Direct Measurement of Interface Strength between Copper Submicron-Dot and Silicon Dioxide Substrate*

Hiroyuki HIRAKATA**, Takayuki KITAMURA** and Yoshitake YAMAMOTO***

We develop an experimental evaluation method of interface strength for ductile submicron-dots on a hard substrate without collapse of the dot. The validity is examined by a copper (Cu) submicron-dot on a silicon dioxide (SiO₂) substrate with the rigid-layer of tungsten (W), which restrains the deformation and decreases the influence of complicated stress field due to the contact of tip. The diamond tip is dragged horizontally along the SiO₂ surface and the load is applied to the side edge of the W layer at a constant displacement rate using a modified atomic force microscopy. Both the lateral and the vertical load and displacement are continuously monitored during the test. The lateral load, \( F_l \), increases almost in proportion to the lateral displacement, \( \delta_l \), and the Cu dot with the W layer is clearly separated from the SiO₂ along the interface. The restraint by the W layer works well so that there are little damages in both the delaminated W/Cu dot and the substrate. The delamination lateral load, \( F_{lC} \), is successfully measured.

**Key Words:** Delamination, Material Testing, Micromechanics, Submicron Dot, Atomic Force Microscopy, Interface Strength, Micro Material

1. Introduction

Since micro-electronics and micro-mechanical devices consist of various components, they have many intrinsic bi-materials interfaces. Stress concentration on an interface due to the deformation mismatch sometimes causes delamination, which brings about fatal malfunction of the device. On the other hand, the size required for the components becomes submicron-scale in order to shrink the device. It is necessary, therefore, to evaluate the interface strength among the submicron-components and the substrate.

Two steps are required to evaluate interface strength. The first is development of an experimental method for separating a submicron-component from a substrate, and the second is an investigation of the mechanics dominating the interface fracture. Here, it should be noted that the fracture of components is avoided during the delamination test, because it changes the measured strength. The focus of this study is on the first step.

Significant numbers of approaches\(^{(1)}\)–\(^{(17)}\) have been investigated to evaluate the interface strength among thin films and a substrate. They are classified into two types of methods. The one is directly loading methods, in which a load is applied to the film using a needle, such as scratch tests\(^{(1)}\)–\(^{(3)}\), indentation tests\(^{(4)}\)–\(^{(6)}\) and other ones\(^{(7)}\),\(^{(8)}\). The most of these methods causes fracture of the film and/or the substrate. The other is sandwich type methods such as four-point bending tests\(^{(9)}\), pull/topple tests\(^{(10)}\) and cantilever tests\(^{(11)}\)–\(^{(14)}\). A hard substrate is glued on the film and a load is applied through the substrate. These methods can restrain plastic deformation and fracture of the film. However, these can not be applied to materials with strong interface due to the limitation of the glue strength. In addition, the stress-concentrated region on an interface, which dominates interface delamination, is affected by the length scale\(^{(12)}\),\(^{(18)}\).

Then, we developed a method\(^{(19)}\) for applying a load to dots, of which three-dimensional scale is small, on a substrate by a modified atomic force microscopy (AFM), and successfully separated a hard tungsten (W) micron-dot from a silicon (Si) substrate. However, it can not be applied on ductile dots such as copper (Cu) because the contacted zone collapses before the delamination. It is necessary, therefore, to develop a new method that can separate...
ductile dots from a substrate without the collapse.

In this study, a delamination method for ductile submicron-dots on a substrate is proposed, and the validity is examined by a copper (Cu) submicron-dot on a silicon dioxide (SiO₂).

2. Proposed Method

The delamination test\(^{(19)}\) is schematically illustrated in Fig. 1. A dot is prepared on a substrate. A diamond tip is dragged horizontally along the surface of the substrate and a lateral load is directly applied to the side edge of the dot using a modified AFM. Both the lateral and the vertical load and displacement are continuously monitored during the test. This method successfully separates hard dots from a substrate.

Figure 2 shows schematic view of proposed delamination test for ductile dot. A rigid-layer is deposited on the dot and a load is directly applied to the rigid-layer. The rigid-layer restrains the deformation and collapse of the dot. Moreover, in this method, the influence of complicated stress field due to the contact of tip is decreased because the contact zone is away from the interface edge at which delamination occurs.

