Mixed-mode crack initiation at the edge of Cu/Si interface in nanoscale components

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Abstract The crack initiation behavior under the mixed-mode at the edge of a Cu/Si interface in nanoscale components has been investigated by a novel torsion method using a double nano-cantilever specimen consisting of a 20-nm-thick Cu layer and a Si substrate. By using this nano-cantilever torsion method, the shear stress is applied to the Cu/Si interface with the nanoscale stress concentration through the transverse arm. The mode mixity, which is the ratio of the shear stress to the normal stress, can be precisely controlled by changing the loading position. During the experiment, the crack is successfully initiated at the Cu/Si interface edge for various mode mixities by the developed method. The detailed stress fields along the Cu/Si interface at the critical loads for crack initiation are analyzed by the finite element method, and the stress concentration region near the interface edge in all specimens is within the scale of 100 nm. The critical normal stress and maximum shear stress at crack initiation have a circular relation.

Keywords Nanoscale, Mixed-mode, Crack initiation, Interface, Torsion test

1. Introduction

Micro- and nano-mechanical systems and electronic devices consist of dissimilar components, of which size is on the nanometer scale. Since intrinsic bi-material interfaces are inevitably introduced into these devices, deformation mismatch between the dissimilar materials induces a stress concentration at the interface. Thus, the interface is one of the potential sites of fracture [1–3].

Since the stress concentration region is proportionally scaled down for the shrinkage of component size, the stress concentration region at the interface edge is confined to the nanoscale in advanced devices [4–5]. On the other hand, as the state of internal stress is complex in micro-devices, the interface cracking usually takes place under a mixed-mode due to asymmetric loading across the interface and the difference in elastic properties [6]. Thus, to ensure the reliability of these devices, it is important to investigate the mixed-mode fracture criterion under a nanoscale stress concentration. Many investigations for various mode mixities have been conducted for macroscale materials [7], and they revealed that the critical normal and shear stresses for the interface fracture exhibit an elliptical relationship [7]. However, due to the difficulty in handling and applying effective loads to nanoscale specimens, conventional methods [8–11] cannot be applied in fracture experiments of nanoscale components. Recently, although the interfacial crack initiation by nanoscale stress concentration has been experimentally studied by a bending method using a straight nano-cantilever [12–14], the normal stress dominated the crack initiation at the interface edge, *i.e.*, mode-I fracture. It is thus necessary to develop a suitable method for performing mixed-mode fracture experiments on nanoscale components and to investigate the criterion in the mixed-mode due to the nanoscale stress concentration.

In this study, we develop a nano-cantilever torsion method and use it to perform crack initiation experiments for the interface between 20-nm-thick copper (Cu) film and silicon (Si) substrate under a 100-nm-scale stress concentration with different mode mixities.

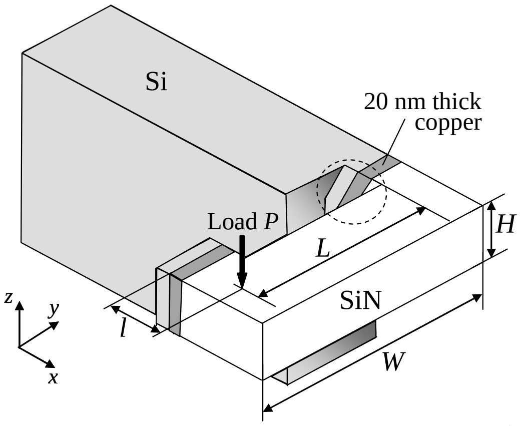
2. Experiments

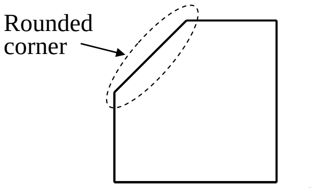
2.1. Tested material

The nano-cantilever torsion specimen is cut from a multi-layered material, *i.e.* silicon/copper/silicon nitride (Si/Cu/Si_3N_4). A Si(100) wafer surface is cleaned by inverse sputtering to remove the native oxide layer and then a 20-nm-thick Cu layer is deposited by radio-frequency magnetron sputtering in a 0.67 Pa argon atmosphere. An approximately 1000-nm-thick silicon nitride (SiN) layer is subsequently deposited by the same method without breaking the vacuum. This study focuses on the interface between the Cu layer and the Si substrate.

