1. Introduction

Rapid progress of information technologies for the internet of things has been promoted by the functionalization of single integrated circuit (IC) chips. Improvement of IC chips has been achieved by densification of transistors in IC chips following Moore’s law. In the past decade, three-dimensional (3D) ICs have become a research trend to overcome the limitation of Moore’s law.[1, 2] A 3D IC is constructed of vertically stacked and interconnected thinned IC chips. Such a configuration lowers power consumption because it decreases the resistance by shortening the wire length, and also shortens propagation delays as well as increasing the transistor density.[3, 4] One important technology in 3D ICs is the vertical interconnections formed between the stacked layers using metal bonding techniques. Cu has been widely used as an interconnection material because it shows high electrical conductivity ($1.68 \times 10^{-8} \, \Omega \cdot \text{m}$)[5, 6] and higher electromigration resistance rather than that of Al.[7] Therefore, interconnections formation using an effective Cu bonding technique is desired. Cu–Cu bonding has advantages for 3D IC technology compared with Cu solder bonding, which is widely used in present IC chips. In particular, Cu–Cu bonding can be applied to fine pitch bonding because it suppresses bump deformation, and Cu–Cu bonding provides bonds with improved electrical and mechanical performance compared with those produced by solder bonding.[8]

Cu–Cu bonding generally has been demonstrated using thermocompression bonding, which is achieved by atom interdiffusion and grain growth at the bonding interface induced via application of heat and pressure. Thermocompression bonding is promising for Cu–Cu bonding because of its high reliability, although Cu–Cu thermocompression bonding requires a high temperature of 350–400°C.[9, 10] A high temperature is required because a native oxide
layer present on the bonding Cu surfaces acts as a barrier against the interdiffusion of Cu atoms and also bulk Cu has a high melting point (1,083°C). Such a high bonding temperature brings challenges such as degradation of IC chips, bond-alignment accuracy, and thermal stress management; thus, many studies have been conducted with the aim of lowering the Cu–Cu bonding temperature. Suga’s group[11] performed Cu–Cu direct bonding using a Cu surface that was cleaned and activated via Ar-ion beam treatment to remove the surface Cu oxide layer; this process was coined surface-activated bonding. Liu and Chen[12, 13] used highly (111)-oriented nanotwinned Cu for Cu–Cu bonding because the Cu(111) surface has the fastest surface diffusivity of all the crystallographic planes and high oxidation resistance. Cu–Cu bonding with nanostructured Cu has also been proposed because size effects should enhance the surface melting of nanostructured Cu compared with that of bulk Cu.[8, 14] These methods are promising to lower the Cu–Cu bonding temperature, although ultra-high vacuum (UHV) or specific fabrication conditions are required. Other methods to lower the Cu–Cu bonding temperature have also been reported, including inserting a passivation layer (Ti,[15] Au,[16, 17] Cu nitride,[18] and a self-assembled monolayer of alkanethiol[19]) on the Cu surface to suppress its oxidation. However, a metal intermediate layer with a certain thickness leads to formation of Kirkendall voids, which lower the bonding strength, because of the difference between the atom diffusivities of Cu and the inserted metal layer.[20, 21] In addition, Kirkendall voids, inserted metal layers, and impurities may cause deterioration of the electrical properties and reliability of IC chips.

In this study, we report a Cu–Cu quasi-direct bonding method using a thin metal intermediate layer prepared by atomic layer deposition (ALD). The concept of this Cu–Cu quasi-direct bonding method is shown in Fig. 1. ALD can be used to deposit thin films suitable for Cu–Cu quasi-direct bonding because it allows exceptional control of film thickness at the angstrom level under low vacuum conditions. Such control is realized in ALD because atom deposition occurs layer by layer without UHV.[22, 23] To minimize the effect of the inserted metals at the bonding interface, a thin metal intermediate layer that disperses into Cu in low concentration is deposited. In addition, the thin metal layer is selectively deposited on the Cu surfaces without any masks using the high deposition selectivity of ALD.[24–27] The Cu-Cu quasi-direct bonding is performed without any surface activation method. Shear test results show that the thin metal intermediate layer of Pt deposited by ALD improves the Cu–Cu bonding strength by five times compared with that of the system without a thin Pt layer. Moreover, a mechanism for Cu–Cu quasi-direct bonding is proposed based on the atomic diffusivity of ALD-deposited Pt into Cu.

2. Experiment

2.1 Preparation of bonding samples

A planar Cu substrate (15 × 15 mm) and Si substrate (12 × 12 mm) with Cu bumps (diameter of 8 μm, pitch of 16 μm, 500 × 500 array) were prepared for the bonding test. To avoid burrs at their corners, the bumps were fabricated via a bilayer resist process (details of this process are shown in Supplementary Information S1).[28] A 1-μm-thick polydimethylglutarimide (MicroChem) sacrificial layer and 1-μm-thick photoresist (TSMR, Tokyo Oka Kogyo) layer were spin coated on the Si substrate and then annealed at 110 °C. After ultraviolet exposure to form bump patterns, the photoresist and sacrificial layers were etched simultaneously using NMD-3 developer (Tokyo Ohka Kogyo). The Cu layer was then annealed at 110 °C. After ultraviolet exposure to form bump patterns, the photoresist and sacrificial layers were etched simultaneously using NMD-3 developer (Tokyo Ohka Kogyo). The annealing temperature and etching conditions were optimized to form a re-entrant resist profile. Subsequently, a 500-nm-thick Cu layer and underlying...
30-nm-thick Ti adhesion layer were deposited by ion beam sputtering (IBS; M820, Hakuto) with an ion beam voltage of 950 V and current of 80 mA. Finally, the bilayer resist was stripped by soaking in Remover PG (MicroChem). A Si substrate with a 300-nm-thick planar Cu film was prepared in the same manner as a counter substrate.

