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# Fabrication of through-silicon vias by supercritical CO<sub>2</sub> emulsion-enabled nickel electroplating



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#### ABSTRACT

Compared to the 2D plane, 3D integrated circuit (IC) structure could provide larger patterning areas by stacking the multi-planar chips, in which the electrical signals can be vertically conducted via through-silicon vias (TSVs). Thus, its advantages are lowered costs and reduced packaging space, size and weight. In this study, the TSVs used for 3D integration are fabricated and characterized. Four through holes with a diameter of 70  $\mu$ m on a silicon wafer are first etched by inductively coupled plasma reactive ion etch (ICP) and filled by nickel electroplating in supercritical CO<sub>2</sub> emulsion. The chip is cut for observation and examination of the cross-sectional view of the TSVs. For hermeticity testing, a helium leaking detector was performed on all TSVs before and after the heat treatment process (heating up to 350 °C). The average electrical resistance across the TSVs was measured to be 0.01  $\Omega$ . Then the fabricated TSVs can be applied a maximum current of 10 A continuously without burnout.

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#### 1. Introduction

Current electronic products are constantly improved through innovative research and development. Thus, as the hardware requirements are higher, the traditional 2D IC structure has been unable to meet the current criteria for electronic products. Some research teams began to focus their research in the field of 3D IC integration. Recently, due to the technical breakthroughs in the field of MEMS technology, the semiconductor IC circuits and sensors can be miniaturized. However, with the size limit of 3C products (computer, communication and consumer electronics), circuits and wire patterning on a chip are now more constrained. Hence more studies are carried out on the through-wafer interconnects technology to explore how to implement an integrated circuit onto a 3D

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http://dx.doi.org/10.1016/j.mssp.2014.02.025 1369-8001 © 2014 Elsevier Ltd. All rights reserved. structure. Ko et al. reviewed the wafer-level bonding/ stacking technologies for 3D IC integration [1]. Fischer et al. inserted the nickel wires inside the through wafer vias to conduct electrical signals between the upper and lower chips [2]. Fujimoto et al. thinned many wafers by chemical mechanical polishing (CMP) and stacked up all the layers on a base wafer. The copper was filled inside the patterned TSV for interconnection [3]. Wei et al. used the general electroplating process to fill the blind holes and thinned the wafer by CMP to expose the TSVs for interconnection [4]. Chuang et al. used the traditional copper electroplating method (DC current) to complete the TSV wafers for interconnection and hermeticity [5].

Dixit and Miao then did a series of complete studies on the bottom-up electroplating techniques [6]. The contact angle analysis was first performed to study the hydrophilicity of the TSV inner sidewall and the SC1 solution (30% H<sub>2</sub>O<sub>2</sub>, 25% NH<sub>4</sub>OH and deionized water in 1:1:5 ratio) was suggested to apply on the inner surface of TSVs to improve the hydrophilicity. Thus, the plating solution can flow more smoothly along the sidewall of TSV into the deeper hole. In addition, they also proposed a new electroplating method, aspect-ratio-dependent electroplating technique. As the plating time increases, the electroplated metal will be more close to the anode, so that the distance between the cathode and anode will be changed accordingly. In order to maintain current efficiency during the electroplating, the current density was suggested to be adjusted periodically depending on the distance between cathode and anode. However, the above method requires a longer time to complete the entire TSV filling electroplating process.

In 2002, Sone et al. [7,15] proposed a new electroplating method, supercritical-CO<sub>2</sub> in emulsion, for the electroplating of nanostructured nickel. Their results show that a finer grain, a higher hardness and a smoother surface morphology on the plated metal can be obtained by using this new electroplating method. Compared with the general electroplating process, the electroplating in supercritical-CO<sub>2</sub> emulsion has a better performance in solution permeability and coverage. They have continued to improve the uniformity of the CO<sub>2</sub> and plating solution (emulsion) under the supercritical status by adding the nonionic surfactants [8,9]. In addition, Nguyen et al. [10,11] focused on studying the internal stress of Ni coating electroplated in the supercritical-CO<sub>2</sub>. In this study, several through-silicon vias were filled by the nickel electroplating in a supercritical-CO<sub>2</sub> status for applications in 3D IC integration and an aspect ratio of 1:7.5 was achieved. After a series of tests, our electroplated TSV structure is vacuum sealed, has low resistance, and is capable of carrying high current. In addition, it only took 3 h to complete the entire TSV electroplating process in a supercritical-CO<sub>2</sub> status, and thus, substantially enhancing the efficiency of the TSV filling electroplating process.