2.2. Nano-cantilever torsion specimen

Figure 1 schematically illustrates the nano-cantilever torsion specimen used to investigate the mixed-mode cracking at the Cu/Si interface edge in the present study. As shown in Fig. 1(a), the nano-cantilever torsion specimen is a three-dimensional bent structure in the shape of "⊐" that has three arms (see top view of specimen in Fig. 1(b)). The Cu/Si interface is located at arm ①. The load is applied to the SiN layer in arm ③ by a diamond loading tip to stress the Cu/Si interface by a torque and bending moment transferred through arm ②. In addition, to eliminate the strong stress singularity at the sharp corner of the rectangular cross-section of arm ① and obtain a controllable mild concentrated stress field, the inner corner is rounded by focused ion beam (FIB) processing, as shown in the cross-sectional view of Fig. 1(a). Three specimens with different size are prepared and their scales are summarized in the table of Fig. 1(a).

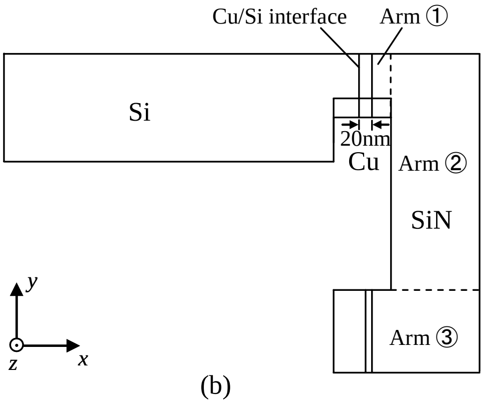




Cross section of arm ①

Specimen No.	*L*	*l*	*W*	*H*
T1	2075	657	2879	760
T2	2850	600	1661	656
T3	2391	115	2915	584

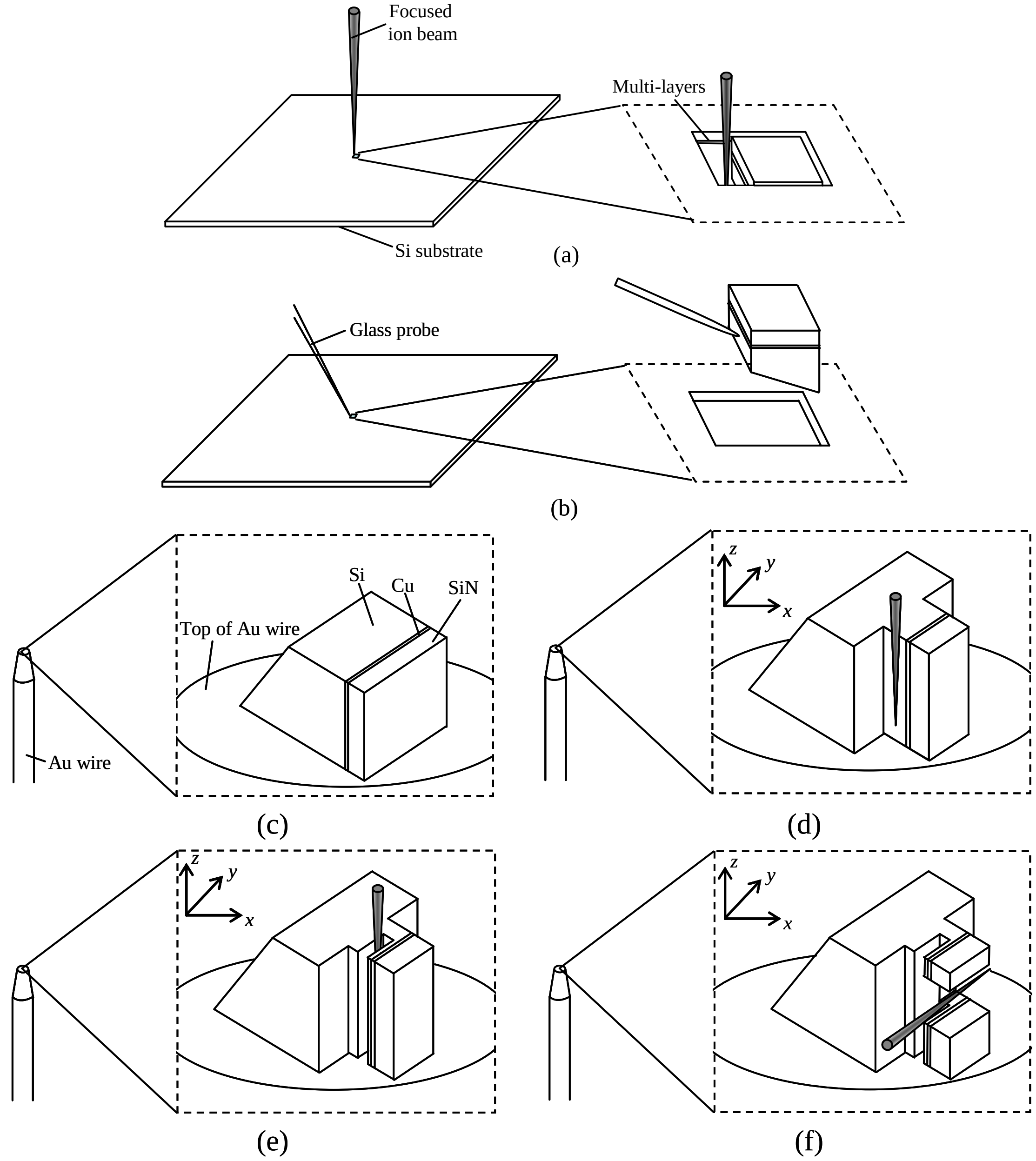
(a)