2.2 Cu–Cu quasi-direct bonding

Pt was used as an intermediate layer in Cu–Cu quasi-direct bonding because Pt ALD is the most well-established metal ALD process.[23] The thin Pt layer was deposited using an ALD reactor (AT-400, Anric Technologies). The precursor and reductant for Pt ALD were trimethyl(methylcyclopentadienyl)platinum(IV) (Oakwood Chemical) and O₂ gas, respectively. Nitrogen gas (>99.9999%) was used as the carrier and purge gas during the ALD process. The Pt layer was formed based on the dissociative chemisorption of O₂ gas on the substrate surface for oxidative decomposition of the precursor ligands.[23, 25, 26] The Pt ALD process was performed layer by layer under 24 Pa via alternating exposure of the precursor and reductant gases for 100 cycles at 270 °C, which produced a Pt layer with a thickness of 20 Å.

The Cu samples with thin Pt layers were stored in ambient air and then Cu–Cu quasi-direct bonding was performed within two days of the Pt ALD. Two samples with thin Pt layers were bonded with a wafer bonder (SB6e, SUSS MicroTec) using a temperature of 300°C, applied pressure per chip of 8.3 MPa, bonding time of 15 min, and vacuum of <1 kPa. No surface treatment was performed before the bonding test. The bonding temperature of 300°C used here is lower than that required for Cu–Cu direct bonding with a native oxide layer reported previously.[9, 10] Details of the process are shown in Supplementary Information (Fig. S2). In addition, Cu substrates without a Pt layer and with a Pt layer on one side were also bonded as references.

2.3 Evaluation method

The shear strength of the bonded samples was evaluated using a digital shear strength indicator (DPX-50T, Imada). Three samples were measured for each set of conditions. Shear strength was calculated by dividing the measured value by the total Cu bump area. The surface of the bonded samples was observed using scanning electron microscopy (SEM; S8240, Hitachi). Surface roughness was evaluated by atomic force microscopy (SPM-9600, Shimadzu). Energy-dispersive X-ray spectroscopy (EDS; Octane Super, Ametek) was performed to verify the chemical composition of each Cu surface. The acceleration voltage for EDS analysis was 30 kV to efficiently excite heavy elements.

The diffusion of Pt into Cu was evaluated using glow discharge optical emission spectrometry (GDOES; JY-5000RF, Horiba). We prepared three types of samples with 20-nm-thick Pt layers: a Pt layer deposited by ALD on Cu with native oxide, a Pt layer deposited by IBS on Cu with native oxide, and a Pt layer deposited by IBS on Cu without native oxide. The sample without native oxide was prepared by depositing Cu and Pt layers continuously. The samples were annealed in ambient air at 200 or 300°C for 15 min before GDOES analysis.

3. Results and Discussion

SEM images of the Cu bumps are shown in Fig. 2. The height, top diameter, and bottom diameter of the Cu bumps measured from the SEM images are 501 nm, 7.6 μm, and 8.5 μm, respectively. A tapered edge structure was observed at the edge of the Cu bumps, as depicted in Fig. 2(b). These results indicated that the bilayer resist process allowed us to fabricate Cu bumps with an error of less than 6.3% from the designed value without any burrs, helping to prevent the formation of partial contacts.

The chemical compositions and surface morphologies of the samples were analyzed by EDS and SEM, respectively. Figure 3(a) and (b) show the EDS results for the Cu surfaces before and after Pt ALD. Strong peaks at 0.94, 1.73, 4.51, 8.03, and 8.94 eV originated from Cu, Si, and Ti in the prepared samples. A peak at 0.54 eV corresponded to oxygen originating from the Cu oxide layer and organic surface contaminants. After Pt ALD, peaks at 2.08 eV from Pt appeared. Pt particles were observed on the Cu surface after ALD (Fig. 4(b)), which were not observed on the Cu surface before ALD (Fig. 4(a)). These results indicated that the Pt layer was deposited as the particular shape on the Cu surface as previously reported via Volmer–Weber island growth model during the initial growth stages.[29–31] We also evaluated Pt ALD on an Si surface (Fig.
The Pt layer on the Si surface was not identified in EDS and SEM analyses, consistent with a previous report.[25, 26] This result further confirmed that a thin Pt layer can be selectively deposited on a Cu surface by ALD. Elam and Fang[24, 25] reported that the deposition selectivity of Pt is caused by differences of the chemisorption ability of oxygen on the substrate surface, leading reactivity differences of the precursor. Therefore, ALD can be used to deposit a Pt intermediate layer on fine-pitch Cu interconnections without using a mask. In addition, the arithmetic average surface roughnesses of the Cu layer before and after Pt ALD were 1.8 and 12.2 nm, respectively.

