Effect of Loading Frequency on Fatigue Crack Growth between a Submicron-Thick Film and a Substrate

Do VAN TRUONG**, Hiroyuki HIRAKATA*** and Takayuki KITAMURA***

The crack growth along the interface between a submicron-thick film (Cu) and a substrate (Si) under fatigue is experimentally investigated at the load-frequencies of 0.1 Hz and 1 Hz in a laboratory environment (45±5% RH). A modified four-point bend specimen, which has only one interface crack to facilitate the control of crack growth, is used for the tests. The results reveal that the clear interface crack between Cu and Si grows under the cyclic load. The crack growth rate per cycle, \( \frac{da}{dN} \), is governed by the stress intensity factor range, \( \Delta K_i \), at each frequency and the sigmoidal relationship consisting of three stages are observed in the \( \frac{da}{dN} - \Delta K_i \) curve; the threshold, the stable growth and the critical growth. \( \frac{da}{dN} \) greatly increases as the frequency decreases in the stable growth region. The crack growth rate per time, \( \frac{da}{dt} \), shows a good correlation with the maximum stress intensity factor, \( K_{imax} \), independently of the loading frequency. This indicates that the environmental effect due to humidity in air plays a dominant role on the crack growth.

Key Words: Fatigue, Crack Propagation, Fracture Mechanics, Thin Film, Interface

1. Introduction

Microelectronics devices consist of small structured materials such as thin films. One of the major failures observed in these devices is delamination because the stress concentrates at an interface between different materials due to the mismatch of deformation. Many studies have been conducted to evaluate the strength of an interface\(^{(1)}-^{(11)}\) between a thin film and a substrate. However, most of the studies have focused on evaluation of the interface strength under the monotonic loading\(^{(1)}-^{(8)}\), and few studies have investigated on subcritical crack growth under the cyclic loading\(^{(9)}-^{(11)}\). In particular, fatigue crack growth along the interface between a submicron-thick film and a substrate has not been reported.

A crack propagates in bulk materials or along an interface due to cyclic loading, and the fatigue crack growth rate is characterized by the stress intensity factor range or the energy release rate range\(^{(12)}-^{(13)}\). Although the fatigue crack growth is essentially a time independent phenomenon which is caused by irreversible dislocation motion, it is well known that the crack growth is greatly influenced by the stress corrosion and shows the dependence on the loading frequency\(^{(14)}-^{(17)}\). However, the effect of loading frequency on the crack growth along a thin film/substrate interface has not been investigated so far.

The fatigue crack growth behavior along the interface between a submicron-thick metal film and a substrate differs from that in bulk bi-materials, since the dislocation motion is restricted in the thin layer of metal film and the plastic deformation of the film is constrained by the stiff substrate. The purpose of this study is to elucidate the crack growth behavior between a submicron-thick copper (Cu) film and a bulk silicon (Si) substrate under fatigue loading. Moreover, the effect of loading frequency on the fatigue crack growth is also investigated.

2. Experimental Procedure

2.1 Material and specimen

Figure 1 shows a TEM micrograph of the multilayered material tested. After a Cu film with the thickness of 200 nm is sputtered on a (100) surface of a Si substrate, a silicon nitride (Si\(_3\)N\(_4\)) film of thickness 500 nm is deposited on the Cu film by sputtering to restrain the large plastic deformation of the Cu film during the test.

The modified four-point bend specimen\(^{(11)}\) shown in Fig. 2 is adopted for the test. Because the standard four-point bend specimen has two symmetrical cracks, the
same singular stress field theoretically takes place near both crack tips under an applied load. However, in reality, it is difficult to control the equal growth of the cracks because a slight unbalance in the loading brings about the asymmetrical singularities. The modified specimen, which has only one interface crack, enables us to control the stable crack growth.

A plate of stainless steel is polished with emery papers and diamond paste, and the plate is cleaned by ultrasonic vibration in acetone and isopropyl alcohol. After the plate is glued carefully on the wafer with the Si$_3$N$_4$/Cu films by standard epoxy, the wafer is cut into rectangular coupons using a dicing machine. The shape and the size of specimens are given in Fig. 2. The thickness of epoxy layer and specimen width is carefully examined in each specimen, and they are used for the stress analysis.