#### 2. Experiments

### 2.1. Electroplating in supercritical CO<sub>2</sub> emulsion and the apparatus

Supercritical phase is different from the ordinary three phases, solid, liquid and gaseous. The formation of phase of any matter can be different by changing its temperature and pressure. The supercritical phase is in between the gaseous and the liquid phase boundary before the pressure and temperature of matter reach the critical point. There will be another homogeneous phase generated, supercritical phase, once the pressure and temperature of the matter go beyond the critical point [7,8]. Since the supercritical fluid is in between the liquid and gaseous phase, its advantage is having positive physical properties of both phases, such as lower viscosity, higher permeability and lower surface tension than its liquid phase; higher diffusivity and better solubility than its gas phase. In addition, the plated metal in supercritical-CO<sub>2</sub> emulsion has smaller grain size, higher hardness, better coverage, lower surface roughness and pinhole-free microstructure compared with the conventional electroplating process. In the traditional plating process, some specific chemical additives such as brightener and leveling agent are generally added into the plating solution to achieve the shiny surface and improve

the smoothness of the electroplated metal layer. However, the repeated electroplating process depletes the additives which must be resupplied constantly in accordance with a certain concentration of plating solution, and thereby increasing the complexity of the plating process. If the electroplating is carried out in supercritical-CO<sub>2</sub> emulsion, the same plating quality can be achieved without adding any additives. In addition, the reason to use the CO<sub>2</sub> in the supercritical phase is that it can dissolve the hydrogen bubbles, and thus, can reduce the occurrence of voids [7,9]. Moreover, it is non-pyrophoric, non-toxic, inexpensive and the condition of a supercritical-CO<sub>2</sub> status is closer to the room temperature and normal atmosphere. In this study, the electroplating is carried out under the supercritical-CO<sub>2</sub> emulsion which allows the plating solution to flow into the TSV holes more easily due to its low surface tension and viscosity. Then TSVs can be filled by the bottom-up nickel electroplating in supercritical-CO<sub>2</sub> emulsion. From the results, it shows that it only took 3 h (DC setup) to complete the TSV plating in supercritical-CO<sub>2</sub> emulsion which is much shorter than using the traditional electroplating process (DC, 72 h) [5] for the same diameter and thickness of nickel TSV. Therefore, the electroplating in supercritical-CO<sub>2</sub> status can greatly shorten the electroplating process.

The apparatus used for electroplating in supercritical-CO<sub>2</sub> is shown in Fig. 1. In this study, the bottom-up plating technology is used to fill up the TSV in order to meet the requirements of the 3D IC interconnection. During the electroplating process, it is found that if the current density is set too high, the deposition of the nickel pillar will be too fast which can easily lead to an uneven TSV electroplating. From the results, if a lower current density was utilized for TSV plating, although the deposition rate was slow, the plated metal structure was found to be more firm which makes TSV pillars more robust. In this study the diameter and depth of all TSVs are 70  $\mu$ m and 525  $\mu$ m, respectively. An aspect ratio of 1:7.5 was obtained by taking the advantages of low surface tension, better permeability in supercritical-CO<sub>2</sub> electroplating.



**Fig. 1.** High pressure supercritical electroplating reactor used for electroplating in supercritical-CO<sub>2</sub> emulsion. (1) CO<sub>2</sub> cylinder, (2) cooler, (3) air compressor, (4) high pressure pump, (5) circulating hot water jacket, (6) electroplating bath with a stirring bar, (7) power supply, and (8) hot water reservoir. P: Pressure gauge, V: Pressure valve, and T: Temperature indicator.

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