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Investigation on the Gold-Wire Bondability for the IC Device

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In order to develop a microstructure observing technique for wire-bonded interfaces, which is important in the IC industry, thin films of wire-bonded gold-aluminum samples were prepared by ion milling and microtome techniques and observed by transmission electron microscopy (TEM). The effect of organic contamination for wire-bonding and the dependence of wire-bondability on the thickness of plated gold film and the plating method were also investigated by TEM and shear tests. The results obtained are as follows.

(1) In the films prepared with a microtome no amorphous phase is observed, whereas in the films prepared by ion milling a large part of the intermetallic compound formed on the interface has become amorphous. (2) At the whole interface of the cleaned samples the intermetallic-compound is observed, whereas at the interface of the contaminated samples an organic contamination layer is observed instead of the intermetallic-compound. The cleaned samples are generally higher in shear strength than the contaminated samples. (3) Voids are produced by heating along the gold-intermetallic compound interface. The voids deteriorate the bond strength. (4) Without heat treatment the bond strength does not depend on either the gold-film thickness or the plating method. (5) The nickel (from the substrate) concentration at the film surface increases with increasing heating period, and as a result the bond strength decreases. (6) The bond strength of samples bonded after heat treatment recovers when a surface layer of the gold film is removed before wire-bonding.

From these results, a suitable technique for TEM observation of the wire-bonded interface and the effects of contamination on wire-bondability are discussed.

1. Introduction

The wire-bonding is the most popular method for joining an electrode in an IC device (or chip) with that in a circuit board.

A gold wire (20~30 μm in diameter) is passed through a press tool called 'capillary' and the end is melted by electric spark. Then a ball is formed at the end by surface tension. The ball is bonded by pressing to an aluminum electrode called 'pad' (1~2 μm in thickness) in an IC device or to a gold-plated electrode in an circuit board heated to a temperature in a range from 350 K to 400 K. The capillary is vibrated ultrasonically during the bonding for improving the bondability. In the case of aluminum electrode, the surface oxide layer of the electrode is broken by plastic deformation. Thus the newly yielded active surface of the electrode can be well bonded with the wire¹⁾.

Since the wire-bonding is the key technology for IC assembling, many investigations concerning the bonding mechanism have been reported²⁾⁻⁸⁾. Cold-welded bulk samples have been used for those fundamental investigations and examined by means of X-ray diffraction and electron probe microanalyser (EPMA). The size of the electrode for wire-bonding is usually 100 μm or less in diameter at present and becoming finer and finer.

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Since the area needed for the X-ray diffraction or EPMA is too large to study the very fine wire-bonded interface, it is necessary to establish another technique to characterize the very fine interface. TEM may be the most suitable technique for this purpose. However, it is difficult to prepare a film for the TEM observation, because the bond is fragile and has a multi-layer structure. As far as the authors know it has never been reported concerning the high-resolution TEM observation of wire-bonded interface.

The organic contamination of the electrode surface due to the out-gas from the conductive bond which is used in the IC assembly process may reduce the bond-strength and increase the electric resistance. This is another problem to be settled. The effect of aluminum oxide on the gold-aluminum wire-bonding has been reported⁹⁾. However, the effect of organic contamination has never been reported. In this study, the effect was also investigated by TEM and shear test.

A gold film plated on a nickel-plated copper film is used as an electrode for a circuit board. As the film is made thinner it is more advantageous in terms of cost¹⁰⁾. The electroless plating is better than electroplating currently used for the plating^{11)–12)}.

In the current techniques, the thickness of the plated film is empirically recommended to be more than $0.3\ \mu\text{m}$. However, the reasons why the plating method should be electroplating and the film thickness should be more than $0.3\ \mu\text{m}$ have never been reported.

Therefore we further tried to make clear the dependence of wire-bondability on the film-thickness and the film plating method, and establish the plating conditions most suitable for the wire-bonding.

2. Experimental

2.1. Silicon chips with aluminum electrodes

Silicon chips were used as the substrates for the bonding. The substrate had aluminum electrodes formed by means of evaporation.

In the IC assembling, chips are joined to the circuit board with epoxy adhesive. Then the joined assembly is heat-treated for curing of the adhesive. Considering this practical process, the chips were heat-treated in the out-gas atmosphere from the adhesive at 423 K for 3.6 ks. The chemical composition of the out-gas analysed by means of gas-chromatography is shown in **Table 1**.

The effect of cleaning the surface on the bondability was also investigated. The contamination layer was removed by an argon plasma-etcher (PC 10 FG type: Kyushu Matsushita Electric Co., Ltd.). The flux of argon gas was set to $4.2\ \mu\text{m}^3/\text{s}$, output of radio frequency power 500 W, the etching period 60 s, and the temperature 383 K.

2.2. Circuit boards with gold plated electrodes

A copper film (of thickness $35\ \mu\text{m}$) laminated on the circuit board was used as a substrate for gold plating. The substrate was plated with nickel by using the deoxidation plating method. The plated nickel film is useful as the diffusion barrier for the underlying copper into the uppermost gold film to be described¹³⁾.

The nickel plated substrates were further plated with gold. Three different plating methods were used; substitution plating, deoxidation plating and electroplating. In the substitution plating, gold (nobler metal) is precipitated by dissolving nickel (baser metal).

