Process Optimization for Improved Adhesion of Ti/Pt/Au to SiN and GaAs

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***Abstract*—This paper presents a methodology to improve adhesion of DC magnetron sputtered Titanium thin film to Silicon Nitride (SiN) surface and Gallium Arsenide (GaAs). Design of Experiments has shown that the argon pressure at which Titanium is sputtered is indicative of its adhesion. Higher Argon pressure reduces the mean free path length and maximum collisions occur in vacuum and lesser collisions on the substrate may cause minimal damage and improved adhesion. The improved adhesion in-turn also requires an improved lift-off process post deposition. Experiments have indicated that adhesion of the metal stack can be improved when Titanium (Ti) is sputtered at 17*mTorr*.**

INTRODUCTION

Ohmic contacts to GaAs are formed by deposition of a multilayer metal scheme such as Ti/Pt/Au on GaAs substrate. A technique such as sputter deposition or electron beam evaporation are generally used to deposit these materials. Ti is used as an adhesion layer while gold is a metal and has a relative permittivity of 6.9; Ti tends to diffuse completely into polycrystalline Au thin films at as low as a temperature of 250 C[1]. To avoid this from happening, Pt is used as a diffusion barrier between Ti and Au. Auger electron spectroscopy depth-composition analysis has also indicated that the metallurgical stability of Ti/Au is much lower than Ti/Pt/Au[2]. In many other cases different diffusion barriers are used such as Titanium Nitride (TiN). These diffusion barriers may be good up to about 400C -450C[3].

Adhesion of these multilayered stack is very critical to be able to make a very low resistance ohmic contact with specific contact resistance as low as 1.6 x 10^{-9} Ωm^2 and a factor η of 1.4. [2] The mechanical properties of Ti/Pt/Au are determined by several factors such as process conditions at which the films are sputtered and surface preparation of the substrate. The thermal expansion between film and substrate can influence stress in a thin film. This stress can change the adhesion properties of the thin film to the substrate.

THEORY

The mean free path length and sputter process pressure are inversely proportional to each other. The Structural Zone Diagram (refer to Fig.1) explains that the grain structures vary as a function of increase in Argon sputter pressure. The sputtered atoms due to higher sputtered pressure encounter a high number of collisions with the process gas before the redistribution of these atoms occurs at the surface of substrate. These atoms that redistribute on the surface will have much lower energies while still increasing the intrinsic temperatures in the vacuum system due to the collisions between sputtered atoms and argon gas. The collisions that occur in vacuum in a Physical Vapor Deposition (PVD) sputter system are partial inelastic collisions which are assisted by secondary electrons, and these electrons induce optical excitation of the gas (visible glow) used in sputter process. The structure of the film grown follows the Structural Zone Diagram - improves the grain structures and adhesion accordingly. As indicated in the Structural Zone Diagram below, Zone 1 is a porous structure consisting of tapered crystals separated by voids, Zone T is densely packed fibrous grains, Zone 2 is columnar grains and Zone 3 is recrystallized grain structure. The homologous intrinsic temperature is defined as *T/Tm,* where *T* is the substrate temperature and *Tm* is melting temperature of the target material in vapor (under vacuum in a PVD sputter).

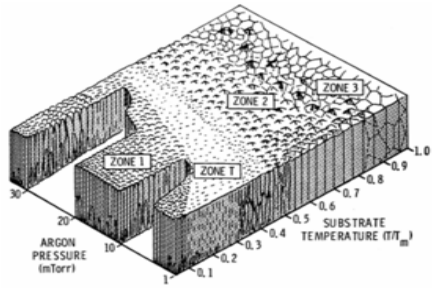


Fig 1. Structural Zone Diagram [4]

EXPERIMENTAL SECTION

All layers were deposited by a technique called PVD. The Structural Zone Diagram has been studied to understand the behavior of grain structure formation of thin film in its atomic level and its influence on adhesion through several experiments. Ti/Pt/Au (see Fig.2) was deposited on GaAs and SiN after patterned with photolithography and was then followed by a Liftoff process. The 3-inch wafers are treated with a wet Ammonia clean prior to deposition to remove native oxides. Ammonia clean is performed in three steps, dilute 1:10 Ammonium Hydroxide to water for 2 minutes, dump rinse for 5 cycles and spin rinse dry for 5 minutes. The failure mode in this case was that films (Ti/Pt /Au) indicated by hatched green were found to be delaminating and a bulk of the material would delaminate from SiN surfaces indicated in solid green and GaAs indicated in grey as illustrated in the below diagram (refer to Fig.2).

Fig 2. Illustration of Metal stack

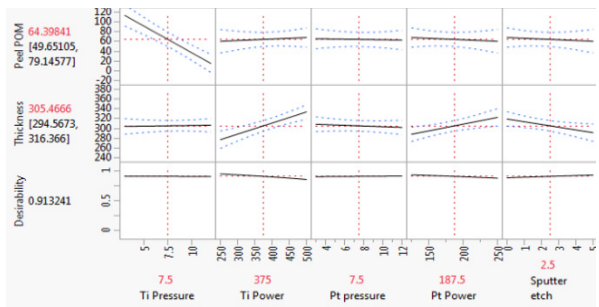
To improve this adhesion, critical parameters that can influence the adhesion were considered in the sputter process. The parameters chosen were Pressure (*mTorr*), Power (*W*) at which Ti and Pt were sputtered and Sputter Etch time (*min*) as preparation condition for the substrate. The output parameter that was considered was the number of dies that peeled. The number of dies peeled was termed "Peel POM" as shown in the analysis. The number of dies that peeled were counted after a Liftoff process. JMP statistical software has been used to analyze the data. A 2-level full factorial design has been chosen to perform these experiments (see Table 1).

