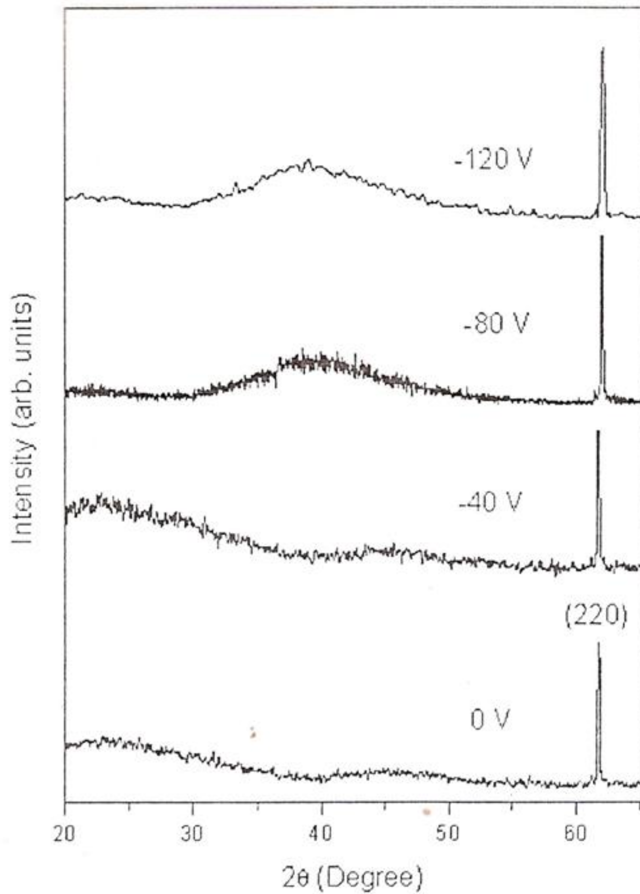


Figure 2: XRD pattern for TiN films deposited at different substrate biasing (ion current was kept 200ma)

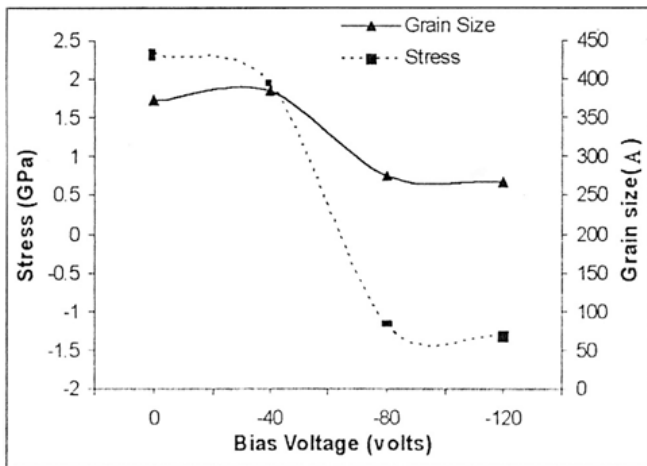


Figure 3: Variation of stress and grain size of the crystalline TiN films with substrate bias voltage

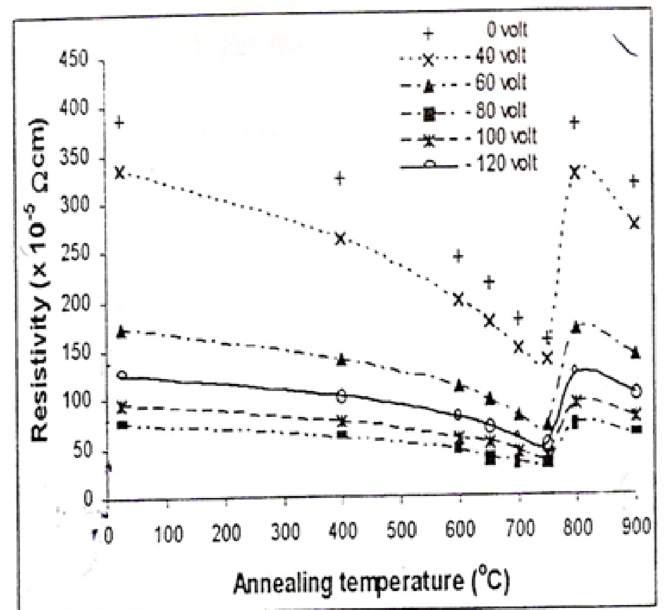


Figure 4: Resistivity variation of the amorphous TiN films with annealing temperature (Films deposited at 180ma ion current)