The effect of the thin Pt intermediate layer on Cu–Cu quasi-direct bonding was evaluated via shear tests, as shown in Fig. 5. The Cu–Cu bonded sample without an intermediate layer displayed a bonding strength of 1.9 MPa, whereas that of the sample with a thin Pt intermediate layer successfully improved bonding strength. In addition, the bonding strength of the sample with thin Pt intermediate layers on both sides (9.5 MPa) was higher than that of the sample with a thin Pt intermediate layer on one side (7.7 MPa). As shown in the SEM images of the bonding interface after the shear test (Fig. 6(c) and (d)), part of
the Cu layer was fractured in the samples with a thin Pt intermediate layer, meaning that the bonding strength in some regions of these interfaces was higher than that of bulk Cu. On the other hand, the small bonding area induced large variation in the sample with thin Pt intermediate layers on both sides.

We then evaluated the diffusivity of the deposited Pt into Cu to investigate the mechanism of Cu–Cu quasi-direct bonding. Samples with a Pt layer deposited on Cu by ALD or IBS were annealed at different temperatures (without annealing and with annealing at 200 or 300°C) to induce diffusion of Pt into Cu. The diffusion depth of the Pt atoms was detected by GDOES. The depth of Pt diffusion into Cu for the samples is shown in the Supplementary Information (Fig. S3). Figure 7 presents the Pt peaks detected for the samples exposed to different annealing temperatures. The sample with the Pt layer deposited by ALD showed deeper diffusion of Pt than that of the sample with the Pt layer deposited by IBS. Furthermore, the Pt diffusion depth into the ALD sample, in which the Pt layer was deposited over the native oxide layer, was almost the same as that of the IBS sample without the native oxide layer. This result suggests that Pt atoms slightly diffused into the Cu layer during the ALD process and a Pt–Cu interface may exist after ALD. From these results, we assumed that the thin Pt layer deposited by ALD had the following effects on Cu–Cu bonding: (i) the surface Pt layers, which have high oxidation resistance, promote initial bonding, and (ii) the Pt–Cu interface, which appeared during ALD process, accelerates atom interdiffusion. Therefore, the thin Pt layer deposited by ALD realized Cu–Cu quasi-direct bonding at a lower temperature than 350°C. The effect of the shape of the Pt layer on the bonding mechanism is currently under study.

4. Conclusions

We proposed a bonding method with a thin metal intermediate layer using ALD called Cu–Cu quasi-direct bonding. A thin Pt intermediate layer with an island structure was selectively deposited on a Cu surface by ALD under low vacuum conditions. The Cu–Cu bonding was successfully performed without any surface activation method, and the bonding strength was increased by five times when the Pt intermediate layer was present. Cu–Cu quasi-direct bonding with a shear strength of 9.5 MPa was achieved at 300°C, which is lower than the temperature needed for Cu–Cu direct bonding. Therefore, the developed bonding method using a metal intermediate layer prepared by ALD is promising for low-temperature Cu–Cu quasi-direct bonding in future electronic devices. This Cu–Cu quasi-direct bonding technique with a Pt intermediate layer cannot achieve complete destruction of bulk Cu; however, we believe that other metals that can be deposited at lower temperature than Pt and have high diffusivity into Cu can be used to help further improve Cu–Cu bonding quality.

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Fig. S1  Schematic of the bilayer resist process.
(i) A 1-μm-thick polydimethylglutarimide sacrificial layer and 1-μm-thick photoresist layer were spin coated on the Si substrate. (ii) After ultraviolet exposure to form bump patterns, the photoresist and sacrificial layers were etched simultaneously using developer. (iii) A 500-nm-thick Cu layer and underlying 30-nm-thick Ti adhesion layer were deposited by ion beam sputtering. (iv) The bilayer resist was stripped by soaking in a remover.

Fig. S2  Schematic diagram of the bonding process.
The sample was loaded in the bonding system, and then the chamber was evacuated under 1 kPa. After that, pressure and heat were applied to the sample. The sample was bonded at 300°C with applied force of 8.3 MPa for 15 min. Finally, the sample was unloaded after removing the applied pressure.
Figure S1 shows the GDOES results for Pt atom diffusion into Cu. We prepared three types of samples with 20-nm-thick Pt deposited layers: (a) a Pt layer deposited by IBS on Cu with native oxide, (b) a Pt layer deposited by IBS on Cu without native oxide, and (c) a Pt layer deposited by ALD on Cu with native oxide. The sample without native oxide was prepared by depositing the Cu and Pt layers continuously. The Pt atoms diffused into the Cu during annealing at 200 and 300°C. The sample with a Pt layer deposited by IBS on Cu without native oxide and that deposited by ALD with native oxide showed higher Pt atom diffusivity than that deposited by IBS on Cu with native oxide.
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