The stress in the modified four-point bend specimen is numerically analyzed under the plane strain condition by a commercial FEM code, ABAQUS 6.5. Elastic analyses are conducted for a specimen with different crack length, where the elastic constants used are listed in Table 1. Since carefully deposited thin films have almost the same elastic modulus as that of their bulk\cite{18,19}, we use it for the Cu film. Figure 3 shows the boundary condition and mesh division near the crack tip in an FEM model. The region near the crack tip is carefully divided into fine elements with the smallest element size of 2 nm to precisely analyze the stress field on the basis of continuum mechanics concept. Figure 4 shows the dependence of the total stress intensity factor, $K_i$, and the phase angle, $\Psi = \tan^{-1}(K_2/K_1)$, on the crack length, $a$. Here, the stress field near the tip of an interface crack is characterized by the complex stress intensity factors, $K_1$ and $K_2$, which is in the form,

$$\sigma_y + i\tau_{xy} = \frac{K_1 + iK_2}{(2\pi r)^{1/2}} \left( \frac{r}{h_i} \right)^{\varepsilon}$$

and the total stress intensity factor, $K_i$, is defined as follows.

$$K_i = \sqrt{K_1^2 + K_2^2} = \lim_{r \to 0} \sqrt{2\pi r \left( \sigma_y^2 + \tau_{xy}^2 \right)}$$

Here, $\sigma_y$ and $\tau_{xy}$ are the normal and the shear stresses, respectively. $r$ is the distance from the crack tip. $\varepsilon$ is the oscillation index and $h_i$ is the characteristic length which can be set arbitrarily\cite{20}. In Fig. 4, $K_i$ is normalized by the magnitude of $K_i$ at $a = 2$ mm. The figure clearly indicates that $\Psi$ and $K_i$ are almost constant under a constant applied load if the crack tip is sufficiently inside the inner loading point. $\Psi$ is about 47 degrees for the characteristic length, $h_i$, of 1 nm. Meanwhile, $K_i$ decreases when the tip is in the vicinity of the inner loading point.

Since the interface between a thin film and a substrate is usually brittle, the introduction of a pre-crack with a desired length is not an easy task. In each speci-

<table>
<thead>
<tr>
<th>Material</th>
<th>Young's modulus $E$, GPa</th>
<th>Poisson's ratio $\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless</td>
<td>200</td>
<td>0.30</td>
</tr>
<tr>
<td>epoxy</td>
<td>2.5</td>
<td>0.30</td>
</tr>
<tr>
<td>Si$_3$N$_4$</td>
<td>304</td>
<td>0.27</td>
</tr>
<tr>
<td>Cu</td>
<td>130</td>
<td>0.34</td>
</tr>
<tr>
<td>Si</td>
<td>130</td>
<td>0.28</td>
</tr>
</tbody>
</table>

Table 1 Elastic constants

![Fig. 1 TEM micrograph of a multi-layered material tested](image1)

![Fig. 2 Modified four-point bend specimen for the multi-layered material Si$_3$N$_4$/Cu/Si](image2)
men, however, the stress intensity factor prominently decreases as the crack tip approaches the inner loading point as shown in Fig. 4 and thereby a well-controlled interfacial pre-crack can be introduced by decreasing the length between the inner loading points in Fig. 2.

Residual stress in thin films affects the crack growth behavior in many specimen configurations in general. However, in the four-point bending specimen, stress relaxation of the thin film due to debonding is essentially constrained by the stiff substrates. Therefore the effect on the crack propagation is small\(^1\).

### 2.2 Fatigue Test

A cyclic load is applied to the specimen by a micro fatigue machine with an electro-magnetic actuator (Shimadzu MMT-100N) as illustrated in Fig. 5. The load, \(P\), and the displacement, \(u\), are monitored at the loading point during the tests by a load cell and a differential transformer, respectively. The resolution of the load is 0.01 N which corresponds to the stress intensity factor of \(K_i \approx 0.002 \text{ MPam}^{1/2}\). All the tests are performed in laboratory environment at relative humidity (RH) of 45 \(\pm 5\%\) at room temperature of 295 \(\pm 2\) K.

Fatigue tests are conducted under constant stress am-
plitude of a sinusoidal waveform at the frequencies of 1.0 Hz and 0.1 Hz with the load ratio, \( R = P_{\text{max}} / P_{\text{min}} \), of 0.54. Here, \( P_{\text{max}} \) and \( P_{\text{min}} \) are the maximum and minimum loads, respectively. Test conditions are listed in Table 2. The crack length, \( a \), is evaluated by the compliance method\(^{11}\). The relationship between the compliance, \( \lambda \), and \( a \) is calculated by the FEM for each specimen.

The fracture toughness is also evaluated by applying a monotonic load on the specimen.

3. Results and Discussion

3.1 Fatigue crack growth behavior

Figure 6 shows the relationship between the compliance, \( \lambda \), and the number of cycles, \( N \), in specimen A-2 (\( \Delta K_i = 0.22 \text{ MPam}^{0.5} \)) under the cyclic load with the frequency of 1.0 Hz. The converted crack length, \( a \), is indicated on the right side as a reference. The crack begins to propagate just after the test is started, and the crack growth rate, \( da/dN \), is almost constant up to approximately \( a = 10 \text{ mm} \) (450 cycles). This is consistent with the region where the applied \( \Delta K_i \) is constant. After that, the crack growth slows down and stops. This is because the crack approaches the inner loading point where \( a = 12.4 \text{ mm} \) and the magnitude of \( \Delta K_i \) decreases rapidly as shown in Fig. 4. In the linear region, \( da/dN \) is evaluated from the slope of the \( a - N \) curve as about \( 1.91 \times 10^{-3} \text{ m/cycle} \). The fluctuation is estimated as \( 1.23 \times 10^{-5} < da/dN < 2.32 \times 10^{-5} \text{ m/cycle} \) by the chained and the dashed lines shown in Fig. 6.