Table 1 Gas chromatography of out gas from epoxy paste.

		out gas weight/sample weight (%)		
		573K × 180s	603K × 180s	623K × 180s
Phenol	C ₆ H ₅ OH	0.02	0.6	0.9
Mono epoxy	OCH ₂ CHOCH ₂	2.3	2.6	2.8
Acrolein	CH ₂ CHCHO	0.0018	0.0022	0.0029
Butyl aldehyde	CH ₃ CH ₂ CH ₂ CHO	0.0050	0.0027	0.0019
Methyl ethyl ketone	CH ₃ COC ₂ H ₅	0.0033	0.0030	0.0030
Ethanol	C ₂ H ₅ OH	—	—	0.0001
Toluene	C ₆ H ₅ CH ₃	0.0001	0.0002	0.0004
Xylene	C ₆ H ₄ (CH ₃) ₂	0.0001	0.0080	0.01
Carbon monoxide	CO	—	0.01	0.01
Carbon dioxide	CO ₂	0.16	0.24	0.37
Ammonia	NH ₃	0.02	0.03	0.04
Water	H ₂ O	0.09	0.20	0.33
total		2.6	3.7	4.5

In this method, however, it is very difficult to plate a thick film, because the reaction is driven by the difference in electronegativity of metals and the reaction stops when all the surface is substituted by the nobler metal. Accordingly, films of various thicknesses could not be plated, and only one kind of film of thickness 0.05 μm could be prepared.

On the other hand, by using the de-oxidation plating or electroplating, various film-thicknesses could be plated. Since one objective of this study was to observe how wire-bondability depends on the film-thickness, gold films with three different thicknesses, 0.15 μm , 0.5 μm and 0.8 μm , were intended to prepare. Film-thicknesses measured by means of X-ray film-thickness gauge (SFT7000 type; Seiko Instruments Inc.) are shown in **Table 2**.

2.3. Bonding test

A 99.99% pure gold wire 25 μm in diameter was used for wire-bonding. An automatic thermosonic wire-bonder (HW 21 type; Kyushu Matsushita Electric Co., Ltd.) was used for the wire-bonding. An ultrasonic power of 55 mW was used, and a bonding pressure of 80 MPa was applied for a period of 20 ms at a temperature of 383 K.

Table 2 Thickness of gold film plated using three different methods¹⁸⁾.

Method	Thickness, $t/\mu\text{m}$
Electroplating	0.17
	0.50
	0.87
Substitution plating	0.07
Deoxidation plating	0.16
	0.49
	0.99

After the wire-bonding, the bond strength was measured by means of micro-shear tester (PTR 100S type; Rhesca Co., Ltd.). The shear strength values were considered as a measure of the wire-bondability.

Cold-welded bulk samples were also investigated to compare with the wire-bonded samples. Both the aluminum and gold samples were cylindrical, 2.8 mm in diameter and 99.99% purity. They were joined by compression at room temperature by cold-welder (KBM-5 type; Asahi Kinzoku Seiko Co., Ltd.).

2.4. TEM observation

Two techniques were used to prepare films for TEM observation. One is an ion milling technique and the other a microtome technique. The ion milling was conducted after mechanical polishing to about 50 μm thick, the acceleration voltage was set to 4 kV, the current 0.4 mA, and the tilt angle 15 degree. For the microtome technique, a microtome (ultracut S type; Nissei Sangyo Co., Ltd.) was used. The thickness of the film prepared with the microtome was about 15 nm. The films were observed by TEM at 400 kV and 200 kV using an electron microscope of JEM-4000EX type (JEOL Co., Ltd.).

2.5. Heat-treatment

The contaminated chips and cleaned chips were heat-treated at 573 K for various periods from 7.2 ks to 172.8 ks after the wire-bonding to investigate the effect of heating on the bond strength. After the heat-treatments, the growth of intermetallic-compounds was observed on a cross section by scanning electron microscopy (SEM) (T-300 type; JEOL Co., Ltd.) and the bond strength was measured by the shear tester.

Considering the practical process, samples with gold film were heat-treated in air at 473 K for various periods from 0 to 7.2 ks and then wire-bonded to observe the changes in bond strength with the heat-treatment¹⁴⁾. After the heat-treatment, the samples were analyzed by Auger electron spectroscopy (AES) (650 type; ULVAC-PHI INC.) and electron spectroscopy for chemical analysis (ESCA) (850 type; Shimazu Co., Ltd.).

Cold-welded bulk samples were heat-treated in air at various temperatures from 473 K to 733 K for various periods from 7.2 ks to 90 ks and then analyzed by means of EMPA (S-650 type; Hitachi Ltd.) to investigate how the intermetallic-compounds at the interface changes with the heat-treatment. The residual stress near the cold-welded interface was measured by position sensitive proportional counter (Rigaku Denki Ltd.) as a function of heat-treatment time.

3. Results and Discussion

3.1. TEM observations

Figure 1 shows a high resolution TEM image of the wire-bonded interface in a film thinned by the ion milling. A large part of the intermetallic-compound is seen to be amorphous.

Figure 2 is a high resolution TEM image of the cold-welded interface. The specimen was thinned by the ion milling. Amorphous regions are again observed. These observations suggest that the amorphous phase should not be particular to the wire-bonded interface, but general.

