Available online at www.sciencedirect.com

ScienceDirect

Scripta Materialia 65 (2011) 596–599

Scripta MATERIALIA

www.elsevier.com/locate/scriptamat

Fatigue-resistant silicon films coated with nanoscale alumina layers

E.K. Baumert, P.-O. Theillet and O.N. Pierron*

G.W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA 30332-0405, USA

Received 13 May 2011; revised 22 June 2011; accepted 23 June 2011
Available online 29 June 2011

The fatigue properties of monocrystalline Si thin films coated with nanoscale alumina layers (ranging from 4.2 to 50 nm) are compared to those of uncoated Si films, in air at 30 °C, 50% relative humidity (RH) and 80 °C, 90% RH. The presence of alumina coatings results in more than two orders of magnitude longer fatigue lives, even though subcritical cracking of alumina occurs. The effects of the alumina coating thickness on the overall fatigue degradation behavior are discussed.

Keywords: Fatigue; Thin films; Coatings; Silicon

The fatigue properties of silicon thin films are strongly influenced by the environment (see Refs. [1,2] and references therein). Specifically, Si films do not fatigue in ultrahigh vacuum [3], while their fatigue lives (N_f) in air can be reduced by orders of magnitude when increasing humidity levels under stress amplitudes in the GPa range [4–6]. Several mechanisms have been proposed to explain this behavior, including the reaction-layer fatigue mechanism for stress amplitudes substantially lower than the film's strength [7] and, more recently, time-dependent crack extension of Si for stress amplitudes commensurate with the film's strength [8]. From an engineering perspective, understanding these fatigue mechanisms can help developing robust approaches for reliable microelectromechanical system devices in harsh environments. Recently, we showed that ~20 nm atomic layer deposited (ALD) alumina coatings had beneficial effects on the fatigue degradation properties of polysilicon films [9], which we attributed mainly to the oxygen and water diffusion barrier properties of alumina [10]. Cracking of the ALD coatings was, however, suspected to occur, a result that is expected to be highly dependent on coating thickness [11]. The thickness of the ALD alumina coating should therefore be optimized to obtain crack-free effective diffusion barriers and, ultimately, Si films that are resistant to fatigue degradation in harsh environments. This letter reports on an experimental investigation of the effect of nanoscale alumina coating thickness on the overall fatigue degradation properties of the coated Si films. While other studies have already focused on the mechanical robustness of ALD coatings [12–14], this study focuses specifically on the fatigue degradation properties of nanoscale ALD coatings and its effects on the fatigue of the underlying Si films, in both mild and harsh environments.

ALD alumina coatings of four different thicknesses were deposited on 10 μm thick monocrystalline Si fatigue resonators fabricated from the 20th and 29th runs of the SOIMUMPs process[1] (see Fig. 1(a)), and the fatigue properties of the coated resonators were compared to the uncoated ones [6] in two different environments: 30 °C, 50% relative humidity (RH) and 80 °C, 90% RH. The alumina layers were deposited with an in-house ALD tool, using trimethyl aluminum (TMA) and water. The deposition temperature was 200 °C, and the pulse durations for water and TMA were 10 and 17 ms, respectively. The numbers of cycles for the four alumina thicknesses were 30, 90, 179 and 358, corresponding to nominal thicknesses of 4.2, 12.6, 25 and 50 nm, respectively. The measured thicknesses (using scanning electron microscopy (SEM) images of fractured devices) were 7 ± 2, 17 ± 5, 40 ± 5 and 50 ± 5 nm, indicating deposition rates that may be larger than the nominal values[2]. Fatigue tests were performed on 40 kHz resonators driven at resonance, leading to fully reversed

* Corresponding author. Tel.: +1 404 894 7877; e-mail: olivier.pierron@me.gatech.edu

[1] No difference in behavior was observed between the two runs, based on resonant frequency measurements. Also, the surface roughness of the coated (see Fig. 1) and uncoated [6] devices is similar.

[2] Given the uncertainty in the measured values using SEM, the nominal values are used throughout the rest of the manuscript.

1359-6462/$ - see front matter © 2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.scriptamat.2011.06.034

