

# Room-Temperature Bonding of Wafers with Smooth Au Thin Films in Ambient Air Using a Surface-Activated Bonding Method

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**SUMMARY** Wafers with smooth Au thin films (rms surface roughness: < 0.5 nm, thickness: < 50 nm) were successfully bonded in ambient air at room temperature after an Ar radio frequency plasma activation process. The room temperature bonded glass wafers without any heat treatment showed a sufficiently high die-shear strength of 47–70 MPa. Transmission electron microscopy observations showed that direct bonding on the atomic scale was achieved. This surface-activated bonding method is expected to be a useful technique for future heterogeneous photonic integration.

**key words:** heterogeneous integration, room-temperature bonding, surface-activated bonding, Au-Au bonding

## 1. Introduction

The heterogeneous integration of materially different optical components made with wide ranges of fabrication processes onto a single platform enables us to construct small, high-performance, and multifunctional optoelectronic devices. Although traditional bonding techniques such as fusion bonding [1], thermo-compression bonding [2], and eutectic bonding [3] are widely used, they typically require high-temperature treatment (> 300°C), which generates serious problems such as defect diffusion, dopant diffusion, and the introduction of large thermal stresses caused by a mismatch in the coefficients of thermal expansion between dissimilar materials. Therefore, in recent years, low-temperature bonding techniques have become very important integration methods to create unique device structures for a wide range of photonic applications [4].

Among the various low-temperature bonding techniques, surface-activated bonding (SAB) [5] and atomic diffusion bonding (ADB) [6] methods are promising candidates for achieving room-temperature bonding. Here, we focus on intermediate layer bonding using gold (Au). Since Au has several highly desirable properties, such as oxidation and corrosion resistance, the Au–Au SAB method has been applied for the die bonding of laser diodes and photodiodes in ambient air [7]–[11] and the chip size packaging of optical microsystems [12]. In the case of the Au-Au ADB method, Au thin films were deposited on the wafers by magnetron sputtering in vacuum, and then the wafers

were bonded at room temperature in air [13], [14]. However, freshly vacuum-deposited Au thin films are contaminated spontaneously when they are exposed to ambient air or treated by chemical solutions. Surface contamination by carbon obstructs the Au-Au bonds at the interface and results in low bonding strength.

In this paper, the room-temperature bonding of wafers with smooth Au thin films was demonstrated using the SAB method.

## 2. Smooth Au Thin Films

### 2.1 Materials and Methods

A conventional electron beam (e-beam) evaporation system (ANELVA, EVC-400B) was used to deposit metal thin films in the present work. Titanium/gold (Ti/Au) films were deposited on commercially available silicon or synthetic quartz glass wafers (2 or 3 inches in diameter) at a substrate temperature of 25°C. Ti was used for improving the adhesion of Au on to the substrate. The pressure in the chamber during deposition was less than  $3 \times 10^{-4}$  Pa. Atomic force microscopy (AFM, SII nanotechnology, L-trace II) and scanning electron microscopy (SEM, JEOL JSM-7001F) were used to observe the surface morphology of the deposited films. The texture of the deposited films was characterized using electron back-scattered diffraction (EBSD) techniques by use of a JEOL JSM-7001F SEM with TSL OIM EBSD system. A transmission electron microscope (TEM, JEOL JEM-ARM200F) system was used to measure the thickness of the metal thin films.

### 2.2 Film Microstructure

Figure 1 (a) and (b) shows typical SEM and AFM images of the Ti/Au (50 nm/500 nm) film on a Si substrate, respectively. The grain structure was clearly observed in the as-deposited film. Figure 1 (c) shows an inverse pole figure obtained from EBSD analysis. The result shows that most of the grains on the as-deposited film have a <111> orientation. The measured root-mean-square (rms) surface roughness obtained from AFM was 3.6 nm. Most studies of Au-Au SAB using Au films with a surface roughness of a few nanometers showed that a high bonding pressure of 300 MPa was required for the interatomic attraction to overcome surface asperities, and this makes it difficult to achieve