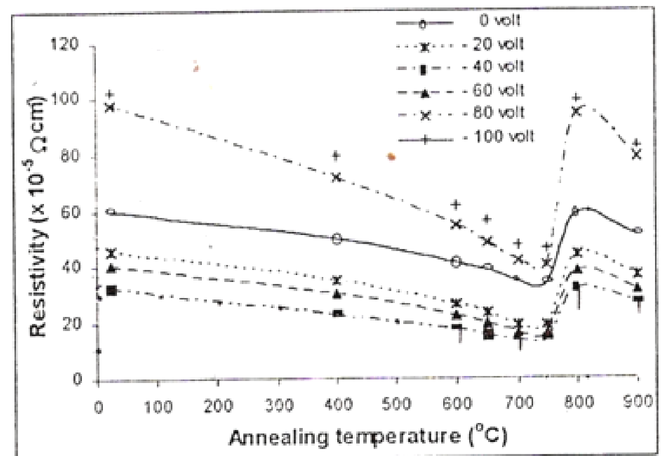
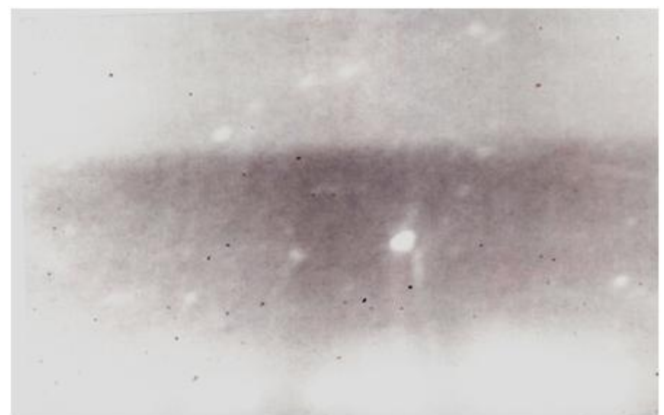


Figure 5: Resistivity variation of the crystalline TiN Films with annealing temperature (film s deposited at 200ma ion current)



(a)

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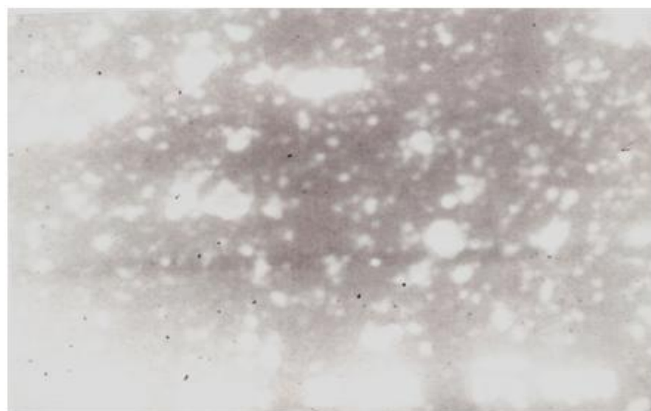


(b)

Figure 6: Surface SEM photographs of TiN films, annealed at (a) RT, and (b) 900^{0}C

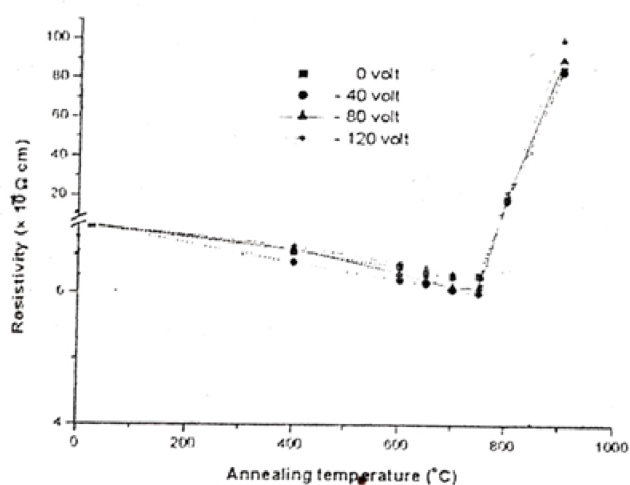


Figure 7: variation of resistivity of Cu/TiN/Si structure with annealing temperature