Figure 3 is a high resolution TEM image of a wire-bonded interface observed in a film

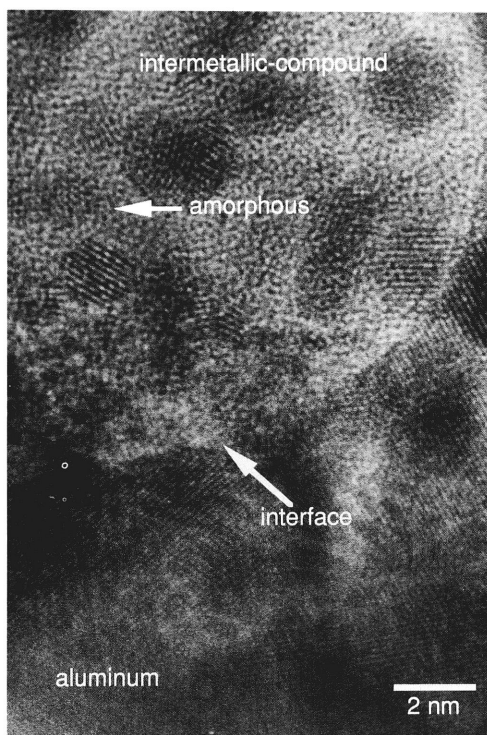


Fig. 1 High resolution TEM micrograph of a gold-aluminum wire bonded interface. The specimen was thinned by ion milling¹⁷⁾.

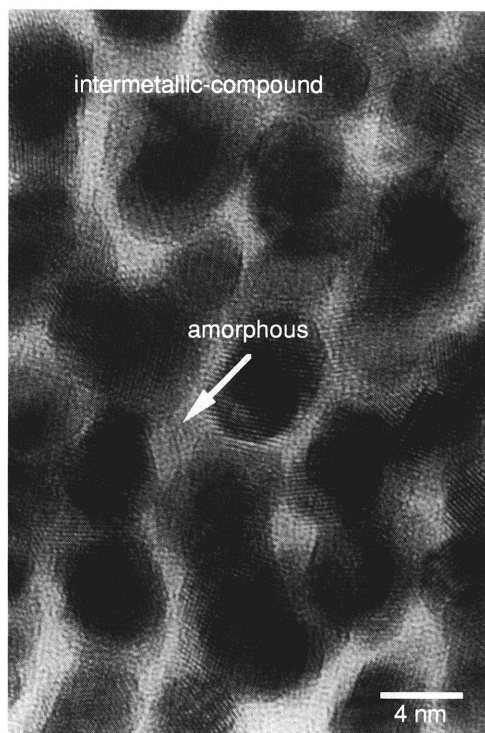


Fig. 2 High resolution TEM micrograph of a gold-aluminum cold welded interface. The specimen was thinned by ion milling.

prepared with the microtome. In this case, no amorphous phase is observed. From **Figs. 1, 2** and **3**, it is clear that the amorphous phase is formed by the ion milling.

The wire-bonded samples consisted of four components of gold ball of $30\ \mu\text{m}$, aluminum electrode of $1\ \mu\text{m}$, silicon oxide layer of $0.6\ \mu\text{m}$ and silicon chip of $300\ \mu\text{m}$ in thickness. It was difficult to obtain a sufficiently flat and thin film for TEM observation by ion milling. Further, the ion milling changes the crystalline intermetallic-compounds to amorphous. Consequently, the microtome method is more suitable technique to prepare a film for TEM observation of the bonded interface.

3.2. Effect of organic contamination on wire bonding

Figure 4 shows fluorescence microscopic images of the aluminum electrodes for (a) contaminated and (b) cleaned samples, respectively. Luminesced regions are observed on the contaminated sample, whereas no luminesced region is seen on the plasma-etched electrode.

Figure 5 shows the composition change determined by the Auger analysis as a function of depth from the electrode surface for (a) contaminated and (b) cleaned samples. **Figures 4** and **5** show that the organic contamination is removed sufficiently by the plasma-etching.

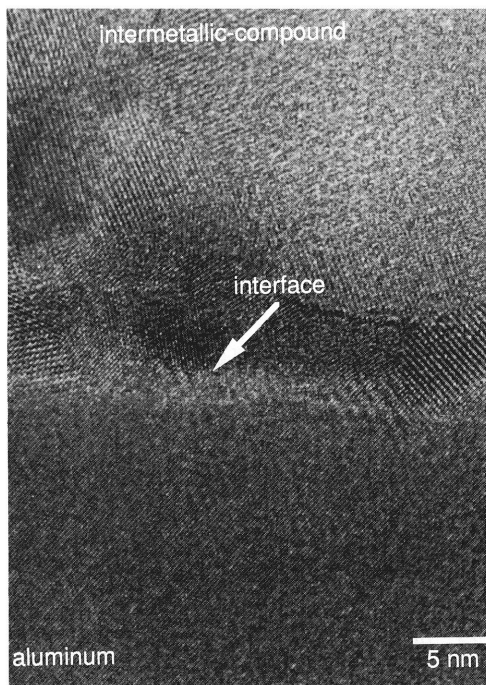


Fig. 3 High resolution TEM micrograph of a wire-bonded interface. The specimen was prepared by the microtome technique¹⁷⁾.

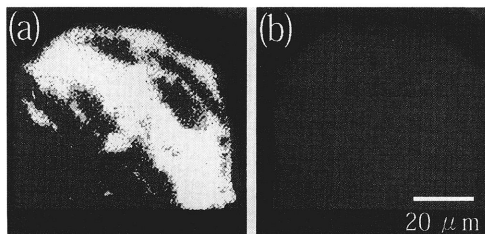


Fig. 4 Fluorescence micrograph of the aluminum electrode surface, (a) contaminated sample and (b) cleaned sample.

