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Influence of mechanical stress on adhesion properties of DC magnetron sputtered Ti/NiV/Ag layers on n^+Si substrate

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ABSTRACT

Due to insufficient adhesion of sputtered Ti/NiV/Ag metallization scheme on n^+Si substrate when annealed below 550 °C, investigation was focused on the influence of process parameters on adhesion properties. Adhesion between metallic stack and Si substrate was found to be a strong function of annealing temperature. Additional investigations showed that the cause for poor adhesion was also rather high residual stress in metal thin layers, particularly stress in sputtered NiV layer. High residual stress was measured for NiV (1883 ± 252 MPa) and lower for Ti (334 ± 60 MPa) and Ag (310 ± 10 MPa) layer, respectively. The influence of sputtering parameters on the stress behavior was experimentally verified. By reducing the cathode DC power and Ar working pressure during NiV sputtering we were able to reduce the stress within the structure. A clear correlation was found between the residual stress magnitude and adhesion properties within the temperature range from room temperature to 550 °C. By residual stress reduction within a metallic stack, the necessary annealing temperature to obtain optimal adhesion was reduced from 550 to 500 °C.

1. Introduction

In order to obtain a reliable metallization for devices that require multilayer metallization schemes, we have to provide good adhesion to the underlying silicon active device and appropriate contact resistance. Adhesion of metal films on silicon surfaces is in many aspects a process dependent property. By choosing the structure of the metallic stack, deposition methods and thermal treatments we assure appropriate adhesion properties and avoid delamination problems during bonding and packaging stage, which is essential in the production of MEMS and similar discrete electron devices.

The main reason for using Ti/NiV/Ag metallization scheme is that the end user packaging technology requires solderable type of metallization for particular photosensor devices used in rotary and linear optical encoder systems. With respect to the similar solderable metallization schemes such as Ti/Ni/Au or Cr/Ni/Au, the Ti/NiV/Ag scheme is the most economic and still reliable.

The adhesion of deposited layers is usually determined by applying external mechanical stress until failure occurs at the interfacial region. In some cases, when adhesion is low, delamination occurs spontaneously. In general, adhesion tests are used as comparative method and not to obtain absolute values of the adhesion strength. In this work a scotch tape method was used to evaluate the adhesion of deposited layers.

The adhesion of the sputtered Ti/NiV/Ag metallic stack on n^+silicon used in our standard applications is found to be excellent only when annealed at a temperature of 550 °C. The adhesion of Ti/NiV/Ag was severely reduced for chosen metallization scheme when thermal annealing was performed at lower temperature (385–450 °C) where partial or total peeling of metallic stack was found.

The delamination of Ti/NiV/Ag metal layers from the silicon devices during removal of the dies from adhesive foil after the dicing operation was found to be a frequent problem and had to be investigated thoroughly. In the first approximation, this is usually attributed to a low degree of chemical bonding due to insufficient thermal treatment, poor surface preparation or presence of contaminants. Delamination can occur between individual layers or between Si and the metallic stack. Very thorough investigations have been performed by several authors regarding the adhesion related mechanisms of the multilayer metallization and different solutions were proposed [1–3]. In other studies, where adhesion was not a problem, reduction of stress in multilayer metal structures on very thin Si substrates was of importance [4].

In the previous report [5] we analyzed the delaminated samples by Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) and confirmed that delamination occurred between the silicon and the amorphous Ti–Si layer which is formed at Ti–Si interface when annealing is performed at temperatures below 500 °C.

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The aim of this work was to identify the most influencing process parameters which affect and reduce adhesion strength such as surface preparation, sputtering conditions, annealing temperature, annealing ambient and mechanical stress. The final task was to establish appropriate parameters of sputtering process to improve adhesion at lower annealing temperature. Reducing the annealing temperature was shown to improve the photoresponse properties of the photosensor device. Our analyses were focused in particular on residual stress in individual layers and within the complete metallic stack.

2. Experimental details

Experimental work concerning the adhesion test was carried out on 100 mm diameter, 400 μm thick, high resistivity (500 Ωcm), *n*-type, one side mechanically polished float zone (FZ) silicon wafers with <1 0 0> crystal orientation. Metallization schemes were realized with DC magnetron sputtering method by MRC603 three target batch sputtering tool operated in a manual mode. The system was evacuated by CT-8 cryo pump backed with two stage mechanical pump. Base vacuum of the system was maintained at 2×10^{-7} torr prior to each deposition process. The planar NiV target composition consists of 93% Ni and 7% V, with declared purity of 99.95%. The other two targets were a planar Ti target with the purity of 99.997% and an inset Ag target with the purity of 99.995%, respectively. Standard sputtered metallic stack applied in our p^+n n^+ photo sensitive device consists of Ti (70 nm)/NiV (350 nm)/Ag (1200 nm). During the sputtering process, silicon substrates attached on the pallet are passing the target in a vertical position. Scan speed of the pallet with substrates was 25 cm/min for Ti deposition, 25 cm/min for NiV deposition and 12 cm/min for Ag deposition. Short presputtering step (t = 30 s) was performed prior to each thin film deposition to clean the target surface. Ar working gas pressure was varied between 3 and 12 mtorr. For the adhesion tests with scotch tape, samples were diced to equal size (2.3×1.1 mm^2).