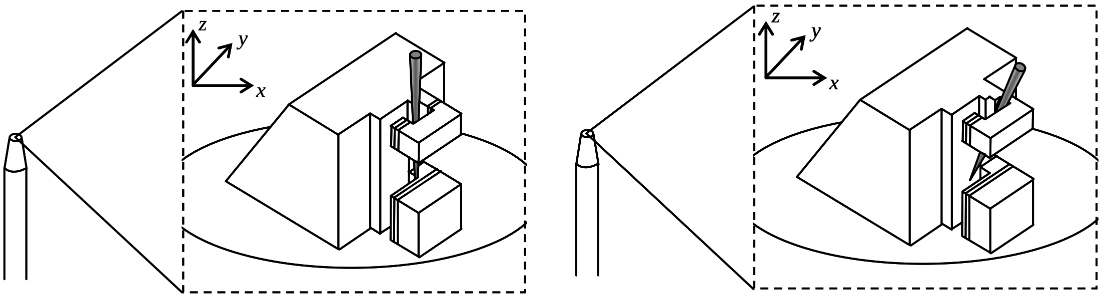


(b)

Figure 1 (a) Schematic illustration of a nano-cantilever torsion specimen with 20-nm-thick Cu thin film and its dimensions. (b) Top view of specimen. The dimensions in the table are given in nanometers.

Figure 2 schematically illustrates the procedure used to fabricate the nano-cantilever torsion specimen. As it has three-dimensional shape, the process is much more complex than that for previous straight-cantilever specimen. Before the sample fabrication, passivation layers of gold (Au) and carbon (C) are formed on the SiN layer to protect the specimen from damage during processing. After a cubic block with the side length of about 10 μm is cut from the multi-layered material by a focused ion beam (FIB) (JEOL Ltd., SMI9200) (Fig. 2(a)), it is picked up by a glass probe manipulator (Fig. 2(b)) and is carefully glued to the flat top of a gold (Au) wire with a diameter of 0.25 mm (Fig. 2(c)). The block is thinned in the *y*-direction (Fig. 2(d)) and an L-shaped extension that includes multilayer Si/Cu/SiN is formed (Fig. 2(e)). After thinning the L-shaped extension in the *z*-direction to form a cantilever (Fig. 2(f)), the inner middle part of the long arm of the L-shaped cantilever is cut to expose the inner Cu/Si interface edge (Fig. 2(g)). The inner corner of the upper edge of the Cu/Si interface is rounded (Fig. 2(h)). Finally, the Au and C layers are removed. The gallium (Ga) ion beam energy is set to 30 kV and the beam current is controlled between 5 pA and 5 nA depending on the fabrication precision.





(g) (h)

Figure 2 Schematic illustrations of the procedure used to prepare nano-cantilever torsion specimen. (a) A block is cut from a multi-layered material by FIB. (b) The block is manipulated using a glass probe. (c) The block is mounted on the top of an Au wire. (d)–(h) The shape of the nano-cantilever torsion specimen is fabricated.

Figure 3 shows a scanning electron microscopy (SEM) micrograph of a nano-cantilever torsion specimen. It reveals that there is no damage on the Cu/Si interface edge before the experiment.