In order to examine preliminarily the effect of the rigid-layer on the stress distribution in the dot/substrate system, a finite element analysis (FEA) using a commercial FEA code, MSC Nastran, is conducted for the models of a columnar Cu dot of 50 nm height and 500 nm across on a SiO₂ substrate with/without a W layer of 50 nm. The materials are assumed to be elastic and the elastic constants used are listed in Table 1. Figure 3 shows the mesh division. Half of the system is analyzed taking into account the symmetry, and a point load is applied at the edge indicated by the arrow. Because the singular stress field near the free-edge of the interface between Cu and SiO₂ appears due to the mismatch of deformation, the region is carefully divided into fine mesh. The element size at the interface edge is set at 2 nm.

Figure 4 shows von Mises stress distributions, \(\sigma_{\text{mises}}\), obtained by the analyses. Here, \(\sigma_{\text{mises}}\) is normalized by \(\sigma_{\text{mises}}\) at the second element from the interface edge, so

![Diagram](image1)

**Table 1** Elastic constants

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s modulus, GPa</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>410</td>
<td>0.28</td>
</tr>
<tr>
<td>Cu</td>
<td>129</td>
<td>0.34</td>
</tr>
<tr>
<td>SiO₂</td>
<td>70</td>
<td>0.20</td>
</tr>
</tbody>
</table>

![Diagram](image2)
that the stress intensities near the edge in Fig. 4 (a) and (b) are equal. Without rigid-layer, significant stress concentration occurs in the region near the tip-contact as shown in Fig. 4 (a) and this will cause collapse of soft dot. On the other hand, stress concentration in the soft dot is extremely decreased due to the rigid-layer as shown in Fig. 4 (b). Therefore, it is expected to restrain the collapse of dot and separate smoothly it from the substrate.

3. Experimental Procedure

3.1 Specimen preparation

Figure 2 shows a schematic illustration of specimen tested. A columnar Cu dot of submicron-scale with the height of 50 nm is deposited on a Si substrate with a 100 nm thickness of SiO$_2$. A layer made of W with the thickness of 50 nm is employed as a rigid-layer. The effect of adhesion area on the delamination behavior is examined by the specimens (specimen 1 – 5) with different diameters from about 300 to 800 nm. The reasons why the W/Cu/SiO$_2$ system is selected in this study are: (1) Cu is a ductile material, (2) W and SiO$_2$ have a high yield stress, and (3) these are important materials in micro-electronic devices. In order to examine the effectiveness of the rigid-layer, a specimen without the rigid-layer (specimen S, thickness of Cu dot: 150 nm) is also prepared.

The specimens are prepared using a conventional electron-beam (EB) lithography technique. The fabrication procedure of the specimens is schematically illustrated in Fig. 5.

(1) A Si wafer (thickness: 520 µm, surface: (100) plane) is thermally oxidized to grow a 100 nm thickness of SiO$_2$. After cleaning the wafer by ultrasonic vibration with organic solvents (acetone and isopropyl alcohol), an EB resist with the thickness of about 350 nm is deposited by spin-coating.

(2) The dot patterns are drawn using an EB lithography system.

(3) The exposed part of the EB resist is removed by developer.

(4) After reducing a pressure to $4.4 \times 10^{-4}$ Pa at room temperature, the Cu film of thickness 50 nm is sputtered on the specimen in argon (Ar) gas of 0.8 Pa.

(5) The W film of thickness 50 nm is sputtered in the same manner.

(6) The EB resist with the W/Cu films is removed by remover solvent (lifted off).

The AFM images of the specimen are shown in Fig. 6. Although unevenness is observed on the dot side surface (Fig. 6 (a)), the magnified view of the edge (Fig. 6 (b)) reveals that it is fairly smooth in comparison with the dot diameter. Here, the precise angle between the side surface and the interface cannot be measured by means of AFM due to the pyramidal shape of the probe. A field emission scanning electron microscopy observation (Fig. 7) confirms the sharp corner at the free-edge of interface.