Due to determined strong influence of process steps on the final adhesion, tests were performed on the wafers with full thermal and chemical history and not just on the bare test wafers. To accomplish this, tests were performed with simulated chemical processes and n^+ deposition on the rear side to provide ohmic contact. This n^+ layer was accomplished by doping silicon with phosphorus from solid diffusion sources PH950 (Saint Gobain). Thermal annealing of metallized wafers was performed in forming gas atmosphere (5 % H_2+95 % N_2) in the temperature range from room temperature (RT) to 550 °C.

To measure residual stress of sputtered individual layers and complete metallic stack, 3 in., (1 0 0) silicon test wafers with resistivity 1–2 Ω cm were used. Residual stress in thin metal layers was determined by wafer curvature technique [6] and the thickness of metal layers was measured with surface profiler Taylor–Hobson model Tallysurf form 1.

3. Results and discussion

Sputter RF etching was used in early experimental stage as *in-situ* cleaning to remove in particular the native oxide from the Si substrate. As a consequence, contamination from the re-sputtering process took place and instead of clean silicon surface some re-sputtered material from the pallet and eventually from the chamber was commonly obtained on the Si surface thus reducing the adhesion. To improve this, a 3 min treatment in HF (49%): DI water (1:100) followed by DI water rinse and N_2 drying was used instead.

Beside the mentioned role of silicon surface preparation and different temperature dependant chemical phases which form at metallic stack/Si interface [5], mechanical stress has also a significant influence on the adhesion properties of metal layers on Si substrate. It is common that all physical vapor deposited (PVD) as well as chemical vapor deposited (CVD) films have residual stresses due to the growth conditions of the layer, thermal gradients within the deposited layers and stress arising from the differences of thermal expansion coefficient of the layer and the substrate. These phenomena may cause a problem particularly in multilayer structure and facilitate the delamination process. If the mechanical stress in thin films is larger than the adhesion energy of the first layer to the substrate, the delaminating process of the whole stack takes place.

Nucleation density of deposited film atoms is an early indication of adhesion state. A high nucleation density indicates strong chemical interaction of deposited atoms and the substrate surface. Generally, a dense film is desired; however, such a film will transmit stresses more easily than a less dense or more porous film. Densification of the film during crystallization produces stress which can be relaxed at higher temperatures via diffusion controlled viscous flow and dislocation motion at higher temperature [7].

The growth conditions governed by sputtering parameters play important role in structural properties of deposited thin metal layers. This is also reflected by the intrinsic stress built up in the thin film. The stress in thin metal layers can be tailored in the narrow tolerance range by setting the sputtering parameters such as sputtering rate, cathode DC power, RF substrate bias, Ar working gas pressure and substrate preheat temperature. In addition, both, Ti and Ni are relatively reactive materials; therefore vacuum below the 10^{-6} torr range is in general pre-required in the growth process of pure films.

The first set of experiments was performed to evaluate quantitatively the stresses of each metal layer and the combinations of metal layers within the established sputtering process parameters used in our standard process as presented in Table 1. After sample preparation and sputtering the required metal layers, residual stress σ_r in thin metal layers was evaluated by wafer curvature measurements and calculated according to the Stoney equation Eq. (1), which is valid in the linear, elastic region of the stress–strain plot [6]

$$\sigma_r = \frac{E_s}{1-\nu_s}\frac{t_s^2}{6t_f}\left(\frac{1}{R_1}-\frac{1}{R_2}\right)[\mathrm{Pa}]. \quad (1)$$

Here, E_s is Young's modulus of the substrate, ν_s Poisson ratio of the substrate, t_s silicon substrate thickness, t_f thickness of the thin film producing the stress, R_1 is the radius of curvature under stress, andR_2 is the radius of curvature after removing the stress inducing film, i.e. curvature of the bare wafer. The value of 181 GPa for silicon substrate biaxial modulus $E_s/(1-\nu_s)$ was taken from the literature [3,8]. The above equation Eq. (1) is valid for $t_s \gg t_f$, a criterion our structure complies with.

Table 1
Sputtering conditions, material properties and measured stress of as deposited metal layers

	Ti	NiV	Ag	Si
p_{Ar} [mtorr]	6	6	8.5	
T [°C]	200	180	180	
P [kW]	3.5	3	2.5	
t_f [nm]	70	350	1200	
E [GPa]	116	207	76	132
ν	.34	.31	.37	.27
α 10^{-6} [°C^{-1}]	8.9	13.1	19.9	2.5
σ_r [MPa]	334 ± 60	1883 ± 252	310 ± 10	
σ_{th} [MPa]	197	556	367	
σ_i [MPa]	137 ± 60	1327 ± 252	−57 ± 10	