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Piezoelectric in situ transmission electron microscopy technique for direct observations of fatigue damage accumulation in constrained metallic thin films

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A piezoelectric in situ transmission electron microscopy (TEM) technique has been developed to observe the damage mechanism in constrained metallic thin films under cyclic loading. The technique was based on the piezoelectric actuation of a multilayered structure in which a metallic thin film was sandwiched between a piezoelectric actuator and a silicon substrate. An alternating electric field with a static offset was applied on the piezoelectric actuator to drive the crack growth in the thin metallic layer while the sample was imaged in TEM. The technique was demonstrated on solder thin films where cavitation was found to be the dominant fatigue damage mechanism.


Stress-induced phenomena in metallic thin films have received great attention, in the past two decades largely due to reliability concerns in microelectronic devices, and more recently to interests in understanding the scaling effect in mechanical behavior of metals.1–3 The reliability concerns stem from potential failures of interconnects in microchips by stress-induced voiding or electromigration, under high-current densities and/or mechanical stresses.4–8 The source of the stress may be residual from the film deposition process or external when the interconnect is subject to external thermal, electric, and mechanical excitations. Under these stresses, the scaling effect becomes significant as the one dimension of the solid is reduced to a scale comparable to the characteristic dimensions related to distribution and motion of the crystalline defects.

Stresses in microelectronic interconnects may be static or cyclic in nature, depending on the device fabrication and service conditions. While most studies on stress-induced voiding of interconnects were made under a static or monotonic loading, the interconnect metal may experience cyclic stresses when the substrate on which the interconnect resides is deformed under electric, thermal, and mechanical cycling. For example, in surface acoustic wave (SAW) devices, the interconnect is stressed cyclically as a surface wave is excited on the piezoelectric substrate to cause cyclic deformation of both the substrate and interconnect.9–11 Since SAW devices have to operate at very high frequencies (i.e., 2 GHz in telecommunication devices), the accumulative effect of stress cycles can lead to fatigue failures. In fact, interconnect failures by voiding have been reported and are considered a major reliability issue in SAW devices.9–11

To determine the stress response of thin films, various methods have been developed that are based on applying mechanical stresses to freestanding films,12–14 substrate-constrained films,15–20 and sandwiched films.21 Among them, in situ transmission electron microscopy (TEM) techniques are particularly useful because of their high-resolution and dynamic imaging capability. Both the straining stage13,14,16,17 and hot stage15,16–20 have been used to perform tensile deformation and thermal cycling of thin films. However, the conventional TEM straining stage is limited to monotonic tensile tests. With the hot stage, the temperature may be cycled to apply thermal cycling onto TEM specimens,18–20 but the frequency of thermal cycling is rather low and the temperature rises can change the microstructure of the thin films dramatically.15 In this letter, we report an in situ TEM technique based on two experimental techniques that we recently reported, i.e., a field-driven in situ TEM technique developed for ferroelectrics22,23 and a piezoelectric actuator designed to drive fatigue crack growth in a multilayered structure.24 The technique was applied to a solder thin film sandwiched by Si and a piezoelectric ceramic, and the cyclic-stress-induced voiding of the solder thin film was found to be the dominant fatigue damage mechanism.

The substrates used in this study were single-crystal Si wafers and polycrystalline lead zirconate titanate (PZT) plates. The PZT plate, 1.5 mm thick, was poled along the thickness direction. The plate was polished using diamond paste down to 0.25 μm. Following ultrasonic cleaning in acetone and ethanol alcohol, the Si and PZT substrates were placed in a magnetron sputtering machine and sputter cleaned. A Ti adhesion layer and Cu metallization, 20 nm thick each, were then deposited on the substrates and covered by a thin Au capping layer ~10 nm thick. During sputtering, Ar gas was used as the ionization source at a pressure of 2 × 10⁻³ Torr and the deposition rate was about 2 Å/s. After sputter deposition, the samples were transferred to a thermal evaporator where a thick layer of pure Sn, ~300 nm thick, was deposited at pressures below 2 × 10⁻⁶ Torr. Another Au capping layer was then deposited on the Sn surface to protect it from oxidation in air. The coated Si and PZT sheets with lateral dimensions of 12 mm×10 mm were aligned face to face, clamped, and heated in a vacuum oven (vacuum better than 10⁻⁷ Torr), where the Sn was melted at 280°C for 15
min, before the sample was cooled down in vacuum.