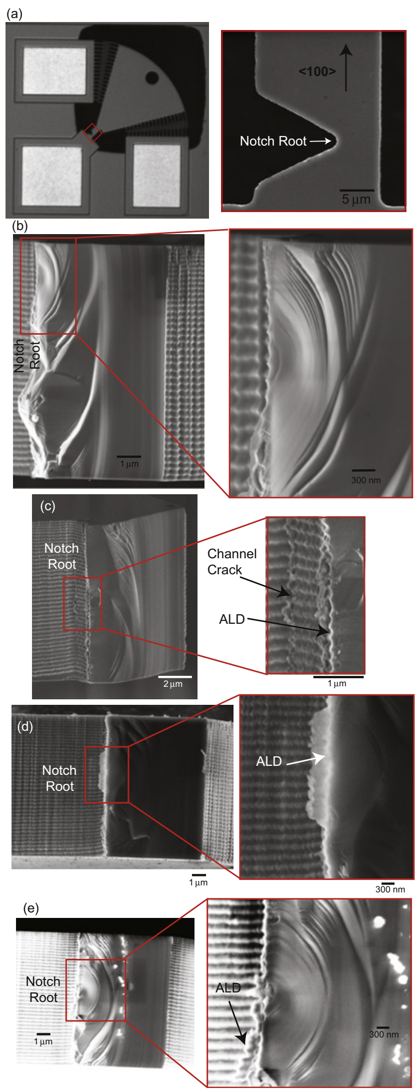

Figure 1. (a) Optical image of the 40 kHz fatigue resonator, along with an SEM image of the notched cantilever beam. (b–e) SEM images of fracture surfaces (coated devices) failed at 80 °C, 90% RH: (b) 4.2 nm alumina, $\sigma_a = 2.57$ GPa, $N_f = 7.7 \times 10^8$; (c) 25 nm alumina, $\sigma_a = 2.42$ GPa, $N_f = 2.0 \times 10^9$; (d) 25 nm alumina, $\sigma_a = 2.97$ GPa, $N_f = 7.7 \times 10^9$; (e) 50 nm alumina, $\sigma_a = 2.46$ GPa, $N_f = 1.4 \times 10^9$.

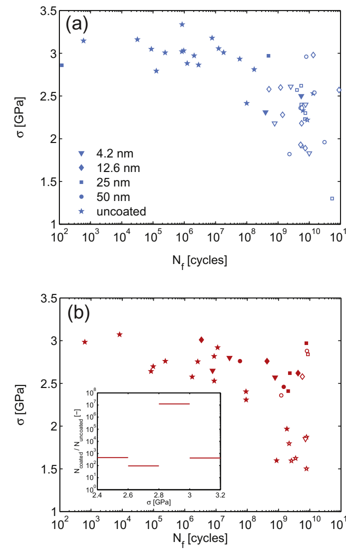

Figure 2. S–N_f curves at (a) 30 °C, 50% RH and (b) 80 °C, 90% RH, for coated and uncoated Si films; empty symbols represent runout tests. The inset in (b) shows the ratio of minimum N_f between coated and uncoated Si films. The large increase in the 2.8–3 GPa range is mainly a result of the limited number of specimens in this stress range.

stresses (for Si) at the notch of the cantilever beam (see Fig. 1(a)). The resolution in stress amplitude (for Si) was ~0.05 GPa [6,8]. The resonant frequency (f_0) was also periodically monitored as a measure of damage accumulation, with a typical precision of 0.02 Hz (0.5 ppm).

The stress–life (S–N_f) fatigue curves for the coated and uncoated devices are shown in Figure 2(a) (30 °C, 50% RH) and Figure 2(b) (80 °C, 90% RH). At 30 °C, 50% RH, most of the coated devices did not fail before the tests were stopped at 10^8–10^{11} cycles, suggesting improved fatigue resistance over uncoated devices in the 2–3 GPa stress range. A comparison between coated and uncoated devices at 80 °C, 90% RH shows a significant increase in N_f with the presence of alumina coatings. As shown in the inset of Figure 2(b), the minimum N_f for coated devices is at least two orders of magnitude larger than the minimum N_f for uncoated devices, for stresses ranging from 2.4 to 3.2 GPa. Examination of the fracture surfaces of fatigued coated devices (see Fig. 1(b)–(e)) reveals features very similar to that of uncoated devices [5,6], implying that fatigue degradation of Si does occur and that the governing fatigue mechanisms for Si may be identical (albeit operating at a lower rate). Specifically, the fatigued Si films exhibit highly localized, semi-elliptical, mirror-like regions, whose dimensions appear to scale with N_f. Only the inner part of the mirror-like region is associated with fatigue damage (critical crack sizes for uncoated Si films range from 20 to 90 nm for the investigated stress range [6,8]), and appears to be smooth (with the exception of one examined device; see Fig. 1(e)). The SEM images also reveal evidence of coating delamination near the failure origin for 25 and 50 nm coatings (see Fig. 1(c)–(e)), which are not observed for thinner coatings (see Fig. 1(b)). While only suspected in a previous study [9], Figure 1(c) clearly shows crack channeling through almost the entire Si film thickness. The feature labeled as "channel crack" in Figure 1(c) was not observed on uncoated devices [6], or on coated devices away from the notch (large stress area). The details of ALD cracking and the influence of coating thickness can be assessed based on f_0 evolution measurements.

The f_0 evolution of the coated specimens is characteristically different from that of the uncoated specimens, a result previously attributed to the cracking of the coatings [9]. Most of the coated specimens experience a steep initial decrease in f_0, followed by a more stable f_0 evolution (a "plateau" regime, consisting of either a small continuous increase or decrease in f_0), while some fail before reaching a plateau (see Fig. 3(a)). Instead, uncoated devices typically show a steady decrease (no observed increase) in f_0 throughout their lifetimes (see Fig. 3(a)) [6]. In most cases, the total decrease in f_0 (Δf_0) for coated devices is considerably larger than that of uncoated devices for a given stress (see Fig. 3(b)). The