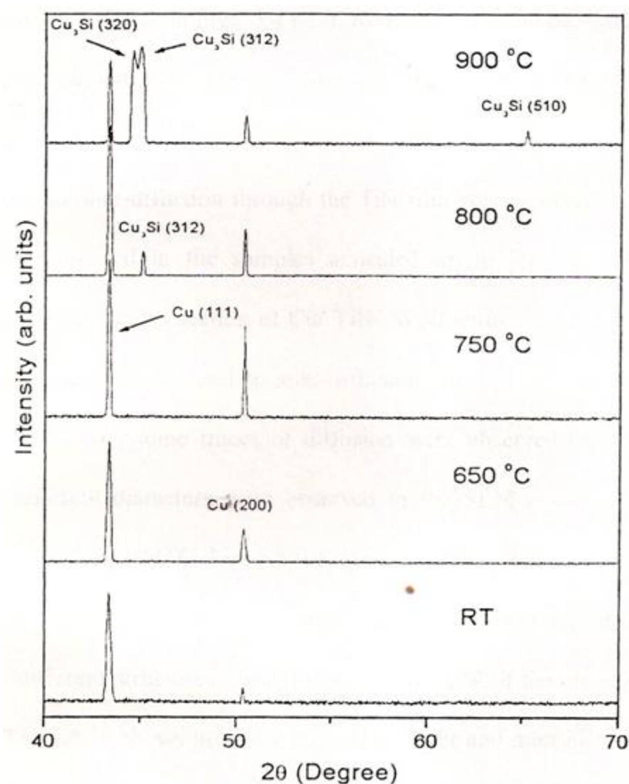
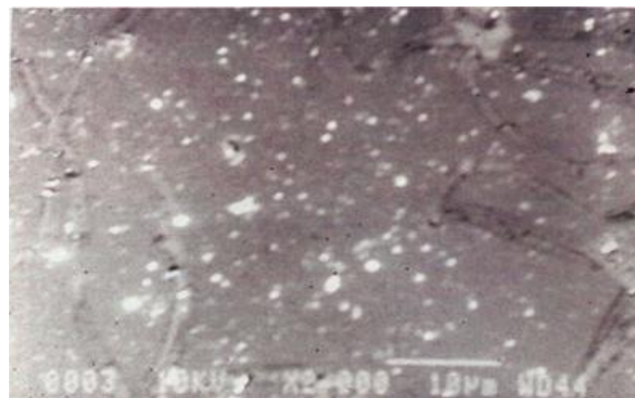


Figure 8: XRD results of Cu/TiN /Si Structure



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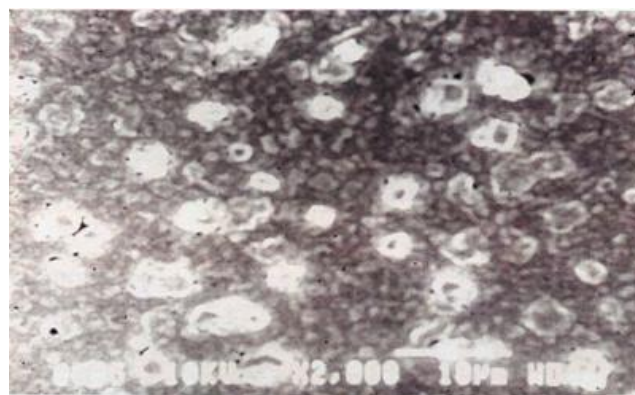

(ii)

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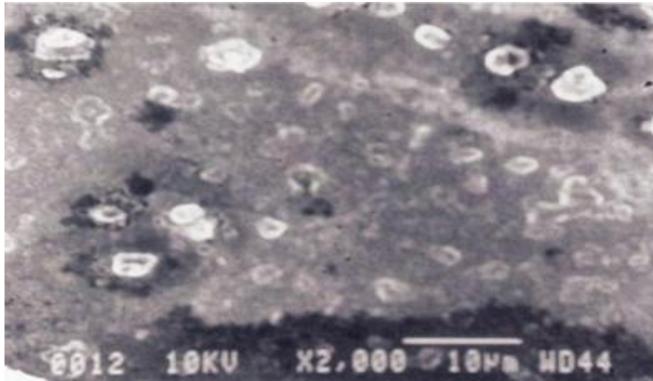
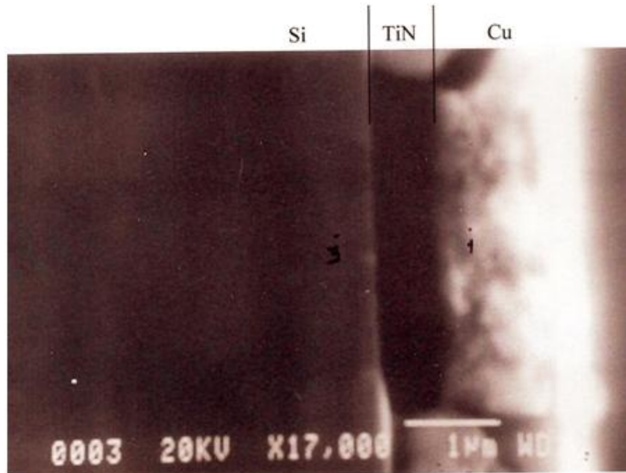
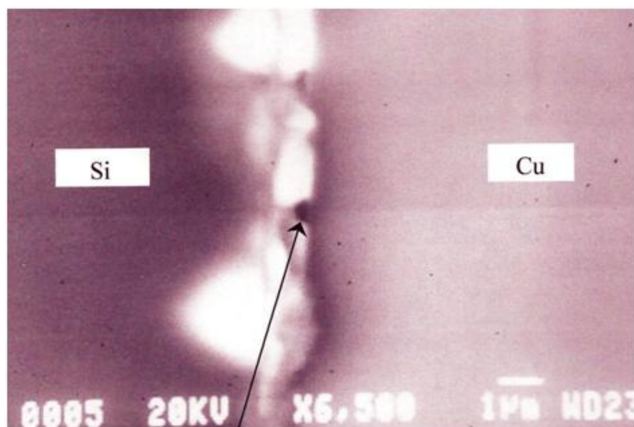


