Mechanism of Damage Process on $\text{Si}_3\text{N}_4/\text{Cu}$ Interface in Nanoscratch Test*

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Abstract

The progressive miniaturization and integration of electronic devices has made interfacial fracture between thin films of several nanometers thickness an important consideration. The present paper attempts to clarify the mechanism of the damage process along an interface between multi-layered films during nanoscratch tests. Using the edge of a Berkovich-type indenter, a nanoscratch test was performed on sub-micron films of $\text{Si}_3\text{N}_4/\text{Cu}/\text{TaN}$ fabricated on a silicon substrate. Initiation of damage occurs near the $\text{Si}_3\text{N}_4/\text{Cu}$ interface before the tip of indenter reaches the interface. This implies that Si_3N_4 is weak in terms of interfacial resistance to failure. A spot-like damaged area appears under the scratched line, and the size of this area is several nanometers or less. Critical values measured in the nanoscratch tests and three-dimensional non-linear finite element analysis reveal that shear stress concentration appears behind the indenter; this agrees qualitatively with the behavior of the spot-like damage area as observed under an optical microscope. In order to examine the validity of the damage mechanism, another nanoscratch test was performed by moving the indenter in the opposite direction. Critical load is greater than that in the original test due to the difference in the shape of the indenter, but stress analysis reveals that initiation of spot-like damage occurs when the two tests are equal in terms of peak stress acting on the interface.

Key words: Micromechanics, Material Testing, Delamination, Finite Element Method, Reliability, Thin Film, Nanoscratch Test, Interfacial Damage, Advanced LSI

1. Introduction

A multi-layered structure consisting of thin films fabricated on a silicon substrate is a typical small structure used in electronic devices such as LSI conductors. Since the multi-layered body includes several interfaces and the devices are decreasing in size, interfacial fracture is becoming a major failure mechanism affecting the reliability of devices. Therefore, several qualitative and quantitative methods for measuring interfacial strength have been devised ⁽¹⁾⁻⁽⁵⁾. The scratch test is a typical method for measuring the adhesion of film ⁽⁶⁾⁻⁽⁸⁾. In the scratch test, a vertical load is applied by the indenter while the indenter is moved horizontally. The critical load in fracture is evaluated with respect to film adhesion. However, since the scratching process is related to the plasticity and failure of films, extracting the target interfacial strength quantitatively is difficult. Moreover, since the scratch test was developed for single-layer films such as coating films, for the multi-layered

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functional sub-micron films in an LSI, we must clarify the damage process in the scratch test and improve the evaluation technique. During the scratching process, several nonlinear phenomena occur, including plasticity, failure, and contact. In particular, identifying the initiation of interfacial damage is important for the quantitative measurement of interfacial strength. In the case of thin films in the LSI, since film adhesion is poor, the initiation of interfacial fracture cannot be confirmed from the surface appearance. Therefore, the mechanism of the damage process during the scratch test remains unclear.

In the present study, a nanoscratch test for multi-layered thin films of sub-micron thickness was carried out and the interfacial damage process was examined. The Si_3N_4 (100 nm)/Cu (300 nm) interface can be observed from the surface, because the nitride thin film is a transparent body. Then, not only the scratching load, but also the damage initiation was evaluated. An FEA model was constructed by configuring an indenter, films, and a substrate. Nonlinear analysis with plasticity of the films and the contact was carried out in order to examine the stress and strain evolutions near the interface during the test. The critical stress for interfacial damage initiation was evaluated quantitatively and was verified by another scratch test.

2. Nanoscratch Test

2.1 Test method

According to the loading system, scratch tests can be classified into two types: the unidirectional scratching type^{(8), (9), (11)} and the swinging type^{(7), (10)}. Both test methods can qualitatively extract the behavior of adhesion on the substrate. Several measures for detecting damage have been proposed, including (i) a change in a normal or lateral load, (ii) Acoustic Emission (AE), and (iii) observation of the surface. Because of the difficulty involved in extraction of the interfacial strength from the measured value in (i) or (ii), the interfacial damage was detected by observation after the test. In the present study, a unidirectional scratching system was employed in order to exclude the dynamic behavior of the indenter. In addition, damage initiation can be easily observed from the surface. Figure 1 shows a schematic illustration of the nanoscratch test. A sharp tip is moved horizontally while the load normal to a surface is increased. When interfacial damage occurs, the critical load can be determined by the relation between the applied load and the scratch length at the damaged point.

