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Stress measurements in tungsten coated through silicon vias for 3D integration

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ABSTRACT

In 3D integration, interconnections between stacked dies are ensured by conductive through silicon vias. Electrical conduction is achieved via coating the vias sidewalls with a metal, such as tungsten. In this work we have compared thermal-dependent stress of thin tungsten films deposited either in full plate oron vias sidewalls. The comparison of stress measurements at room temperature and during heating cycles reveals large differences between full plate and vias samples. At room temperature, in the vias samples, the stress is a factor 4 less than it is in the full plate sample, with both values indicating a tensile stress. While a thermo-elastic behavior is expected for the full plate sample, no stress evolution as a function of temperature is observed in the case of the vias samples.

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1. Introduction

3D integration is presently an emerging technology, which will enable the realization of highly integrated and complex systems by vertically stacking and connecting various materials, technologies and functional components together.

The aim is to miniaturize the interconnections between the dies and to improve the performance in electronic devices. In this technology the dies are vertically interconnected with metal filled holes etched in active chips or silicon substrates. These interconnections are called Through Silicon Vias (TSV). The TSVs may be fully or partially filled with highly conductive metals like copper or tungsten [1].

The very different thermo-mechanical properties of the metal filler and of the surrounding material (silicon substrate) can lead to significant induced stress in and around the TSVs [2,3]. This may highly impact the performances of final devices and also their reliability [4]. The understanding of the thermo-mechanical behavior of TSVs is one of the most important challenges in 3D technology.

In this work we have determined the stress values in thin tungsten films deposited either in full plate oras sidewalls in TSVs, from X-ray diffraction measurements with a 4-circle diffractometer. At room temperature, the sin²(ψ) method was used to assess the stress in blanket films, whereas in TSVs a simplified model has been developed, because of the specific geometry. In a second step a comparison of the stress evolution during a thermal loading in full plate and TSVs is presented.

2. Experimental details

2.1. Samples description

The samples are $1 \times 1 \text{ cm}^2$ dies with a specific stack deposited on the silicon substrate. The stack is composed of silicon oxide (500 nm), titanium (40 nm), titanium nitride (55 nm) and tungsten (200 nm), with Ti/TiN/W stack repeated 2 times. Two kinds of samples will be compared: full plate geometry and TSV geometry where the stack is sidewall-deposited inside the TSVs (Fig. 1). The TSVs present an aspect ratio of 2:5 (250 µm depth, 100 µm diameter) and are spaced 400 µm apart. A specific process sequence was used to manufacture the TSV samples. TSVs are etched with Deep Reactive Ion Etching process after a photoresist mask deposition. The Ti/TiN films are deposited by sputtering and the W films are deposited using Chemical Vapor Deposition.

2.2. X-ray diffraction

In order to obtain the inter-planar distance of crystallographic planes and the thermal evolution of the probed material a 4-circles diffractometer is used (4 angles: ω , 2θ , φ and ψ) (Fig. 6). The Panalytical X'Pert Pro diffractometer is equipped with a sealed copper source generating an X-ray radiation with a wavelength of 1.5406 Å (K α_1). Polycapillary optics is used to induce a parallel like incident beam. The diffracted beam is angularly selected with a parallel plate collimator and a graphite monochromator. The scattered intensity is recorded with a proportional detector. For temperature-dependent measurements, an Anton-Paar furnace (DHS 1100) adjustable on our X-ray diffractometer sample holder is used. For this study, the samples are thermally cycled up to 500 °C and strain measurements

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Fig. 1. Schematic view of a) full plate and b) TSVs samples. Tungsten film is sidewall-deposited inside the TSVs (b).

are performed every 50 °C. For both samples, two thermal cycles were performed under a neutral argon atmosphere.

2.3. Scanning electron microscopy

The presented images were performed with a Scanning Electron Microscope. The microscope is a Philips XL30 SFEG STEM with a minimal spatial resolution of 1.2 nm. The topography of our samples, in top and cross-section view, is obtained with secondary electrons under a 20 kV operating voltage.

3. Results

3.1. Microstructure

To perform any stress analysis, a texture analysis of the studied thin film is recommended. First, a $\omega/2\theta$ -diagram (Fig. 2) is recorded in symmetric geometry for full plate and TSVs samples.

The patterns, with corresponding diffraction peaks of tungsten and titanium, exhibit a polycrystalline behavior with a nearly random orientation of the tungsten grains. Nevertheless, a weakly preferred orientation of the (211) plane parallel to the surface is observed for the full plate sample. The symmetric $\omega/2\theta$ scans revealed that the tungsten in the TSV was polycrystalline in nature, but there is no specific information about the texture. A texture analysis was performed on the tungsten thin film of the full plate sample. This measurement is not possible in the TSV because of the weak diffracted intensity resulting from the small tungsten diffracting volume. The pole figures are recorded with a 5° step in the specific ranges of the azimuth φ (0° to 360°) and the inclination ψ (0° to 85°) angles. Pole figures were recorded



Fig. 2. $\omega/2\theta$ diagrams of full plate and TSVs samples. Intensity for TSV sample has been multiplied by 20. Both samples exhibit a polycrystalline texture with a nearly random orientation of the grains.

for different tungsten crystallographic planes: (110), (211). The direct pole figures shown in Fig. 3 are normalized in order to take into account defocusing effects and absorption [5].

As already shown by the $\omega/2\theta$ diagram, the tungsten film presents a weak (211) fiber texture.

Scanning Electron Micrographs (SEM) (Fig. 4a) show a lateral grain size of 300 nm in the W film deposited in the full plate geometry. Fig. 4b shows the SEM image of the TSV sample in cross-section. It shows that Deep Reactive Ion Etching, which was used to etch the hole in the silicon substrate, induces a wave-like wall, known as scallops [6], with a period of 2 μ m and amplitude of 0.25 μ m.

At higher magnification (Fig. 4c), the W films in the TSV exhibit a columnar grain shape with an average grain diameter equal to the film thickness: 200 nm.

3.2. Stress at room temperature

Strain measurements are performed in a symmetrical geometry using the inclination angle ψ to measure the strain for crystallographic planes non-parallel to the surface. Tungsten is an isotropic elastic material with a Young's modulus E=411 GPa and a Poisson's ratio v = 0.28 [7]. The thin film approximation is used to calculate the inplane stress: the stress is equi-biaxial in the plane of the film: $\sigma_{11} = \sigma_{22} = \sigma$, $\sigma_{33} = 0$. Then the well-known $\sin^2(\psi)$ method (Eq. (1)) is used to determine the stress at room temperature, which links the strain ε_{ψ} to the in-plane stress σ , as follows for an elastically isotropic material [8]:

$$\varepsilon_{\psi} = \frac{\left(1^{\circ} + {}^{\circ} \nu\right)}{F} \sigma \sin^2(\psi) - \frac{2\nu}{E}.$$
 (1)

The unstressed lattice parameter of the studied material can be obtained at $sin^2(\psi_0) - \frac{2\nu}{(1+\nu)}$ through the ε_{ψ} measurement.

A $\sin^2(\psi)$ plot of the strain for Tungsten {310} and {321} planes in the full plate sample is shown in Fig. 5. The slopes obtained from these two sets of measurements indicate a similar stress for both plane families.



Fig. 3. Pole figures of tungsten (110) a) and (211) b) planes in the deposited film in full plate geometry.

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