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Wetting process of copper filling in through silicon vias

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ABSTRACT

In this paper, wetting process of copper filling in through silicon vias was investigated by agitation and vacuum pretreatment. Copper filling followed the pretreatment was fabricated in electrolyte with optimal additives concentration. By agitation pretreatment, a large void was observed at the bottom of the vias where the copper seed layer was clearly seen, which was attributed to the insufficient wetting of vias. In the blind vias with higher aspect ratio, the ambient gas phase was difficult to be completely removed from the vias by agitation. This resulted in electrolyte having better wettability does not reach all the places of the vias by capillarity. The vacuum pretreatment results suggested that the air inside the vias was almost completely evacuated. Hence, the deionized water used as wetting solution easily permeated the blind vias relying on atmospheric pressure. Consequently, the uniform, complete and void-free copper filling was achieved due to better wettability of 273 K deionized water. Nevertheless, deionized water with temperature higher than the critical vaporization temperature yielded a void formation at the bottom of the vias, which resulted from the insufficient wetting caused by the vaporization of deionized water. The conclusions drawn by the experimental results were employed in the through silicon vias, and void-free copper filling in the vias having aspect ratio as high as 16 was fabricated.

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1. Introduction

Copper filling is particularly significant to achieve electrical interconnection in three-dimensional integrated circuits [1]. In order to ensure the safety and reliability, the through silicon vias (TSVs) must be fully filled with deposited copper, which is commonly referred in literature to as bottom-up or superconformal growth [2]. Most previous articles on void-free deposition focused on the behavior of organic additives, such as accelerator [3,4], suppressor [5] and leveler [6]. The presence of these additives carefully controls the enhanced deposition rate at the bottom of vias relative to the suppressed deposition rate on the planar wafer surface and the upper side walls of the features [7–9].

Although the superconformal growth is well-established in electrolyte with optimal additives concentration, void-free copper deposition in TSVs is still a challenge. When the copper was deposited in the vias, the electrolyte cannot wet the vias surface completely and in deep and narrow vias even does not reach some local vias points [10–12]. Due to the insufficient wetting of TSVs, electroplating remains open at incomplete wetting local points, which leads to the void formation and poor electrical performance. In order to overcome the problem of void formation and to achieve uniform and void free deposition in TSVs, the wetting of surface of copper seed layer in TSVs should be improved. Predeep Dixit et al. [13] reported various surface treatment methods such as plasma, UV and wet treatment to improve the wetting characteristics of vias sidewall surface and through-wafer copper interconnects of aspect ratio as high as 20 were fabricated accordingly. Adding proper additives and the chelating in the electrolyte can also enhanced the hydrophilic characteristic of the vias [14]. Although the wetting characteristic of the vias has been improved by these surface treatment methods, theoretical surface tension enabling electrolyte to reach the bottom of the blind vias is still much greater than the actual surface tension of electrolyte [15]. That is because air pressure inside the vias, which is the only resistance to the wetting process in the blind vias, is not decreased by these methods. Hence, the air inside the blind vias must be completely removed to achieve proper wetting of the vias. A variety of different approaches, such as agitation [16,17], ultrasonic vibration [18,19] and vacuum equipment [9] can help electrolyte permeating the vias and evacuating the air inside the vias.

In this paper, agitation and vacuum treatment was performed in wetting process to help the air removed from the vias. The copper deposition was fabricated on wafer patterned with TSVs having different aspect ratio followed the wetting process. Scanning electron microscope (SEM) experiments were carried out to examine copper

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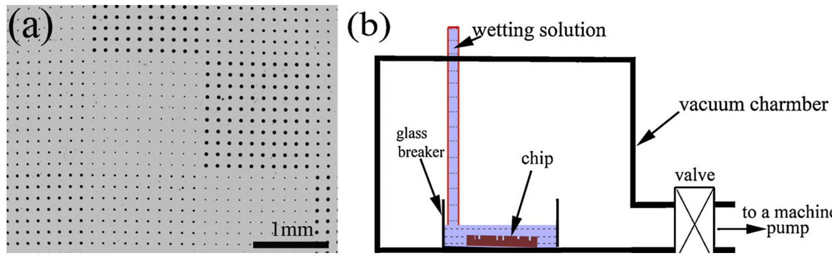

Fig. 1. (a) The SEM surface morphology of vias on the wafer. (b) The schematic diagram of the devices for vacuum pretreatment.

filling performance. Finally, the effect of the temperature of deionized water used as wetting solution after vacuum pretreatment on the wettability was discussed.