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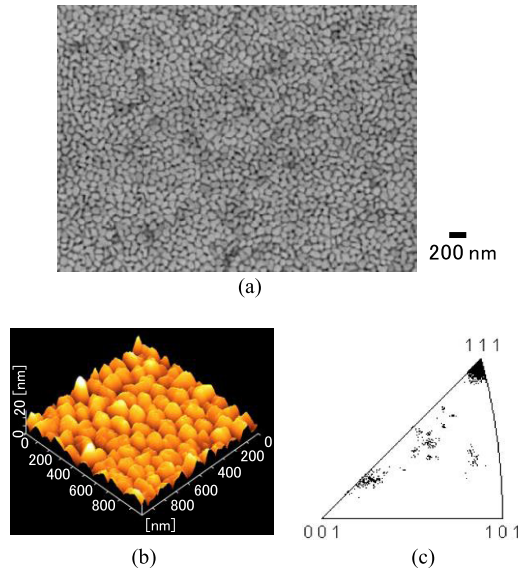
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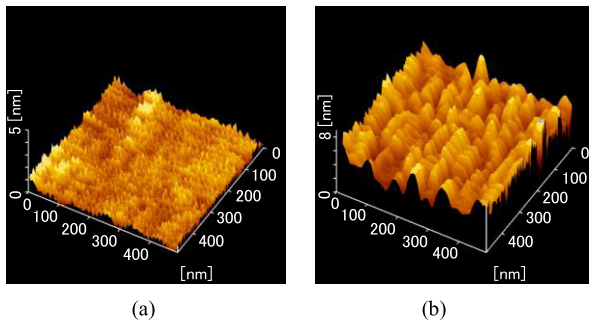
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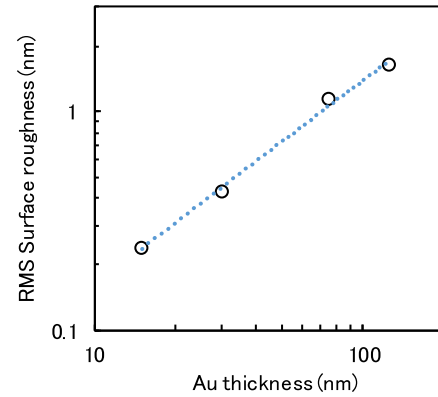
**Fig. 1** Surface morphology and inverse pole figure of Au films prepared by e-beam evaporation with a film thickness of 500 nm. (a) SEM image, (b) AFM image (rms roughness: 3.6 nm), (c) inverse pole figure obtained using EBSD analysis.



**Fig. 2** AFM images of Au thin films prepared by e-beam evaporation. (a) Film thickness: 15 nm, rms roughness: 0.24 nm, (b) film thickness: 75 nm, rms roughness: 1.14 nm.

a wafer-scale bonding process without a high bonding pressure [7], [12].

The present paper focuses on the use of Au thin films with a film thickness of below 50 nm. To study the dependences of the structural and morphological properties on the film thickness, a series of films with an Au thickness of 15 nm~125 nm were prepared on glass wafers. The rms surface roughness of the bare glass wafers was 0.19 nm. Figure 2 shows AFM images of the Ti/Au (3 nm/15 nm) and Ti/Au (3 nm/75 nm) films on the glass substrates. These images clearly show that the grain size increases with the increase in the film thickness. Figure 3 shows the rms surface roughness of the Au thin films as a function of the film thickness. The surface roughness decreases with the reduction of thickness, and a surface roughness below 0.5 nm was obtained when the film thickness was below 50 nm. Surface roughness values below 0.5 nm were smooth enough for direct Au-Au bonding.



**Fig. 3** Surface roughness of Au thin films as a function of film thickness.

The influence of the Ar plasma treatment on the surface roughness and thickness of the Au thin films was studied. The rms roughness was 0.42 nm for the Ti/Au (3 nm/30 nm) film on the glass substrate treated with Ar radio frequency (RF) plasma for 30 s (100 W), compared with 0.39 nm for the non-treated sample, indicating that the Ar plasma treatment does not significantly deteriorate the surface roughness. The change in thickness of the Au thin films between before and after the plasma treatment was typically only a few nanometers from TEM observation.

### 3. Room-Temperature Wafer Bonding

#### 3.1 Experimental Procedures

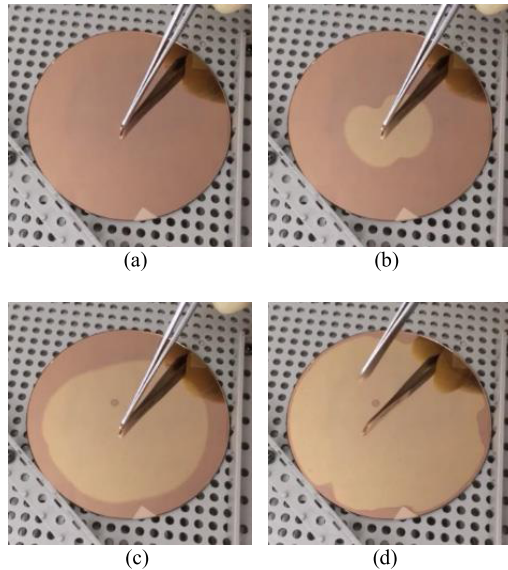
Bonding was based on Au-Au SAB at room temperature in ambient air. The bonding procedure is as follows. Prior to bonding, surface activation using Ar radio RF plasma treatment (100 W, 30 s) is first carried out to remove organic contamination from the Au surfaces and obtain active surfaces. Then, the two wafers are brought into contact manually within 5 minutes at room temperature in ambient air. No additional annealing is applied to the samples.