On the other hand, Fig. 7 shows the crack growth curve of specimen A-1 (\( \Delta K_i = 0.20 \text{ MPam}^{0.5} \)) under the cyclic load with the frequency of 1.0 Hz. The magnitude of \( \lambda \) is almost constant even after \( 10^6 \) cycles and this indicates no crack growth. Thus, there is a threshold in the crack growth, and the stress intensity factor range \( \Delta K_i = 0.20 \text{ MPam}^{0.5} \) is under the threshold. Though a threshold normally exists in the fatigue crack growth in bulk metals and along interfaces in a bulk\(^{9}\), \( ^{21} \), \( ^{22} \), it is rarely reported along an interface between a submicron-thick film and a substrate.

After the fatigue tests, the fracture surfaces of both the film and substrate sides are examined by Auger electron spectroscopy (AES). As typically shown in Fig. 8, only Cu peaks (56, 764, 836 and 916 eV) are confirmed on one side while Si (89 and 1614 eV) is found on the other side for all specimens tested. Thus, the crack propagated perfectly along the interface between Cu and Si.

3.2 Effect of frequency on fatigue crack growth

Figure 9 shows the relationships between \( da/dN \) and \( \Delta K_i \) at the loading frequency of 1.0 and 0.1 Hz. Each curve is expressed as a typical sigmoidal curve with three distinct regions; the threshold, the stable growth and the critical growth. The stable crack growth region is described by the Paris law,

\[
da/dN = C\Delta K_i^m.
\]

The magnitudes of \( m \) are 5.11 and 7.76 at the frequencies of 1.0 Hz and 0.1 Hz, and those of \( C \) are 0.0567 and 21.9, respectively. The crack growth greatly accelerates with a decrease of the loading frequency. This indicates that some environmental effect due to humidity in air at the crack tip plays an important role on the crack growth\(^{23},^{24} \). Although clarifying the mechano-chemical
Fig. 9 Relationship between fatigue crack growth rate and stress intensity factor range at the frequencies of 0.1 Hz and 1.0 Hz.

Fig. 10 Von Mises stress distribution along the interface between the Cu film and Si substrate at $a = 8$ mm.

The upward arrow indicates the equivalent fracture toughness, $(1 - R)K_{ic} = 0.47$ MPa$m^{0.5}$, which is $\Delta K_i$ at $K_{max} = K_{ic}$. Here, $K_{ic}$ is evaluated by a monotonic loading test on the modified four-point bend specimen as $K_{ic} = 1.02$ MPa$m^{0.5}$. The downward arrow indicates the threshold, $\Delta K_{sth}$, where the crack does not propagate in $10^9$ cycles. The threshold is almost independent of the loading frequency.

Figure 10 plots the Mises stress distribution along the Cu/Si interface at $a = 8$ mm at the maximum load in the fatigue test of $\Delta K_i = 0.22$ MPa$m^{0.5}$ in specimen A-2. As the yield stress of submicron-thick Cu film is about one order higher than that of bulk Cu ($\approx 70$ MPa), it reaches about 600 MPa$^{(25),(26)}$. The region where the stress exceeds 600 MPa is about 60 nm and this is much smaller than the region dominated by the singular stress field. Thus, the crack propagates under the small scale yielding condition.

The crack growth rate per time, $da/dt$, is plotted against the maximum stress intensity factor, $K_{max}$, in Fig. 11. There is no difference among the $da/dt - K_{max}$ relationships, and this indicates that the crack grows purely time-dependent. The effect of cyclic loading is little, and it suggests that the environmental effect is responsible for the crack growth.

4. Conclusions

Fatigue crack growth along the interface between a submicron-thick film and a substrate has been rarely reported. In this study, the behavior of interface crack growth between the submicron-thick Cu film and the Si substrate under fatigue is experimentally investigated using a modified four-point bend specimen with a crack, where the stress intensity can be easily controlled. The results are summarized as follows.

(1) The interface crack grows perfectly along the interface between the Cu film and the Si substrate under the cyclic loading.

(2) $da/dN - \Delta K_i$ curve obtained shows the sigmoidal relationship consisting of the three stages: threshold, stable growth and critical growth.

(3) In the stable growth region, $da/dN$ at the frequency of 0.1 Hz is about one order of magnitude higher than that at the frequency of 1.0 Hz for an equal magnitude of $\Delta K_i$. The threshold is almost independent of the loading frequency.

(4) The fatigue crack growth rate per time, $da/dt$, is correlated well with the maximum stress intensity factor, $K_{max}$, regardless of the loading frequency. This indicates that the crack growth is caused by the environmental effect due to humidity in air at the crack tip.

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