2.2 Tested sample and conditions

The test material, shown in Fig. 2, consists of Si_3N_4 , Cu, Ta, and a silicon substrate. The copper and silicon nitride are used as a metal layer and for passivation, respectively, in an

Fig. 1 Schematic illustration of nanoscratch system with an indenter of Berkovich type.

Fig. 2 Schematic illustration of cross-section of tested material.

Fig. 3 Testing apparatus for nanoscratch test.

LSI. Tantalum is fabricated on a silicon substrate for the diffusive barrier to prevent copper atoms from entering the substrate. As a result of a mismatch in deformation between the silicon nitride and copper layers, interfacial fracture often occurs at the $\text{Si}_3\text{N}_4/\text{Cu}$ interface⁽⁵⁾. Since the silicon nitride layer is transparent, this interfacial damage can be observed under an optical microscope. Moreover, since the upper layer has high hardness, friction and fracture on the surface exert little influence on the interfacial damage. The hard coating layer technique has been employed in the indentation test for measuring the interfacial strength, and the effectiveness of the upper hard layer has been verified^{(4), (8)}.

Experiments were carried out at room temperature and in air. A commercial scratch testing system, NanoindenterXP (MTS System Co.), shown in Fig. 3, was used. The system has fine resolutions of load and displacement: 1 nm and 0.002 nm. The testing system was placed on a vibration isolation platform (Nano-k) in a box equipped with a shock absorber. A Berkovich-type sharp tip having a curvature of 50 nm, shown in Fig. 1, was used. The applied load and indented depth can be recorded continuously. The critical load for interfacial damage initiation was evaluated from the relation between applied load and scratch length at the damage area. The horizontal scratch length, L , is set to 200 microns, and the scratching was performed at 1 or 10 $\mu\text{m/s}$. Maximum load ranges from 1 to 5 mN. The scratching direction provides several test conditions, because of the indenter shape. Two scratching directions, $\theta = 0$ and π , were employed. After scratching, the damaged area was observed under an optical microscope (Keyence VH2250). When the damage initiation occurs, the critical load, P_c , was determined from the position of damage initiation.

3. Results and Discussion

3.1 Interfacial damage during the nanoscratch test

Figure 4 shows the profile of the scratching at $P_{\text{max}} = 5 \text{ mN}$ and $dL/dt = 10 \mu\text{m/s}$. The indented depth was plotted with respect to the scratch length ($\theta = 0$). The indented depth increases monotonically, and no sudden change in displacement is observed. At $L = 50 \mu\text{m}$, displacement changes discontinuously, but not dramatically.

Fig. 4 Scratch profile.

Fig. 5 microscopic image of interfacial damage due to nanoscratch.

Figure 5 shows the morphology of scratch at $P_{\max} = 5 \text{ mN}$ and $dL/dt = 10 \mu\text{m/s}$. In the early stage ($P_d = 0.44 \text{ mN}$), spot-like damage initiation was found below the scratch. Observation after a few days revealed discolored spot-like damage due to copper oxidation. Since the scratch did not penetrate the upper layer, copper oxidation took place on the interface. This implies that the internal damage initiates near the $\text{Si}_3\text{N}_4/\text{Cu}$ interface. No crack appears on the upper surface. In the present study, the damage occurring before the cracking of the upper layer is defined as damage initiation, and the critical load, P_c , was examined.