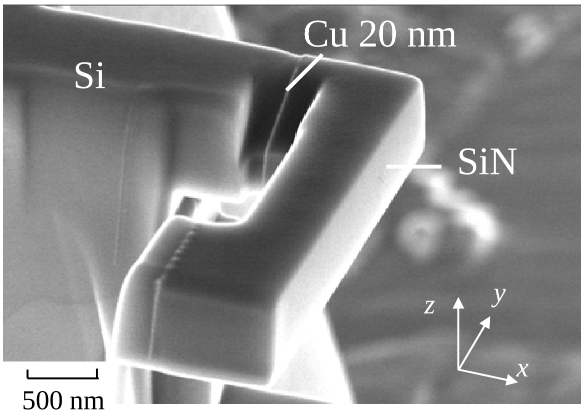


Figure 3 SEM image of a nano-cantilever torsion specimen (T1).

2.3. Loading apparatus

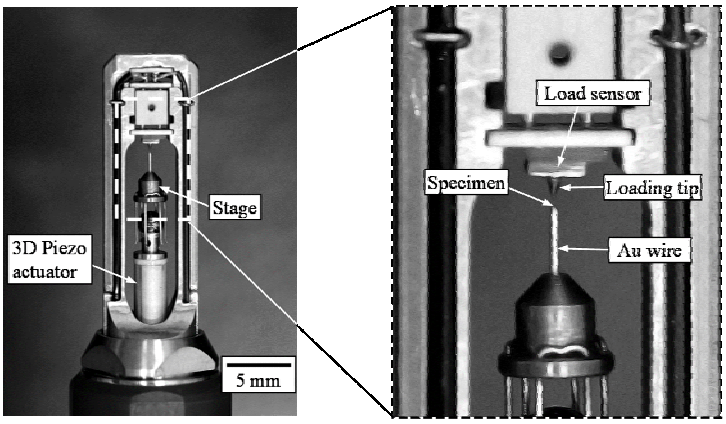


Figure 4 Mechanical loading apparatus used in the mixed-mode interface cracking experiments of nanoscale specimen.

Figure 4 shows the minute mechanical loading apparatus (Nanofactory Instruments AB, SA2000N) used in this study. The apparatus, which consists of a movable sample stage and a diamond loading tip with a load sensor, is built into a transmission electron microscopy (TEM) holder. The gold wire with a nano-cantilever torsion specimen mounted on top is attached to the sample stage, which is actuated three-dimensionally by a piezoelectric actuator. The load resolution of the sensor is 0.1 μN and the alignment resolution of the piezoelectric actuator in each direction is approximately 1 nm.

3. Analytical procedure

To inquire the stress distribution along the Cu/Si interface at crack initiation, elasto-plastic analysis is performed by the finite element method (FEM) using the commercial software, ABAQUS. Figure 5 shows typical finite element meshes for specimen T1. The configuration of the simulation model is reconstructed using three-dimensional computer-aided design (CAD) software on the basis of 3D analysis of SEM and TEM micrographs. The area near the free edge of the Cu/Si interface, where the stress concentration is expected, is carefully divided into fine meshes. A perfect constraint condition is imposed on the back and bottom ends.

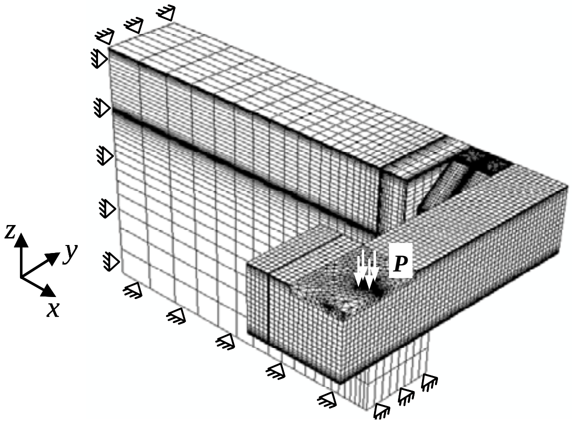


Figure 5 Typical finite element meshes for numerical analysis (specimen T1)

Because the Si substrate is a single crystal, it was treated as an orthotropic elastic material. The Young's modulus of the SiN layer which affects the stress distribution along the Cu/Si interface is measured by an indentation test. Table 1 lists the elastic constants of the component materials.