(iii)

Figure 9: Surface SEM photographs of Cu / TiN / Si, samples annealed at (i) RT, (ii) 650 ^{0}C, and (iii) 750^{0}C

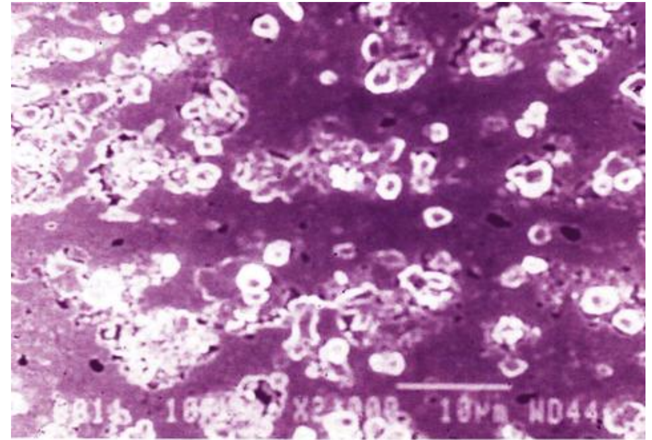


(a)



(b) TiN

Figure 10: Cross Sectional SEM photographs of Cu/TiN/Si, samples annealed at (a) 650^{0}C, and (b) 900^{0}C



(a)

(b)

Figure 11: Surface SEM photographs of Cu/TiN/Si, samples annealed at (a) 800^{0}C, and (b) 900^{0}C

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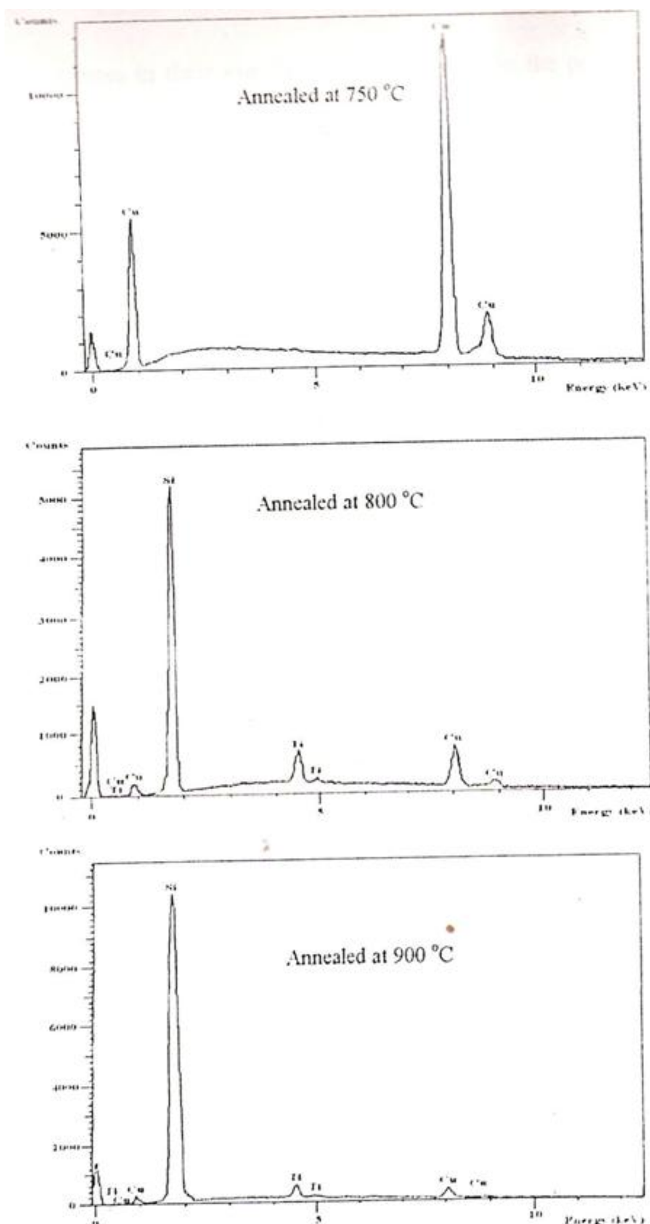

Figure 12: EDAX results of the Cu/TiN /Si Structure annealed at different temperature

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