The influence of the air exposure time after Au deposition on the bonding strength in Au-Au bonding was investigated. After the Au evaporation, the wafer pairs were stored in air and a particle-free environment (temperature: 20–25°C, humidity: 10–15%) for different air exposure times from 10 minutes to 2000 hours. Au-Au bonding with and without Ar RF plasma treatment was performed for comparison.

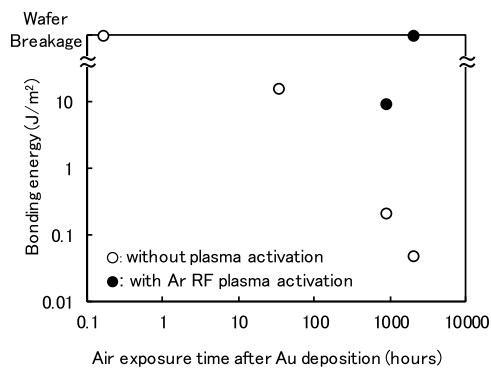
After bonding, the bonding strength was measured by a razor blade test (crack-opening method) [15]. In this test, a razor blade is inserted at the interface and the crack length along the bonded interface is observed through the glass wafer. The bonding energy  $\gamma$  [J/m<sup>2</sup>] (or surface energy) of the bonded pairs, which indicates the bonding strength, is given by

$$\gamma = \frac{3}{32} \cdot \frac{ET_b^2 T_w^3}{L^4} \quad (1)$$

where  $E$  (Pa) is the Young's modulus of the wafer,  $T_b$  (m)



**Fig. 4** Initiation and self-propagation of bonded area in glass/glass wafer bonding with Ti/Au (3 nm/30 nm) thin films (3 inches in diameter). Bright areas correspond to bonded areas. (a) 0.0 s, (b) 0.2 s, (c) 0.4 s, (d) 0.6 s.



**Fig. 5** Bonding energy as a function of the air exposure time after Au deposition [16]. Copyright (2016) The Japan Institute of Electronics Packaging

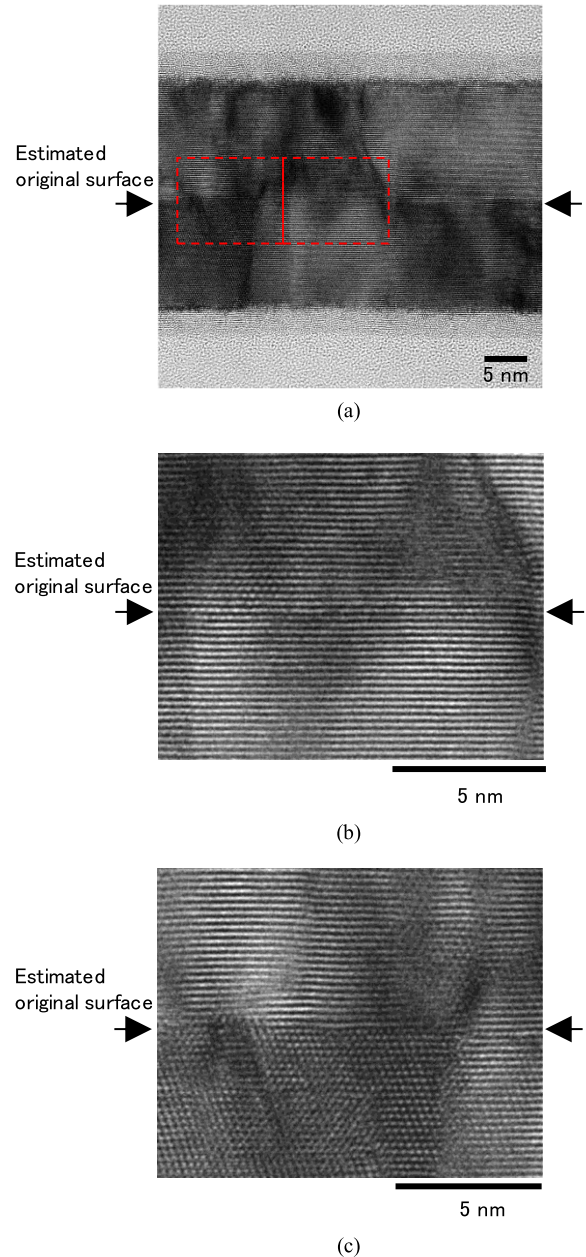
is the blade thickness,  $T_w$  (m) is the wafer thickness, and  $L$  (m) is the crack length.