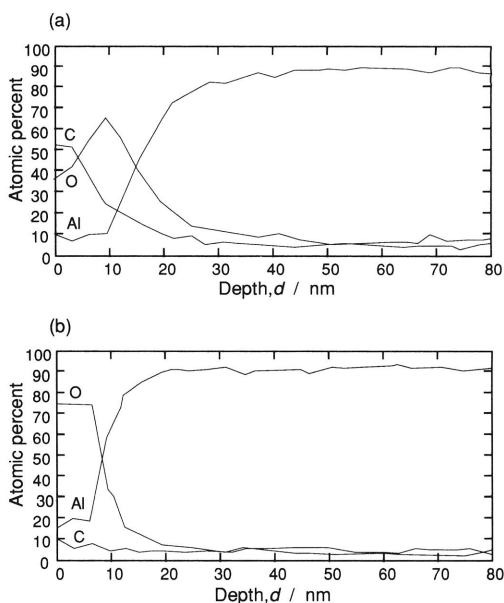


Fig. 5 Auger electron spectroscopy as a function of depth, (a) contaminated aluminum electrode and (b) cleaned aluminum electrode¹⁴⁾.

Figure 6 (a) shows a SEM micrograph of a wire-bonded interface, where the aluminum electrode was cleaned by plasma-etching. An intermetallic-compound layer is seen along the interface. **Figures. 6**

(c) and **(d)** show high resolution TEM micrographs at the regions indicated 'c' and 'd' in **Fig. 6 (a)**, respectively. **Figure 6 (c)** shows the interface between the gold and the intermetallic-compound and **Fig. 6 (d)** shows that between the intermetallic-compound and the aluminum electrode. Fine grains (about 10 nm in diameter) of intermetallic-compound are seen at the interface. **Figures 6 (a), (c)** and **(d)** suggest that the active surfaces of the gold ball and aluminum electrode should have contacted with each other when removed the organic contamination layer.

Figure 6 (b) shows a SEM micrograph of the wire-bonded interface, where the aluminum electrode was contaminated. No intermetallic-compound layer is seen. **Figure 6 (e)** shows a high resolution TEM micrograph at the region indicated 'e' in **Fig. 6 (b)**. A foreign amorphous layer (about 15 nm in thickness) is seen along the interface. The

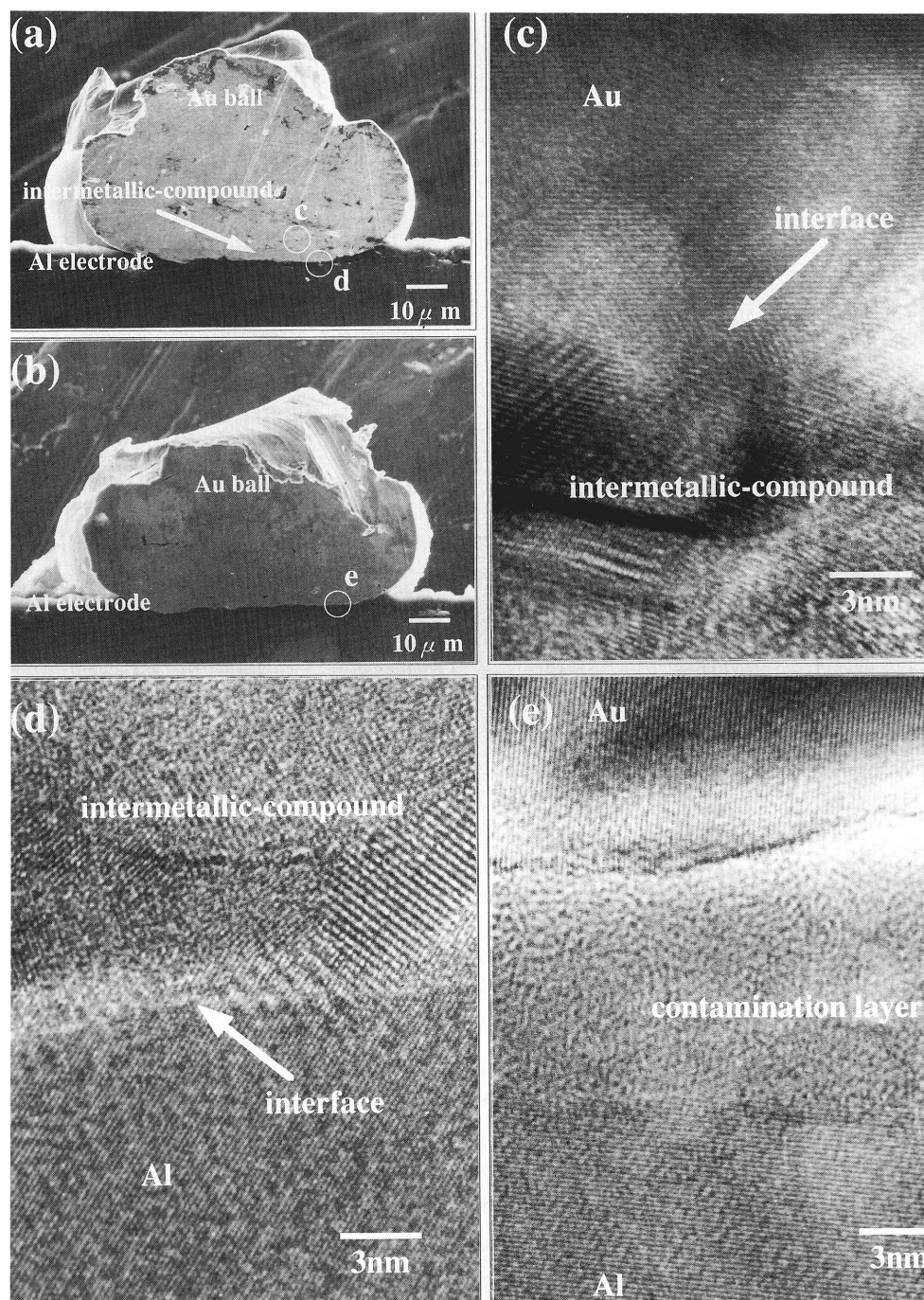


Fig. 6 SEM micrographs of wire bonded interfaces; (a) cleaned and (b) contaminated sample. High resolution TEM micrographs: (c) gold-intermetallic compound interface of cleaned sample, (d) intermetallic compound-aluminum interface of cleaned sample and (e) gold-aluminum interface of contaminated sample¹⁴⁾.