Table 1 Elastic constants of materials

Material	Young's modulus (GPa)		Poisson's ratio
Cu	129		0.34
SiN	197		0.27
Au	78		0.44
C	400		0.30
	C_{11} (GPa)	C_{12} (GPa)	C_{44} (GPa)
Si	167.4	65.23	79.57

Since the yield stress of the Cu layer is much lower than the Si substrate (σ_y > 3.4 GPa [15]) and the SiN layer (σ_y > 8.4 GPa [16]), only the Cu layer is subjected to elasto-plastic deformation during these experiments. The constitutive relation has been experimentally obtained in a previous study [13], and is given by:

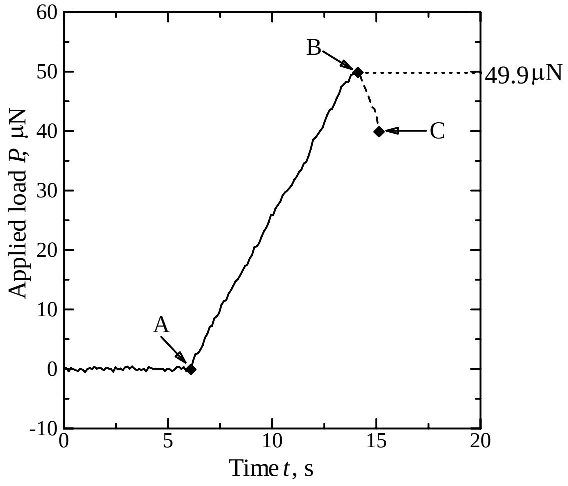
$$\sigma = \begin{cases} 129000\varepsilon & \text{for } \sigma \le 765\,\text{MPa} \\ 3316\varepsilon^{0.332} & \text{for } \sigma \ge 765\,\text{MPa} \end{cases} \tag{1}$$

where σ and ε are the von Mises stress and strain, respectively.

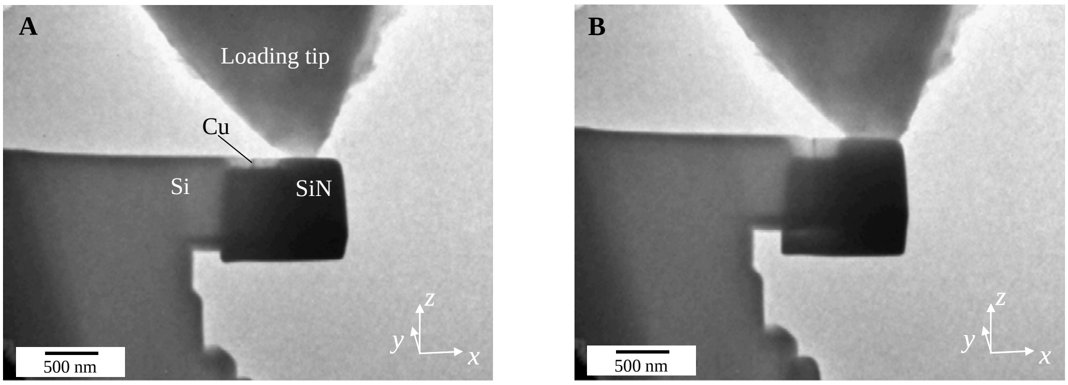
In addition, the residual stresses introduced during processing are measured experimentally (760 MPa for Cu layer; –290 MPa for SiN layer) [13] and are taken into account in the FEM analysis. After corresponding residual stresses are uniformly imposed on Cu and SiN layers respectively, they are relaxed by the FEM calculation.