Die shear testing for bonding strength measurements was also performed with a die shear tester (Rhesca PTR-1100). The bonded wafers were diced into chips of  $3 \times 3$  mm in size for die shear testing.

The microstructure of the bonded interface was observed using a TEM system (JEOL JEM-ARM200F).

### 3.2 Bonding Results

When placing two wafers with the Au sides facing each other, a thin air layer is usually trapped between them, and this prevents immediate spontaneous bonding. Therefore, a light pressure was applied locally on the top wafer to create the first contact point. In the case of bonding with Ar RF plasma treatment, the bonding front spread immediately after pushing the center of the top wafer, regardless of the long-term air exposure (800 and 2000 h). Figure 4 shows



**Fig. 6** Cross-sectional TEM images of Au-Au bonded interface prepared by SAB method at room temperature in ambient air. (a) Low-magnification image, (b) (c) high-magnification image.

a typical example of a bonding front propagation observation. The bonded area expanded across the entire wafer area within 1 s. The bonding process can be simply monitored by observing the color change of the wafers owing to the thickness-dependent optical transparency of the Au thin films as the surfaces are brought together. With the exception of the peripheral areas, the entire area was bonded with few voids.

Figure 5 shows experimental results of the bonding energy as a function of the air exposure time after Au deposition [16]. The wafers with Ti/Au (3 nm/30 nm) thin films on glass wafers were used for the experiments. The surface

energy of Au is  $1.63 \text{ J/mm}^2$  [17]. In the case of bonding without Ar plasma treatment, the bonding energy decreased with the increasing air exposure time. This result is consistent with previously reported results [14]. In the case of an air exposure time of 800 or 2000 hours, the bonded area did not expand by itself across the entire wafer area, and the bonding strength was too low to withstand a dicing process. On the other hand, the Ar plasma treatment increased the bonding energy significantly.

Bonded wafers with a high bonding energy showed a high die-shear strength (47–70 MPa), and the fractures typically occurred within the bulk of the glass.

Figure 6 shows cross-sectional TEM images of the Au (thickness: 15 nm)–Au (thickness: 15 nm) bonded interface with the Ar plasma treatment. The TEM observations showed that even in room temperature bonding in air, a bonded interface at the atomic level was achieved.

Based on these experimental results, the bonding of GaAs and SiC wafers (2 inch in diameter) with Ti/Au (3 nm/30 nm) films was also successfully achieved at room temperature in air. The die shear test showed that the fractures typically occurred within the bulk GaAs rather than at the bonded interface. The wafer-scale GaAs/SiC hetero structure is expected to enable efficient heat dissipation in high-power semiconductor laser applications [18].

#### 4. Conclusions

Room-temperature wafer bonding using smooth Au thin films was demonstrated in air. Without plasma treatment, the bonding strength decreased with the increasing air exposure time after Au deposition. Ar RF plasma pretreatment improved the bonding energy significantly, regardless of the air exposure time. The cross-sectional TEM images of the Au–Au bonded interface showed that bonding occurred directly at the atomic level even at room temperature in ambient air. These results suggest that this technique will be useful for future wafer-scale heterogeneous optoelectronic integration.

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#### References

- [1] Z.L. Liao and D.E. Mull, "Wafer fusion: A novel technique for optoelectronic device fabrication and monolithic integration," *Appl. Phys. Lett.*, vol.58, no.8, pp.737–739, 1990.
- [2] D. Andrijasevic, M. Austerer, A.M. Andrews, P. Klang, W. Schrenk, and G. Strasser, "Hybrid integration of GaAs quantum cascade lasers with Si substrates by thermocompression bonding," *Appl. Phys. Lett.*, vol.92, no.5, 051117, 2008.
- [3] E. Higurashi, D. Chino, and T. Suga, "Residue-free solder bumping using small AuSn particles by hydrogen radicals," *IEICE Trans. Electron.*, vol.E92-C, no.2, pp.247–251, 2009.
- [4] E. Higurashi, "Low-Temperature Bonding Technologies for Photonics Applications," *ECS Transactions*, vol.50, no.7, Semiconduc-