results in **Figs. 5 (a)** and **6 (e)** suggest that the layer should be an organic contamination layer. Since the contamination layer existed between the gold ball and the aluminum electrode, the two metals could not contact and no intermetallic-compound was formed at the interface. Because the contamination layer behaves like a lubricant, the aluminum oxide layer at the surface of the electrode should hardly be broken and active surfaces should not be formed sufficiently during bonding operation.

Figure 7 shows the effect of heating on the bond-strength for contaminated and cleaned samples at 573 K. Both samples have similar features for the change in bond-strength due to heating, provided that the cleaned samples are generally higher in bond-strength than the contaminated ones. **Figure 8** shows SEM micrographs of the wire-bonded interface as a function of heating time at 573 K. The results in **Figs. 7** and **8** suggest that the spreading of the intermetallic-compound area due to heating causes the initial increase in bond-strength, but the smaller area of the intermetallic-compound in the contaminated samples causes a lower bond-strength than that of the cleaned samples. Even after a long period heating, interfaces without intermetallic-compound were observed in some places of the contaminated samples.

Voids were produced by heating along the gold-intermetallic compound interface in both samples. After a long period heating, the voids grew to cracks.

Figure 9 shows an EPMA line analysis of the cold-welded region after heat-treatment at 723 K for 86.4 ks. The three phases are observed and the Al_2Au_5 phase is predominant. If the growth of the intermetallic-compound is controlled by diffusion, the following equation holds¹⁵⁾.

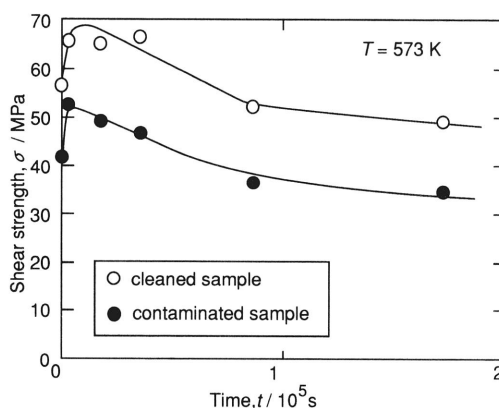


Fig. 7 The relation between shear strength and heat treatment time¹⁴⁾.

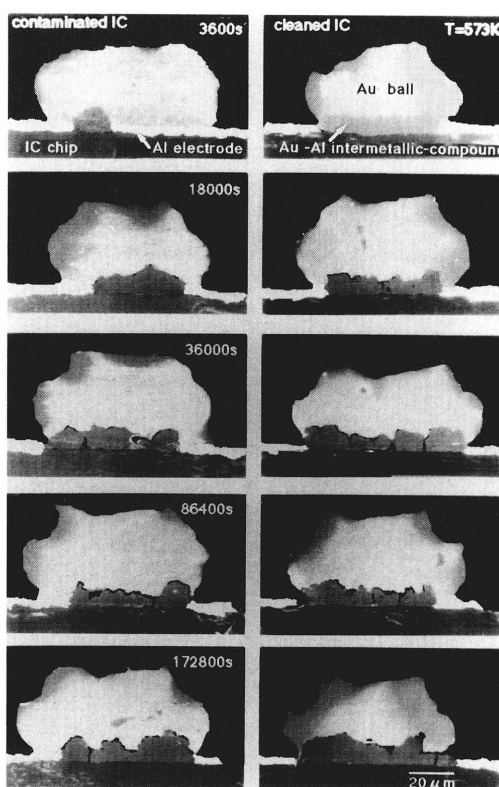


Fig. 8 SEM micrographs of the wire bonded interface, showing the heating effect on the interface structure.

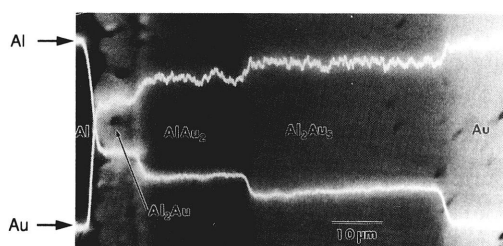


Fig. 9 EPMA of a bonded interface after heat treatment at 723 K for 86.4 ks¹⁷⁾.

$$X = k \sqrt{t} = \alpha \sqrt{t \cdot \exp(-Q/RT)} \quad (1)$$

Where X is the thickness of the intermetallic-compound, α a constant, t the period of the heat-treatment, Q the activation energy, R the gas constant, and T the absolute temperature. The relation between $k^2 (= X^2/t)$ and $1/T$ for the growth of the intermetallic compound is shown in Fig. 10. The relation indicates the Arrhenius type temperature-dependence. The activation energy estimated from the slope is 81.7 kJ/mol and is almost equal to 85.4 kJ/mol reported by Ohnishi¹⁶⁾.