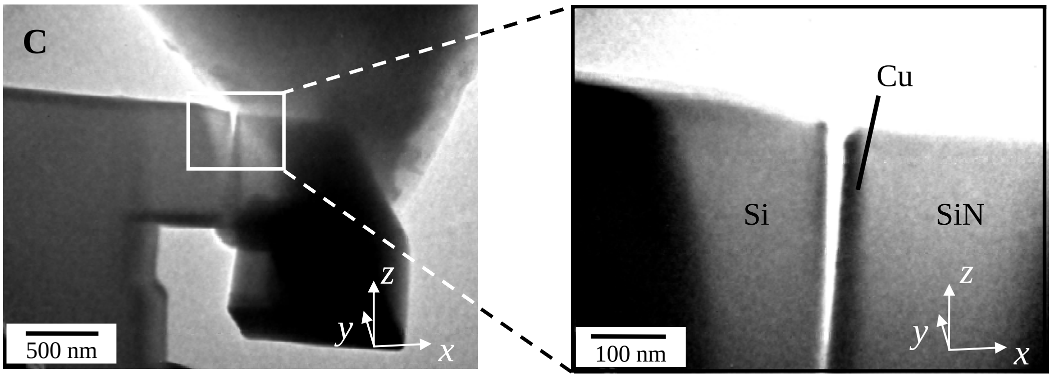
4. Results and discussion

Figure 6 shows the loading curve (P–t) obtained for specimen T1 and TEM micrographs of the specimen at three specific loading levels (A, B, and C). As shown in Fig. 6(a), after the loading tip hits the SiN layer at point A, the applied load, P, increases monotonically up to a peak magnitude of 49.9 μN (point B), after which it abruptly decreases (point C). Figure 6(b) clarifies that no crack initiation is recognized before the peak load while the specimen has an interface crack after. That is to say, the crack initiates at point B at the top edge of the Cu/Si interface and instantly propagates along the interface. A magnified image of the Cu/Si interface edge after crack initiation (at point C) indicates that no dissimilar material remains on either delaminated surface, which confirms the pure interface cracking. Similar behavior is also observed in the other specimens. Thus, the peak load for crack initiation, P_C, is defined as the critical load. Table 2 lists the magnitude of P_C, which shows that it depends greatly on the specimen geometry.



(a)





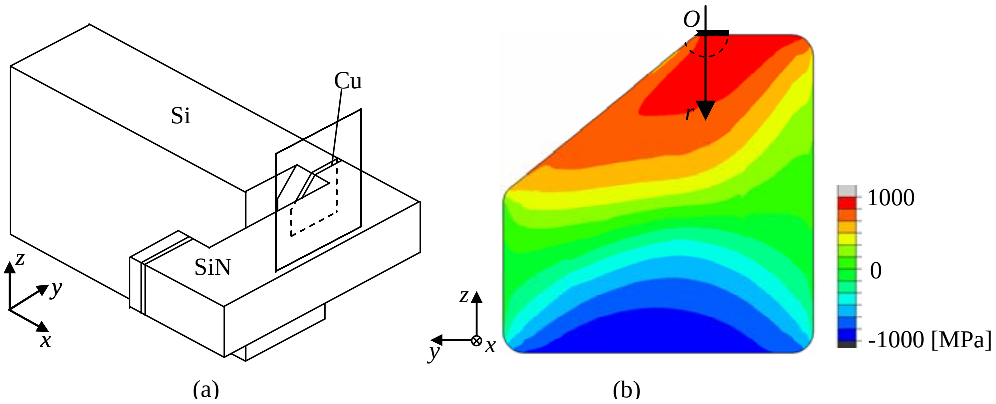
(b)

Figure 6 (a) Load–time relationship and (b) *in situ* observations of interfacial fracture of specimen T1.