In Fig. 6, the critical load for damage initiation is plotted with respect to several test conditions under $\theta=0$. The horizontal axis indicates the maximum load. The scatter in each test condition ranges within 10%, and repeatability was confirmed. No dependency on the scratch rate is observed. This implies that the damage initiation is governed by time-independent phenomena. The critical load ranges from 0.4 mN to 0.6 mN. However, when the applied maximum load exceeds 2 mN, the critical load tends to saturate to a constant value. Meanwhile, Figure 7 shows the critical load at $\theta = \pi$. The critical load at $\theta = \pi$ is approximately two times that at $\theta = 0$. Since the edge of indenter is scratching the surface at $\theta = 0$, higher stresses are concentrated near the scratched area. On the other hand, in the case of $\theta = \pi$, the face of the Berkovich-type indenter is placed at the scratch front. When the scratching rate is low (1 $\mu\text{m/sec}$), the critical load of damage initiation saturates at a constant with respect to the loading rate. When the maximum load is small and the scratching rate is fast, the contact between the indenter and the surface is not static. On the other hand, when the applied load is sufficient and the scratch rate is low, the effect of the scratching rate on the critical load is small. In the present study, the mechanism of static damage initiation was examined.

Fig. 6 Critical load ($\theta=0$).

Fig. 7 Critical load ($\theta=\pi$).

3.2 Three-dimensional elastoplastic finite element analysis of the damage process.

As shown in Fig. 8, three-dimensional nonlinear finite element analysis was carried out for the stress field of damage initiation during the scratch test ($\theta=0$). A half model of localized volume near the scratching was constructed, and the length, width, and depth of the half model are 9, 4.5, and 10 μm , respectively. The model consists of an indenter and films on a substrate. The indenter was fixed in the vertical direction, and displacement u was applied horizontally on the upper side of the indenter. Meanwhile, the horizontal displacement was restricted, and pressure p , which is a load P divided by the area of the bottom, A , was applied normal to the bottom of the substrate. A commercial solver, Marc, and an eight-node solid element were used in this analysis. The number of elements and the number of nodes are 13,504 and 15,588, respectively. For the purpose of simplifying the FEA, the TaN layer fabricated in the scratch test specimen was ignored. In this analysis, the following two-step analysis was employed, because the scratch length analysis overwhelms the hardware resources in the FEA. In the first step, the pressure of the final 250 nm of the scratch was applied to the substrate without horizontal displacement of the indenter. Next, the pressure was increased to the critical load with a horizontal displacement of 250 nm. The minimum element size is set to 50 nm. The option of large deformation for elements was considered, because the indenter causes element distortion. Friction between the indenter and the surface of Si_3N_4 was not considered. The material properties used in the FEA are listed in Table I. Si_3N_4 and Cu films are assumed to be elastoplastic bodies, and other materials are assumed to

Table I Material properties			
Material	Young's Modulus E [GPa]	Poisson's ratio ν	Yield stress σ_Y [MPa]
Si ₃ N ₄	304	0.27	4000
Cu	130	0.34	440 ⁽¹²⁾
Si	170	0.3	
Indenter	1143	0.2

Fig. 8 Analysis model.

be isotropic elastic bodies. The yield stress of copper film was taken from a reference, in which the yield stress of a copper film having a thickness of 400 nm was evaluated from the nanoindentation test⁽¹³⁾. Using the method described in Reference (13), the estimated value of the yield stress of Si₃N₄ was 1/3 the hardness of the Si₃N₄ as determined from the nanoindentation test. Other material properties are taken from Reference (14).

First, the validity of the FEA was confirmed from the scratching profile. The scratching depth in FEA reached almost 50 nm. From Figures 4 and 5, when the first damage initiation occurs (Scratch distance is 20 μ m), measured depth was 60 nm. The difference between FEA results and experimental results is not so large. Figure 9(a) shows a cross-sectional contour map of shear stress in $\theta = 0$. The stress concentration of τ_{xy} appears in the Si₃N₄ film. In particular, the shear stress is concentrated behind the indenter on the Si₃N₄/Cu interface. When the interfacial stress concentration often appears forward of the indenter during the scratch test, large-scale delamination of the upper film occurs. However, the shear stress distribution suggests that the spot-like damage observed in the scratch test was caused by the stress concentration generated behind the indenter. Figure 9(b) shows the distribution of the plastic shear strain in the vicinity of the Si₃N₄/Cu interface. The strain concentrates on the contact area and in the copper film. Since the concentrated area was located in the front of the indenter, the effect of strain concentration on the interfacial damage initiation is not so significant. Therefore, the interfacial damage was generated by the interfacial stress concentration. Figure 10 shows the value of the stresses and strain averaged at the integrated points in each element. These values were plotted by reference to the grey scale indicated at the right of the plots. The element having the highest value of τ_{xy} is located at the scratch line