2. Experimental

The experiments were conducted on wafer fragments patterned with TSVs arrays provided by Shanghai Sinyang Electronics Chemicals Co., Ltd., China. Fig. 1(a) showed the surface morphology of vias patterned on the wafer. The vias were lined with a dielectric insulation layer followed by deposition of barrier layer and Cu seed layer (150 nm). The aspect ratio of TSV was defined as the ratio of the via height and via diameter. In the experiment, the aspect ratio of TSV varied from 2:5 to 16:1.

The vacuum pretreatment of specimen was carried out in equipment consisted of a vacuum chamber and pump, as shown in Fig. 1(b). The specimen was firstly vacuumed for 15 min in the vacuum chamber followed by immersed in the wetting solution and rapidly transferred to the plating cell.

The composition of base plating electrolyte is 40 g/L $Cu(CH_3SO_3)_2$, 60 g/L CH_3SO_3H and 50 ppm Cl^-. The standard superfilling electrolyte contains 1 ml/L accelerator, 9 ml/L suppressor and 3 ml/L leveler in the base plating electrolyte. Copper filling was carried out at room temperature with a current density of 0.4 A/dm^{-2} and deposition time of 5 h. After electroplating, the specimen chips were mounted in epoxy resin and cross sections of TSVs were prepared by grinding and polishing. Polishing was performed carefully with frequent microscopic observation to obtain vias center images. SEM images were obtained using Hitachi TM3000. Contact angles were measured with 4 μL deionized water and electrolyte by a contact angle meter (OCA20, Dataphysics instruments GmbH, Germany) under static conditions.

3. Results and discussion

Prior to the wetting process, the wetting characteristics of the copper seed layer on the wafer were measured with three types of wetting solution, such as deionized water, base electrolyte and standard superfilling electrolyte. The wafer was exposed to the air after received from Shanghai Sinyang Electronics Chemicals Co., Ltd., China, so the copper seed layer may be slightly oxidized decreasing its wetting property. The microscopic images of wetting solution droplets formed on the copper seed layer are shown in Fig. 2. It is clearly illustrated in this figure that the copper seed layer is hydrophilic in nature, that is, the contact angels with three types of wetting solution were less than 90°. Among the wetting solution tested, the standard superfilling electrolyte had shown the best wettability, that is, contact angel is 35° (Fig. 2(c)), making it a more choice suitable for wetting solution in wetting process.

The wetting process was conducted by immersing the wafer patterned with TSV into the plating cell with standard superfilling electrolyte. Then, the agitation was applied by usual magnetic stirrers at a rotation speed of 200 rpm. Followed wetting process, copper deposition of specimen was performed in the standard superfilling electrolyte. The cross-section images of vias with a height of 100 μm and different diameters were obtained to examine the copper filling, as shown in Fig. 3. An evident void was observed at the bottom of the vias where copper seed layer was indeed clearly seen while the upper part of the vias was fulfilled by copper. Note that a significant increase in the height of the void was observed with decreasing the diameter of the vias. The effective explanation for void formation was electrolyte did not reach the bottom of the vias where electroplating remained open in copper filling process.

In order to gain insight into the mechanistic behavior of the wetting process, impact factor was explored. In the wetting process by agitation, the electrolyte firstly contacted the surface of the wafer

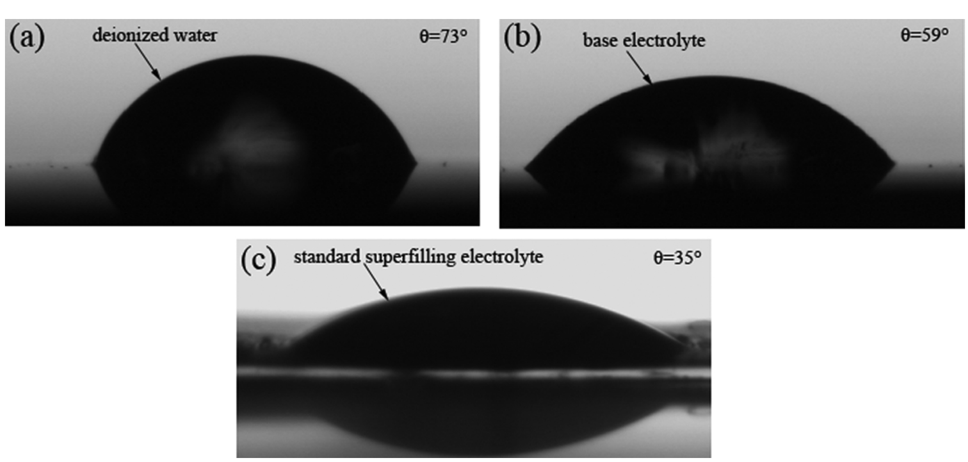

Fig. 2. Contact angle between copper seed layer and (a) deionized water, (b) base electrolyte, (c) standard superfilling electrolyte.

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