Table 2 Critical load, P_C, for crack initiation at interface edge

Specimen No.	Critical load, P_C, μN
T1	49.9
T2	25.6
T3	12.0

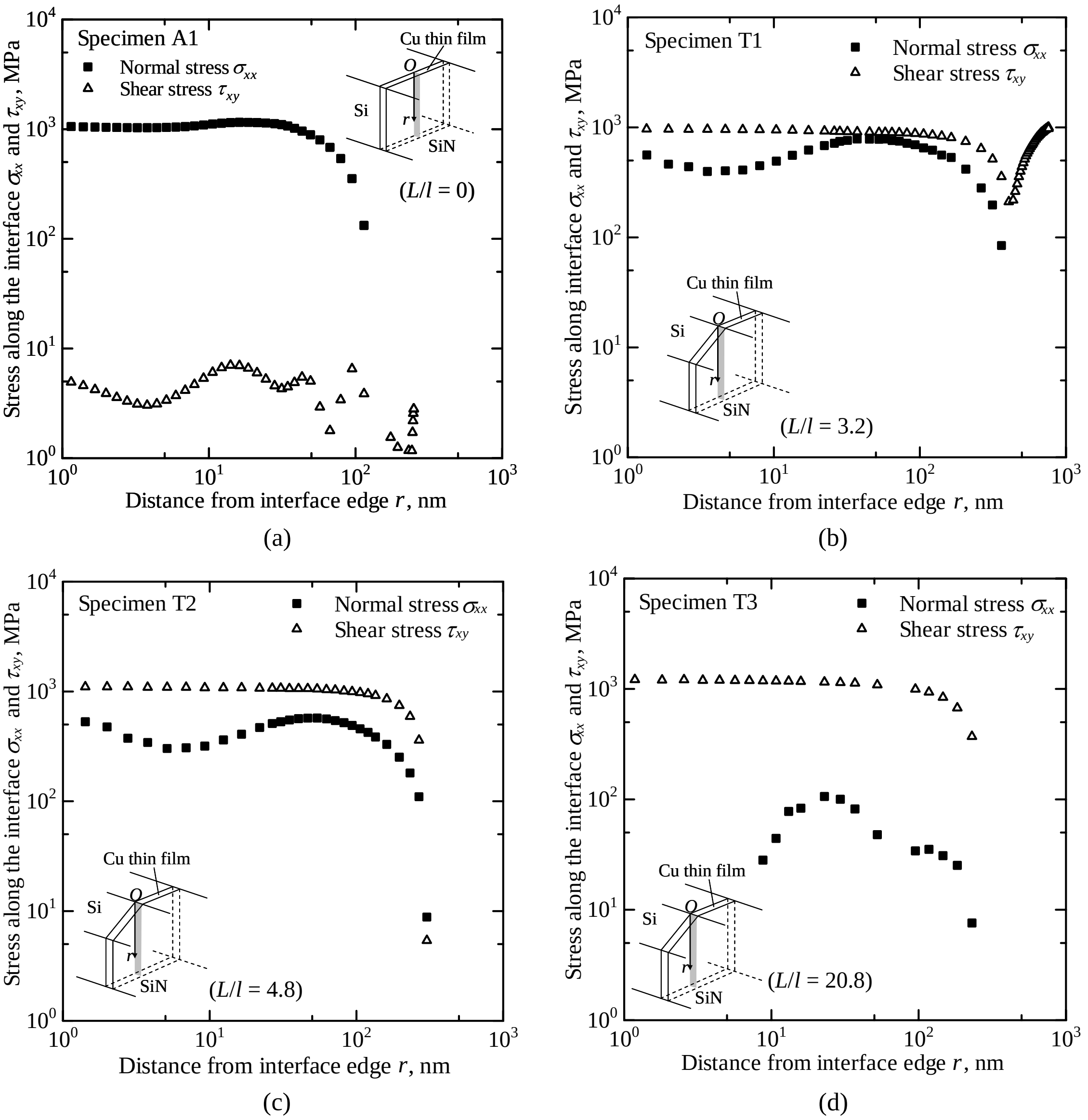
Figure 7 shows the distribution of shear stress τ_{xy} on the Cu/Si interface of a nano-cantilever torsion specimen, T1. It shows that the stress concentrates at the adjacent area of the rounded inner corner and its size is in the scale of 100 nm. Similar stress distribution on the Cu/Si interface is also found in other specimens. Thus, we set the origin, *O*, on the rounded corner to be the site of crack initiation. Since the stress is almost constant within the area of 50 nm away from the origin (the area in the dashed circle in Fig. 7(b)), we take the *r*-axis along the loading direction.



(a) (b)

Figuer 7 Distribution of shear stress τ_{xy} on the Cu/Si interface of a nano-cantilever torsion specimen.

Since FEM analysis on stress distribution of the Cu/Si interface indicates that the shear stress τ_{xy} concentrates at the site of crack initiation and much larger than other shear stress components, following discussions focus on the shear stress τ_{xy} and the normal stress σ_{xx}. Figure 8 shows the distributions of the normal and shear stresses, σ_{xx} and τ_{xy}, on the Cu/Si interface along the *r*-axis at the critical loads for crack initiation in different specimens. For comparison, the critical interfacial stress distribution of a straight-cantilever specimen (A1) is referred in Fig. 8(a). In straight-cantilever specimen A1 (Fig. 8(a)), the normal stress is two orders of magnitude larger than the shear stress, which indicates that the normal stress dominates crack initiation at the Cu/Si interface edge. Figures 8(b)–(d) reveal that with changing the loading position, *i.e.*, the magnitude of *L*/*l*, different ratios of the shear stress to the normal stress are realized. In specimen T1, the shear stress is comparably large with the normal stress (Fig. 8(b)), and a larger ratio of shear stress to normal stress is achieved in specimen T2 (Fig. 8(c)). In specimen T3, the shear stress is 10 times larger than the normal stress (Fig. 8(d)). It should be noted that the stress concentrated area is about 100 nm in all specimens. These indicate the success of the nano-cantilever torsion method for conducting fracture experiments under different mode mixities with the nanoscale stress concentration.



(a) (b)

(c) (d)

Figure 8 Critical stress distributions at crack initiation on the Cu/Si interface near the edge of (a) straight-cantilever bending specimen and (b)–(d) nano-cantilever torsion specimens.