Fig. 9 Contour maps of shear stress and plastic strain near the indenter.

and behind the indenter. This agrees with the observed damage below the scratch line. The region size of the shear stress concentration ranges from 100 to 150 nm and is similar to that of the observed damage area. Figures 10(b) and 10(c) are grey-scale maps of τ_{zx} and σ_{yy} . Both stress concentrated areas are located forward of the indenter. In such cases, spot-like damaged areas tend not to form, because the damaged areas are expanded by the indenter. In addition, the τ_{zx} component concentrates near the scratch line. As a result of the initial indentation, the peak of the shear plastic strain is located behind the indenter. However, the location of the peak depends on the boundary conditions of the FEM, and the strain concentration appears before the scratching in the vicinity of the indenter. The scratch test initiated damage after the scratching. Therefore, the spot-like damage initiation was generated by the shear stress concentration after the scratching.

Figure 11 shows a grey-scale map of τ_{xy} for the case of $\theta = \pi$. The shear stress concentrates after the scratching. The peak value is higher than that concentrated in front of the indenter. Spot-like damage initiation resulted from not only the scratching by the edge of the indenter ($\theta = 0$), but also the scratching by the face of the indenter ($\theta = \pi$). In two cases, the damage was initiated by the stress concentration of τ_{xy} on the scratch line.

The interfacial shear stresses between Si_3N_4 and Cu along the scratch line are plotted in Fig. 12. In the graph, each curve ($\theta = 0$ or π) is plotted at the critical load, and the horizontal axis corresponds to the scratching direction. The value of the interfacial stress was evaluated by the extrapolation of the values of integrated points on the copper side. In the two scratch tests, the peak shear stress reaches almost the same value after scratching. This implies that spot-like damage initiates when the shear stress reaches the critical value. In this nanoscratch

Fig. 10 Distribution of stresses and strain near the $\text{Si}_3\text{N}_4/\text{Cu}$ interface in the case of $\theta=0$ (Side of Cu film).

test, the spot-like damage is independent of the failure of the upper layer, as described in the previous section, and its size is less than 1 micron. Therefore, the critical shear stress is related to the interfacial resistance to damage initiation. In the interface between Si_3N_4 (100 nm) and Cu (300 nm), the critical shear stress values are 239 MPa for the edge scratching and 241 MPa for the face scratching. This value, which is independent of the scratching behavior, characterizes the interfacial strength. This implies that the interfacial strength is evaluated from the nanoscratch test.

4. Conclusion

The interfacial damage process during a nanoscratch test was examined for multi-layered thin films. Nanoscratch tests were carried out on $\text{Si}_3\text{N}_4/\text{Cu}/\text{TaN}$ films on a silicon substrate, which is a typical structure for an LSI. Three-dimensional finite element analysis configuring the scratch test revealed the stress and strain fields near the interface where damages were generated. The obtained results can be summarized as follows:

- (1) During the nanoscratch test for $\text{Si}_3\text{N}_4/\text{Cu}/\text{TaN}$ films, the damage initiates near the $\text{Si}_3\text{N}_4/\text{Cu}$ interface before the large-scale failure of the upper film. The spot-like damage was scaled within 1 micron.
- (2) Scratching rate has a small effect on the critical load in damage initiation. In addition, as the applied load increases, the value saturates at a constant value in the case of edge scratching.
- (3) Three-dimensional FEA revealed that the shear stress, τ_{xy} , becomes concentrated after the scratching. Other stresses and plastic shear strain components become concentrated in front of the indenter.
- (4) Not only the edge scratching, but also the face scratching generated spot-like damage before large-scale fracture of the upper film. Although a higher critical load was measured from the face scratching test as compared to the edge scratching test, the spot-like damage initiates when the peak value of shear stress after the scratching reaches the critical value of 240 MPa.

Fig. 11 Distribution of τ_{xy} near the $\text{Si}_3\text{N}_4/\text{Cu}$ interface in the case of $\theta=\pi$ (Side of Cu).

Fig. 12 Distribution of τ_{xy} along the

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