The relation between X and $\sqrt{t \cdot \exp(-Q/RT)}$ for $Q=81.7$ kJ/mol is shown in Fig. 11 (a). The datum point for the sample heat-treated at 673 K for 90 ks deviates largely from the liner relation. The bonded interface of that sample was fragile after the

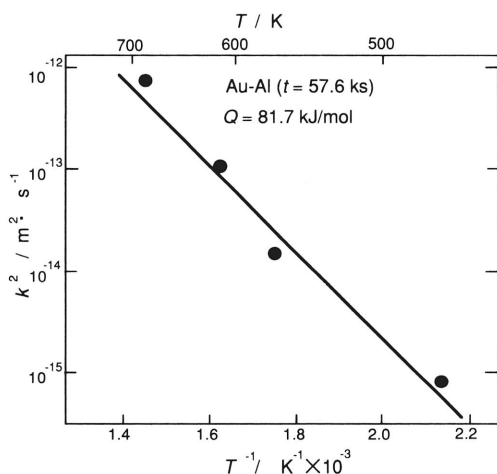


Fig. 10 $k^2 (= X^2/t)$ vs $1/T$ curve for the growth of the intermetallic-compound.

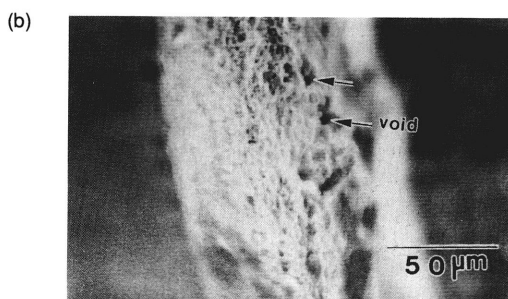
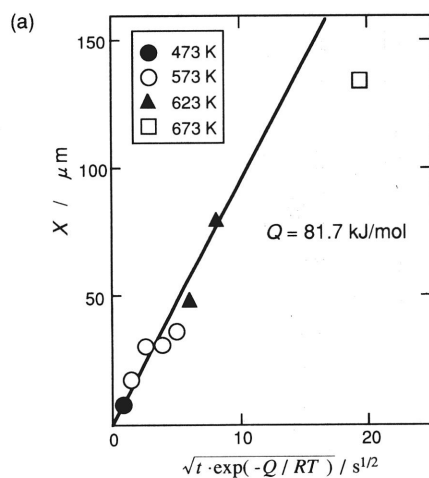


Fig. 11 (a) X vs $\sqrt{t \cdot \exp(-Q/RT)}$ curve for the growth of Al_2Au_3 phase. (b) Fracture surface at a gold-intermetallic compound interface in a bonded bulk material after annealing at 673 K for 90 ks¹⁷⁾.

heat-treatment. A SEM micrograph of a fractured interface is shown in **Fig. 11 (b)**, where many voids are observed. These voids must have obstructed diffusion. The large deviation should arise from the void formation.

Residual stresses measured in aluminum and gold phases near the cold-welded interface are shown in **Table 3**. A larger compressive residual stress is produced after the heating on the gold side than the aluminum side. The relaxation of residual stress on the aluminum side should be larger than that on the gold side, because the homologous temperature of aluminum is much higher than that of gold. Coefficients of thermal expansion and volume changes on the formation of Au-Al intermetallic-compounds are shown in **Tables 4** and **5** respectively. The results in **Fig. 11**, **Tables 3**, **4** and **5** suggest that the decrease in bond-strength should arise from that the Kirkendall voids formed during heat-treatment grow to the crack by the residual stress due to the formation of intermetallic-compound.

3.3. Bondability gold plated electrodes

Figure 12 shows the effect of heat-treatment on the bond strength. The shear strength is seen to decrease as the heat-treatment time is increased. When not heat-treated (i. e. heat-treatment time is zero), the bond strength is independent of the film-thickness and plating method. This means that the difference in gold film thickness and plating method has almost no effect on the bond strength. A gold film as thin as $0.07\ \mu\text{m}$ is sufficient for wire-bonding when the sample is not heat-treated.

However, the bond strength decreases with increasing the heat-treatment time, especially the strength of very thin films, less than $0.17\ \mu\text{m}$, are dropped rapidly. The results suggest that the surface property of the films changes as a result of the heat-treatment.

Figure 13 is a high resolution TEM micrograph of the wire-bonded interface, where the gold film was plated by means of the substitution plating and the sample was heat-treated at 453 K for 3.6 ks before wire-bonding. An amorphous layer is seen at some

Table 3 Residual stresses measured in aluminum and gold phases near the bonded interface¹⁷⁾.

Heat treatment	Aluminum side (MPa)	Gold side (MPa)
As bonded	25.9	55.3
639 K-3.6 ks	35.5	81.6
639 K-57.6 ks	24.9	342

Table 4 Thermal expansion coefficients of aluminum, gold and intermetallic-compounds.

	Expansion coefficient ($1/\text{K} \times 10^{-5}$)
Al	2.3
Al ₂ Au	0.94
AlAu	1.2
AlAu ₂	1.3
Al ₂ Au ₅	1.4
AlAu ₄	1.2
Au	1.4

Table 5 Volume changes for the formation of intermetallic-compounds.