Since both torsion and bending moments are applied to the Cu/Si interface with the nano-cantilever torsion method, the relation between the maximum shear stress and the normal stress is discussed to extract the mixed-mode fracture criterion of interfacial cracking. Here, it should be noted that the magnitude of maximum shear stress is almost same with that of τ_{xy} because of the domination of τ_{xy} for crack initiation compared with other shear stress components. On the other hand, a previous experimental study [14] revealed that the concentrated stress field of 30 nm from the edge of the Cu/Si interface dominates crack initiation at the edge of interface between the 20-nm-thick Cu layer and the Si substrate. Thus, the averages of the normal and the maximum shear stresses over the area of 30 nm away from the interface edge are used to represent the dominant stress components for crack initiation; they are denoted by σ_{mc} and τ_{mc}, respectively. Figure 9 shows the plot of the normal stress σ_{mc} versus the maximum shear stress τ_{mc} for all four specimens (A1, T1, T2, and T3). In the straight-cantilever specimen A1, τ_{mc}/σ_{mc} is nearly zero, indicating the domination of normal stress for the crack initiation at the interface edge. In the nano-cantilever torsion specimens, *i.e.*, T1, T2, and T3, τ_{mc}/σ_{mc} is 1.6, 2.5, and 19, respectively, which shows that the crack is initiated at the interface edge with different mode mixities. By fitting the experimental results with the least-squares method, an elliptical relation that is commonly used for macroscopic materials and a circular relation between the σ_{mc} (MPa) and τ_{mc} (MPa) are obtained and respectively depicted in Eqs. (2) and (3):

$$\left(\sigma_{\mathrm{mC}}/1106\right)^2+\left(\tau_{\mathrm{mC}}/1129\right)^2=1 \tag{2}$$

As shown in Fig. 9, by calibrating with experimental results (circle points), the circular relation (red dash line) gives simple correspondence.

$$\sigma_{\mathrm{mC}}{}^2+\tau_{\mathrm{mC}}{}^2=1143^2 \tag{3}$$

This indicates that the mixed-mode fracture criterion for interfacial cracking in nanoscale components simply exhibits a circular relation between the critical normal stress and maximum shear stress.

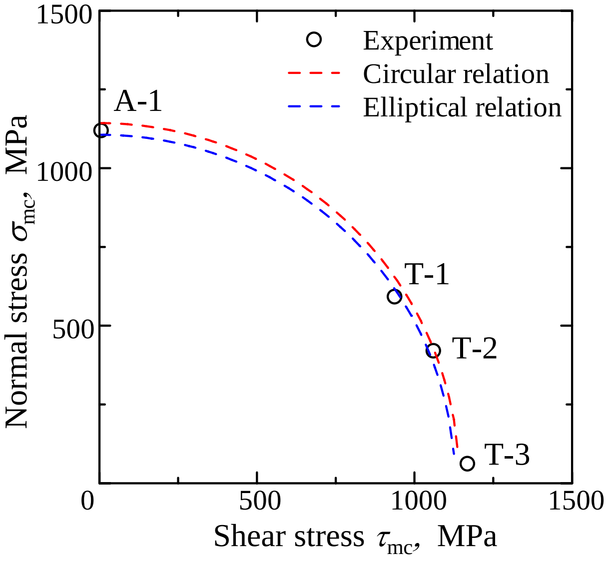


Figure 9 Critical relation of interfacial cracking under the mixed-mode.

5. Conclusions

In order to investigate the mixed-mode crack initiation due to the nanoscale stress concentration at the edge of the Cu/Si interface, we developed a methodology and conducted the fracture experiments using novel nano-cantilever torsion specimens with the interface between a 20-nm-thick Cu layer and a Si substrate. The results are summarized below:

(1). A novel nano-cantilever torsion method was developed that can apply the shear stress to the Cu/Si interface for crack initiation at the interface edge. The ratio of the critical shear stress to the normal one at crack initiation, *i.e.*, the mode mixity, could be controlled by varying the loading position. FEM analysis revealed that the concentrated stress region near the interface edge was within the scale of 100 nm in all specimens.

(2). In the nano-cantilever torsion specimens, the crack was successfully initiated from the edge of the Cu/Si interface under different ratios of the critical shear stress to the normal stress. The corresponding critical load was measured by in situ TEM observations on the cracking.

(3). The mixed-mode fracture criterion for interfacial cracking in nanoscale components is represented by a circular relation between the critical normal stress and maximum shear stress.

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