3.2 Testing apparatus

Figure 8 shows the testing apparatus and the loading system. A special loading apparatus, which can control precisely the lateral displacement of the tip in the resolution of 4 nm, is attached to an AFM. The apparatus consists of three force/displacement transducers, each of which has two fixed outer plates and a spring suspended center plate. A loading tip made of diamond is attached to the center plate of the middle transducer. The normal load, $F_n$, of the tip is electrostatically generated by applying a voltage between the center and the outer plates in the middle transducer, and the resulting normal displacement, $\delta_n$, is detected by change in capacitance. The side transducers generate the lateral load, $F_l$, in the same manner, and control the lateral displacement, $\delta_l$, using the capacitance.
Fig. 6 AFM images of specimen before delamination test (specimen 4)

(a) Entire view of W/Cu dot

(b) Magnified view of specimen edge

Fig. 7 FE-SEM micrograph of specimen

4. Results and Discussion

Figure 9 shows an AFM image of the fracture surface of specimen 4 tested under $F_n = 70 \mu N$. The region indicated by the dashed line designates the place where the W/Cu dot was located. The fracture clearly takes place along the interface between the Cu dot and the SiO$_2$ substrate. There is no ditch due to the scratch on the substrate. In the tests under conditions of $F_n < 45 \mu N$, the tip ran onto the W/Cu dot and the interface fracture between Cu dot and SiO$_2$ layer did not take place.

Figure 10 shows an AFM image of the dot without the rigid-layer (specimen S) tested under $F_n = 70 \mu N$. The contacted area of Cu collapses and the delamination is not observed. On the other hand, the separated W/Cu dot was carefully examined by AFM. Figure 11 (a) shows the entire view of the W/Cu dot, which signifies no trace of col-
lapse. There is little damage in the W layer even near the tip-contacted zone shown in the magnified view of the edge (Fig. 11 (b)). This suggests that the W/Cu/SiO$_2$ system works well for preventing the fracture of Cu dot.

Figures 12 and 13 show the relationships between the lateral load, $F_l$, and the lateral displacement, $\delta_l$, and between the normal displacement, $\delta_n$, and $\delta_l$ in specimen 2 tested under $F_n = 50\, \mu\text{N}$, respectively. It is clear that $F_l$ remains almost zero up to about $\delta_l = 100\, \text{nm}$ before $F_l$ increases almost in proportion to $\delta_l$ up to point “a”. This indicates that the tip hits the edge of the W layer at about $\delta_l = 100\, \text{nm}$ as schematically illustrated in Fig. 2. In other words, the load is applied to the dot in the region where $F_l$ increases. Attention must be paid to the fact that $\delta_n$ also increases and reaches up to about 10 nm at point “a”. The tip contacts only the W layer at point “a” due to the angle of the tip (Fig. 14). The abrupt decrease of $F_l$ and $\delta_n$ at the point indicates the separation of the W/Cu dot from the SiO$_2$ substrate. After then, the tip moves on the SiO$_2$ substrate again. Then, the pure resistance against the deformation and the subsequent delamination is measured because the friction is negligible.

The peak loads, $F_{lC}$ (the lateral load at point “a”), are listed in Table 2. $F_{lC}$ slightly increases with an increase in adhesion area, $A_d$. Then, the interface strength can be estimated by deformation analysis as the concentrated (intensified) stress at the interface edge on the basis of the obtained values of $F_{lC}$. However, there are difficulties in performing the precise analysis, because the nonlinear analysis is inevitable for the deformation of Cu dot and the property of minute materials after yield is complex and is different from those of bulk materials(20). Moreover, the effect of the intrinsic internal stress introduced during the fabrication process has to be taken into account(12), (21). This is next target of this project, and it is out of scope of this paper.

5. Conclusion

The results obtained are summarized as follows:

(1) We develop a method for separating a ductile submicron-dot from a substrate using a modified AFM, which can control and precisely measure the load and the
tip displacement. The validity of proposed method is examined by a Cu submicron-dot on a SiO₂ substrate.

(2) A W layer is deposited on the Cu dot as a rigid-layer to restrain the collapse of the dot and to decrease the influence of complicated stress field due to the contact of tip.

(3) The diamond tip is dragged horizontally along the SiO₂ surface and a load is directly applied to the side edge of the W layer. The Cu dot with the W layer is clearly separated from the SiO₂ along the interface.

(4) The restraint by the W layer works well so that there are little damages in both the delaminated W/Cu dot and the substrate.

(5) The delamination lateral load, $F_{IC}$, is successfully measured.

Acknowledgements

This paper is supported in part by Center of Excellence for Research and Education on Complex Functional Mechanics (COE program of the Ministry of Edu-
Fig. 14 Schematic showing location of specimen and tip at delamination (point “a”)

citation, Culture, Sports, Science and Technology, Japan) and by Grant-in-Aid for Scientific Research of (B) (No.13555026) of Japan Society of the Promotion of Science.

References