	Volume changes
Al ₂ Au	1.087
AlAu	1.013
AlAu ₂	0.953
Al ₂ Au ₅	0.996
AlAu ₄	0.971

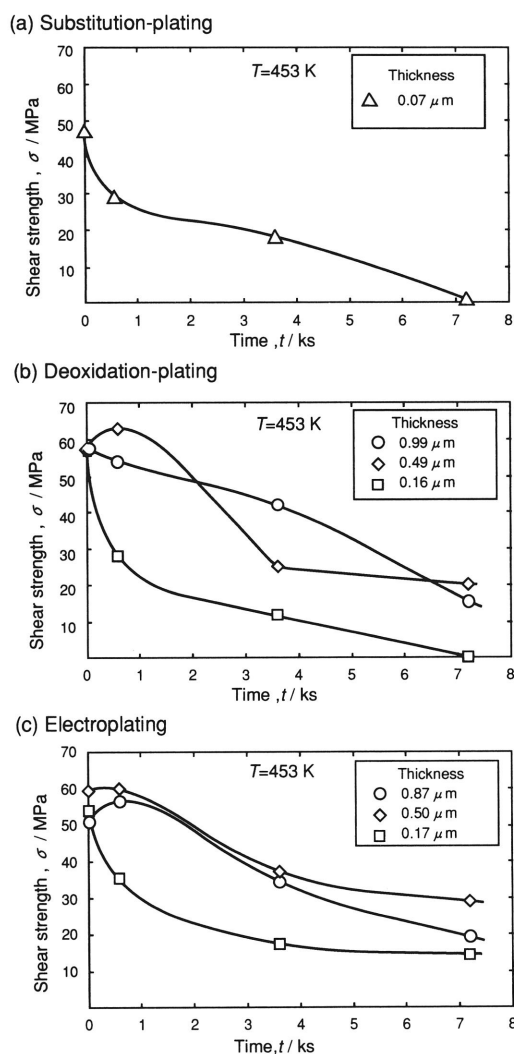


Fig. 12 The dependence of shear strength on heat treatment time¹⁸⁾.

weak Auger peaks of nickel are observed for the substitution plated sample, whereas no Auger peaks for nickel are observed for the deoxidation plated sample or the electroplated sample. After the heat-treatment Auger peaks for nickel are observed for all the samples, especially strong for the substitution plated and the deoxidation plated samples.

Figure 15 shows results from ESCA for the substitution plated film after heat-treatment at 453 K for 7.2 ks. A chemical shift is observed as a function of depth from the surface. A high Ni(OH)₂ peak is observed at the film surface. The nickel which diffuses from the nickel layer to the surface of the gold film is considered to react with water in the atmosphere during the heat-treatment to form Ni(OH)₂. The results in Figs.

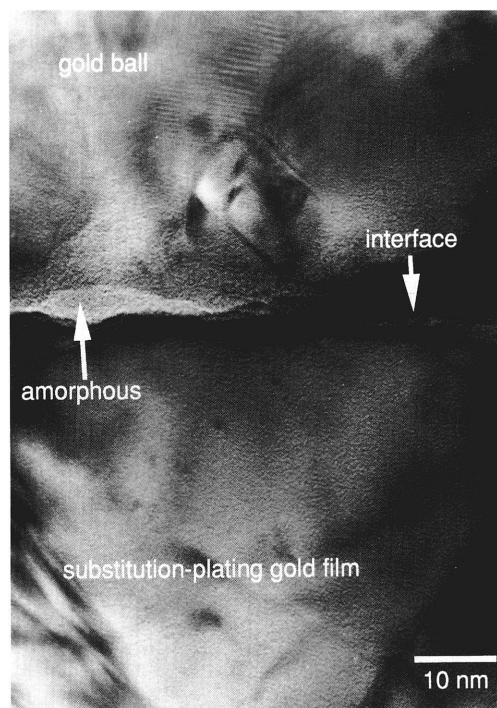


Fig. 13 High resolution TEM micrograph of a wire-bonded interface region after a heat treatment at 453 K for 3.6 ks for the case of substitution plating ($t=0.07 \mu\text{m}$)¹⁸⁾.

region along the interface.

Figure 14 shows results of AES analysis from the surface of the plated gold films before and after a heat-treatment at 453 K for 7.2 ks. Figures 14 (a), (b) and (c) were respectively obtained for samples prepared by substitution plating, deoxidation plating and electroplating. Before the heat-treatment,

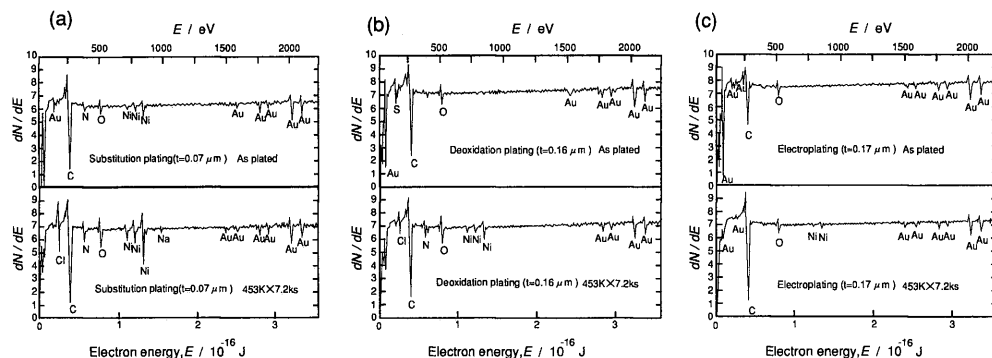


Fig. 14 Auger electron spectroscopy before and after the heat-treatment, (a) substitution plated sample ($t = 0.07 \mu\text{m}$), (b) deoxidation plated sample ($t = 0.16 \mu\text{m}$) and (c) electroplated sample ($t = 0.17 \mu\text{m}$)¹⁸⁾. (dN/dE means differential Auger spectrum intensity.)