TABLE 1. DESIGN OF EXPERIMENTS

INPUTS	Ti Pressure (m Torr)	Ti Power (W)	Pt Pressure (m Torr)	Pt Power (W)	Sputter Etch (min)
	3	250	3	125	0
	12	500	12	250	5

RESULTS

Through experiments, it has been concluded that the higher sputter pressure (refer to Fig.3) of the Ti layer in the multilayer stack of Ti/Pt/Au used to make ohmic contacts, improves the adhesion of the stack layer to GaAs and SiN. The design of experiments indicated that Ti pressure as a factor has the strongest signal to adhesion of these films. The analysis is shown in *Fig. 3*. The prediction model suggested that a better adhesion may be achieved by increasing the Ti sputter pressure. In statistical analysis using *JMP* (refer to Fig.3), a steep line slope of output vs. input parameter indicates an influential factor to the output i.e. number of dies peeled (Peel POM). It is indicative from the parameter estimates that Ti sputter pressure as a factor has a p value less than 0.05 and is the most significant factor while all other factors are insignificant. As process pressure increased, die peeling decreased.



Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	64.39841	6.98988	9.21	<.0001*
Ti Pressure(3,12)	-49.10448	6.57873	-7.46	<.0001*
Ti Power(250,500)	3.7179326	7.770553	0.48	0.6384
Pt pressure(3,12)	-1.31071	6.616339	-0.20	0.8453
Pt Power(125,250)	-3.717933	7.770553	-0.48	0.6384
Sputter etch(0,5)	-3.717933	7.770553	-0.48	0.6384

Fig 3.JMP Analysis

A quick experiment was designed to test this model. 3-inch GaAs substrates were cleaned in Ammonia, patterned with a felt tip pen prior to deposition in PVD. With all other parameters constant, only Ti process pressure was varied between 3*mTorr* and 18*mTorr* in increments of 1*mTorr*. Post deposition, a liftoff process was performed on these samples. It indicated that the feature definition was obtained, and metal had good adhesion at 17*mTorr* as illustrated in *Fig.4*.

Substrates that were sputtered at 17*mTorr* and 18*mTorr* for Ti layer deposition also had better adhesion and did not fail peel tests, scratch tests and metal layers stay adhered well even when followed with the liftoff process as described in the experimental section of this paper (refer to Fig.4).

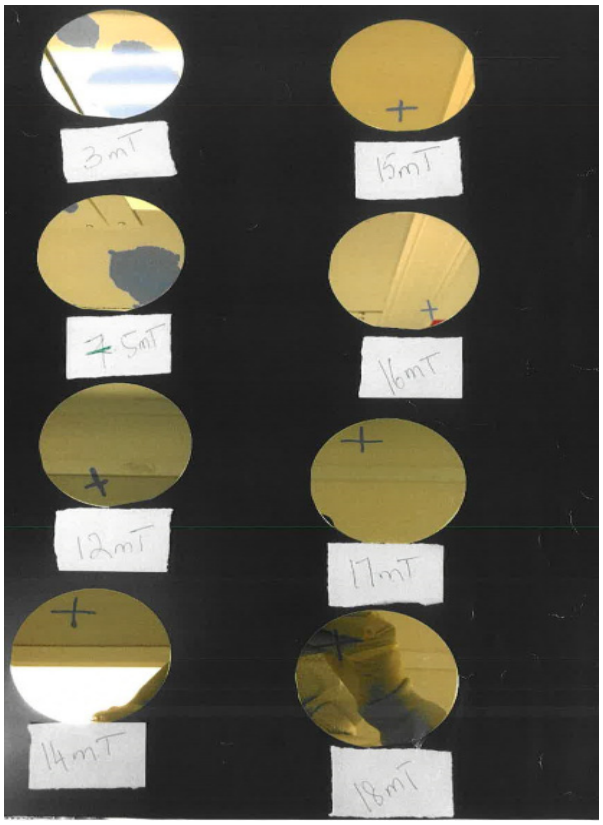


Figure 4. Experimental results

Figure 5 shows the effect of mean free path length of the chosen process from its standard 3*mT*orr process. The adhesion improvement was achieved. These experiments were performed on Plasma lab System 400 by Oxford Instruments. While this paper only discusses the effect on adhesion due to sputtered process pressure, other factors may be influential such as stress, surface preparation, sputter tool capability, sputter process parameters, distance between target material and the cathode etc.

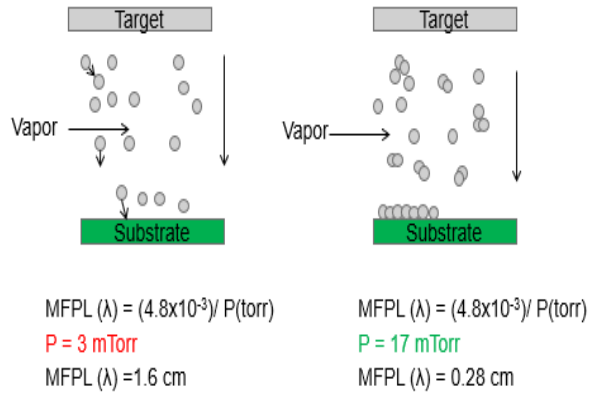


Fig 5. Illustration of Mean free path length decreases with increase in argon sputter pressure.

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