- tor Wafer Bonding 12: Science, Technology, and Applications, pp.351–362, 2012.
- [5] T. Suga, Y. Takahashi, H. Takagi, B. Gibbesch, and G. Ellsner, "Structure of Al–Al and Al–Si<sub>3</sub>N<sub>4</sub> interfaces bonded at room temperature by means of the surface activation method," *Acta Metallurgica et Materialia*, vol.40, pp.S133–S137, 1992.
- [6] T. Shimatsu, R.H. Mollema, D. Monmsma, E.G. Keim, and J.C. Lodder, "Metal bonding during sputter film deposition," *J. Vac. Sci. Technol. A*, vol.16, no.4, pp.2125–2131, 1998.
- [7] E. Higurashi, T. Imamura, T. Suga, and R. Sawada, "Low temperature bonding of laser diode chips on Si substrates using plasma activation of Au films," *IEEE Photon. Tech. Lett.*, vol.19, no.24, pp.1994–1996, 2007.
- [8] E. Higurashi, D. Chino, T. Suga, and R. Sawada, "Au–Au Surface-Activated Bonding and Its Application to Optical Microsensors with 3-D Structure," *IEEE J. Sel. Topics Quantum Electron.*, vol.15, no.5, pp.1500–1505, 2009.
- [9] R. Takigawa, E. Higurashi, T. Suga, and R. Sawada, "Room-Temperature Bonding of Vertical-Cavity Surface-Emitting Laser Chips on Si Substrates Using Au Microbumps in Ambient Air," *Applied Physics Express*, vol.1, no.11, 112201, 2008.
- [10] T. Sato, E. Higurashi, T. Sato, and R. Sawada, "Low-temperature bonding of laser diode chips using Au stud bumps," *IEICE Trans. Electron.* (Japanese Edition), vol.J94-C, no.11, pp.470–471, Nov. 2011.
- [11] E. Higurashi, M. Yamamoto, T. Sato, T. Suga, and R. Sawada, "Room-temperature gold-gold bonding method based on argon and hydrogen gas mixture atmospheric-pressure plasma treatment for optoelectronic devices integration," *IEICE Trans. Electron.*, vol.E99-C, no.3, pp.339–345, 2016.
- [12] S. Yamamoto, E. Higurashi, T. Suga, and R. Sawada, "Low-temperature hermetic packaging for microsystems using Au–Au surface-activated bonding at atmospheric pressure environment," *J. Micromech. Microeng.*, vol.22, no.5, 055026, 2012.
- [13] T. Shimatsu, M. Uomoto, K. Oba, and Y. Furukata, "Atomic Diffusion Bonding of Wafers in Air with Thin Au Films and Its Application to Optical Devices Fabrication," *Proc. 2012 3rd IEEE International Workshop on Low Temperature Bonding for 3D Integration (LTB-3D)*, p.103, Tokyo, Japan, May 22–23, 2012.
- [14] H. Kon, M. Uomoto, and T. Shimatsu, "Room temperature bonding of wafers using Au films with various holding times in air," *Journal of The Japan Institute of Electronics Packaging*, vol.17, no.5, pp.431–435, 2014 (in Japanese).
- [15] M.P. Maszara, G. Goetz, A. Cavigila, and J.B. McKitterick, "Bonding of silicon wafers for silicon-on-insulator," *J. Appl. Phys.*, vol.64, no.10, pp.4943–4950, 1988.
- [16] Y. Kunimune, K. Okumura, E. Higurashi, T. Suga, and K. Hagiwara, "Room-temperature wafer bonding using smooth gold thin films for wafer-level MEMS packaging," *2016 International Conference on Electronics Packaging (ICEP)*, pp.439–442, Sapporo, Japan, April 20–22, 2016.
- [17] L.Z. Mezey and J. Gibber, "The Surface Free Energies of Solid Chemical Elements: Calculation from Internal Free Enthalpies of Atomization," *Jpn. J. Appl. Phys.*, vol.21, pp.1569–1571, 1982.
- [18] E. Higurashi, K. Okumura, K. Nakasuji, and T. Suga, "Surface activated bonding of GaAs and SiC wafers at room temperature for improved heat dissipation in high-power semiconductor lasers," *Japanese Journal of Applied Physics*, vol.54, no.3, 030207, 2015.



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