Thin cross-section slices with thickness less than 0.5 mm were cut from the sandwiched thin-film specimen. Both broad faces of the slices were ground and then polished with diamond paste to a final thickness of 100–120 μm. Disks of 3 mm diam were ultrasonically cut with the metal film running across the center of the circular disk. The remaining steps for the preparation of the TEM samples were similar to the procedures used for the ferroelectric ceramic samples in our previous field-driven in situ TEM studies. The finished specimen is shown schematically in Fig. 1. The ion milling perforation was controlled to occur in Si close to the metal film. When the edge of the perforation reached the metal film, ion milling was stopped. In this way, a sample with a continuous and electron transparent metal film was obtained.

Details of the power delivery system were the same as the one used in our previous field-driven in situ TEM studies. The solder thin film and the Au film on the top surface of the TEM specimen served as the two electrodes to drive the PZT actuator. The gap between the two electrodes was about 200 μm, which resulted in field strength of 1.0 MV/m at an external voltage of 200 V. The actuation mechanism for the PZT actuator is illustrated in Fig. 2. An alternating electric field with a static field offset, as shown in Fig. 2(a), was applied to the PZT actuator. A sinusoidal wave form at a frequency of 30 Hz was used as the control signal from the function generator. The minimum amplitude of the cyclic field, $E_{\text{min}}$, was fixed at zero and the maximum amplitude, $E_{\text{max}}$, was increased step by step, with 9000 cycles at each step, until crack growth was observed. As indicated in Fig. 2(b), the applied electric field, $E$, was parallel to the poling direction, $P$. Since the piezoelectric coefficient $d_{31}$ of this ceramic is $-285 \times 10^{-12}$ C/N, the applied field produced a lateral contraction in the PZT, as schematically shown by the dashed line in Fig. 2(b). The lateral contraction imposed a combined compressive and shear loading onto the interfacial thin film, resulting in tensile and shear loading at the left and right poles of the central perforation. As a pre-crack, the central perforation develops a mixed opening and shear crack tip field upon ac excitation of the piezoelectric actuator, placing the solder thin film under cyclic tensile and shear stresses.

When an electric field was switched on, three major changes were observed in the TEM specimen. First, a crack extending about 10 μm developed in the metal film close to the Si/metal interface after 9000 cycles at $E_{\text{max}} = 1.0$ MV/m. Such a field corresponds to a lateral piezoelectric strain about $3 \times 10^{-4}$ in the free state. At this strain level, the piezoelectric actuator provided a strain energy release rate of about 1 J/m$^2$, which was sufficient to produce fatigue crack growth along a polymer–metal interface. As soon as the crack appeared, $E_{\text{max}}$ was reduced to 0.4 MV/m in order to slow down the crack growth for better image recording. Second, voids were formed in the film at a distance about 0.6 μm ahead of the crack [Fig. 3(a)]. With continued cycling, the void population increased and individual voids grew and eventually linked up to form a continuous crack [Fig. 3(b)]. Such a ductile mode of fracture involving void nucleation, growth, and coalescence is not unexpected in Sn because of the high homologous temperature ($T/T_m = 0.6$) at which the stress was applied. Third, the shape of the void was acicular, bounded by sharp wavy edges (Fig. 4).

Since the stress state in the film was a mixed tensile and shear, the nonspherical nature of the void is believed to be largely the result of the gradual distortion caused by the shear deformation, although the atomic mechanism of the

![FIG. 1. TEM specimen configuration and multilayered metallic film.](image1)

![FIG. 2. Schematic for the piezoelectric actuation.](image2)
shear deformation is yet to be clarified. The wavy edges resembled the surface morphology leading to the formation of the fatigue striations in metals. If the highly elongated void is treated as a crack, the wavy edges may be taken as a result of the repeated sharpening and blunting of the crack tips in the same mechanism as the way in which fatigue striations were formed.25

In summary, an in situ TEM technique was developed to study the fatigue damage mechanism in constrained metal thin films. The technique was based on combining a field-driven in situ TEM technique with a piezoelectric actuator, which was integrated into the TEM specimen. Compared to previous methods for studying mechanical behavior of thin films, the piezoelectric in situ TEM technique offers the advantages of in situ TEM observations, capability to apply cyclic stresses in TEM at high frequencies, and decoupling of mechanical loading from thermal loading. The technique was applied to a sandwiched thin solder film sample where void nucleation, growth, and crack development were directly observed. Although its feasibility was demonstrated with solder thin films, the technique is expected to apply to other metallic thin films used in microelectronic interconnects, e.g., by adjusting the sequence, composition, and thickness of the individual films in the multilayered specimen used in this study.

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