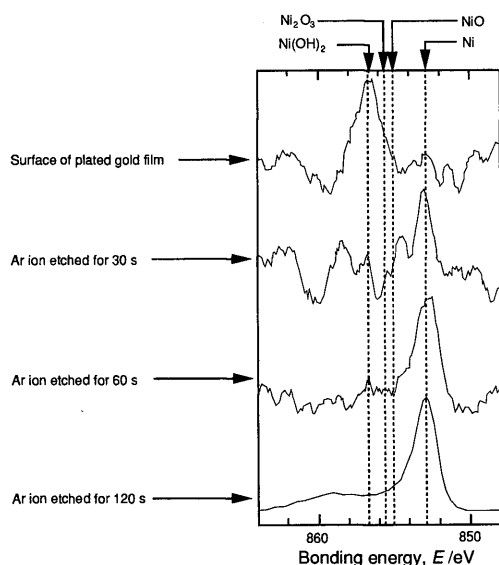


Fig. 15 Electron spectroscopy of ESCA for the substitution plated sample after heat-treatment at 453 K for 7.2 ks ($t = 0.07 \mu\text{m}$)¹⁸⁾.

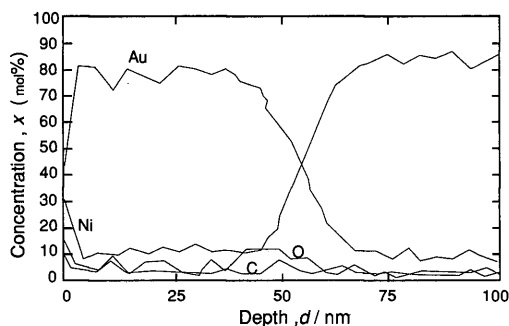


Fig. 16 Auger electron spectroscopy as a function of depth from the surface taken from a substitution gold-plated sample after heat-treatment at 453 K for 7.2 ks ($t = 0.07 \mu\text{m}$)¹⁸⁾.

12, 14 and 15 suggest that the $\text{Ni}(\text{OH})_2$ at the surface should deteriorate the bondability.

Figure 16 shows the composition change determined by AES analysis as a function of depth from the film surface for the same sample used for the ESCA analysis. The nickel and oxygen concentrations decrease rapidly with an increase in depth. If the existence of $\text{Ni}(\text{OH})_2$ at the surface indeed deteriorates the bondability, then bondability should be enhanced by removing the surface layer.

After heat-treatment at 453 K for 7.2 ks, the surface layer of the substitution plated film was removed in-situ in the AES equipment by means of Ar ion etching. The

thickness of the removed layer was about 5 nm. **Figure 17** compares the change in bond strength before and after the heat-treatment, and after ion etching. As expected, the bond strength increases once the surface-layer is removed, although the recovered strength is a little lower than that before the heat-treatment. The lower bond strength may be due to the higher nickel concentration on the plated gold film than that of the sample without heat-treatment even after the ion etching.

4. Conclusion

In order to develop a microstructure observing technique for wire-bonded interfaces, which is important in the IC industry, thin films of wire-bonded gold-aluminum samples were prepared by ion milling and microtome techniques and observed by TEM.

The effect of organic contamination for wire-bonding and the dependence of wire-bondability on the thickness of plated gold film and the plating method were also investigated by TEM and shear tests. The results obtained are as follows.

(1) In the films prepared with a microtome no amorphous phase is observed, whereas in the films prepared by ion milling a large part of the intermetallic compound formed on the interface has become amorphous.

(2) At the whole interface of the cleaned samples the intermetallic-compound is observed, whereas at the interface of the contaminated samples an organic contamination layer is observed instead of the intermetallic-compound.

(3) The cleaned samples are generally higher in shear strength than the contaminated samples.

(4) Both contaminated and cleaned samples have similar features for the change in shear strength due to heating.

(5) Voids are produced by heating along the gold-intermetallic compound interface. The voids deteriorates the bond strength.

(6) Without heat-treatment the bond strength does not depend on either the film-thickness or the plating method.

(7) The nickel (from the substrate) concentration on the film surface increases with an increase in heating time, and a corresponding decrease in shear strength is observed.

(8) A strong $\text{Ni}(\text{OH})_2$ peak is observed at the surface of substitution plated sample by means of ESCA after the heat-treatment. The existence of $\text{Ni}(\text{OH})_2$ at the surface is considered to deteriorate the bondability.

(9) The nickel and oxygen concentrations decrease rapidly with the increase in depth from the surface.

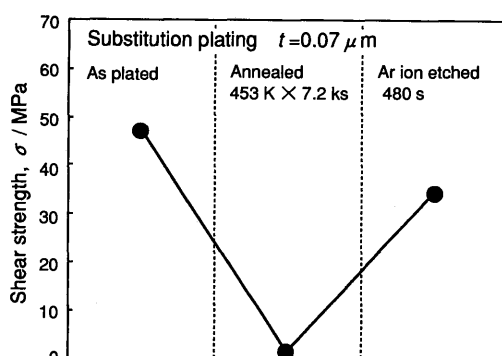


Fig. 17 Change in bond strength due to annealing and Ar etching of the gold plate before bonding. Heat treatment: 453 K for 7.2 ks. Ar ion etching: a surface layer of about 5 nm was removed by etching after the heat treatment¹⁸⁾.

(10) The bond strength of heat-treated samples recovers when the surface layer of the plated gold film is removed before bonding.

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