IN SITU SEM HIGH-THROUGHPUT CYCLIC TESTING OF FREESTADING THIN FILMS

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Abstract

This work presents a small-scale high-throughput technique to characterize the cyclic behavior of freestanding thin films. The technique consists of the microfabrication of a Si carrier composed of an array of grips and freestanding dogbone thin films and of the automated *in situ* Scanning Electron Microscope (SEM) fatigue testing of the microfabricated carrier. The Si carrier functions as a nanomechanical testing device in which multiple dogbones can be simultaneously and independently tested under the same applied mechanical conditions. As a proof-of-concept, the fatigue behavior of nanocrystalline Al thin films was investigated. The technique allows for the simultaneous evaluation of crack nucleation and propagation across the fatigue life of several dogbones, facilitating the understanding of deformation mechanisms in nanocrystalline metals and providing statistically significant data. This technique reduces total testing time by orders of magnitude and allows for the investigation of the stochastic variability in fatigue failure. The current technique can be further expanded to account for different materials, new geometries and different loadings modes.

1. Introduction

Small-scale mechanical testing has been traditionally challenging due to difficulties in sample preparation/handling and load alignments. Microscale fatigue testing proposes an even larger challenge due the strenuous and tedious repetition of single fatigue experiments, which tend to be extremely time-consuming. Therefore, there is immense value in developing new techniques that can increase the efficiency of fatigue testing with high-throughput experimentation.

Over the last decades, high-throughput mechanical experimentation has matured at the macroscale (e.g. grips that sequentially perform tensile tests on coupons of dogbones). The advances of microfabrication techniques have also allowed the expansion of high-throughput experimentation towards the mechanical testing of thin films and small-scale materials. For example, high-throughput fatigue experiments on thin films deposited on Si vibrating cantilever and high-throughput sequential tensile test on microfabricated dogbones. In the work presented here, high-throughput experiments are performed in parallel and in an automated manner (significantly reducing total testing time), and on freestanding samples (decreasing the complexity in experimental analysis).

2. Results

The microfabricated devices (Fig. 1) are the result from microfabrication processes that involve DC sputtering, double-sided lithography and wet/dry etching. The devices are placed onto a custom–built tensile frame for *in situ* SEM testing and are loaded under tension-tension fatigue through pins at the frame.

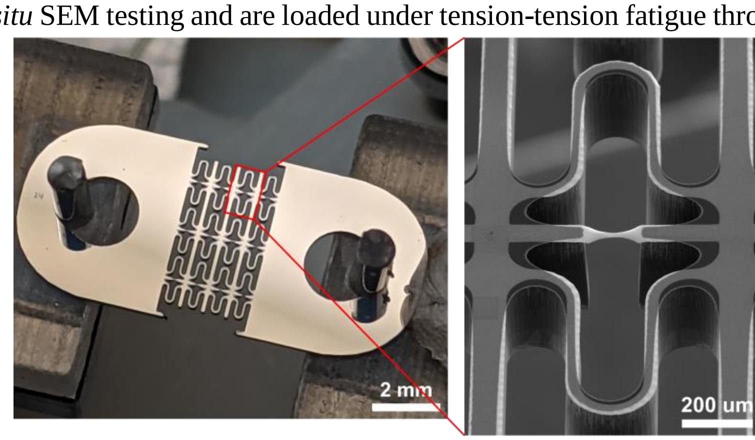

Fig.1 – Resultant microfabricated devices for high-throughput testing

Using the automated and high-throughput *in situ* tests, the fatigue behavior of nanocrystalline Al was characterized with only four tests at different strain amplitudes (Fig. 2a), collecting fatigue life data for approximately 30 thin film samples. These experiments highlight the great degree of stochastic variability in fatigue failure for samples tested at the same loading conditions. The samples tested at 0.44% strain amplitude, for example, exhibited fatigue failure lives that differed by more than two orders of magnitude, while samples tested at other strain amplitudes exhibited around one order of magnitude in variability.

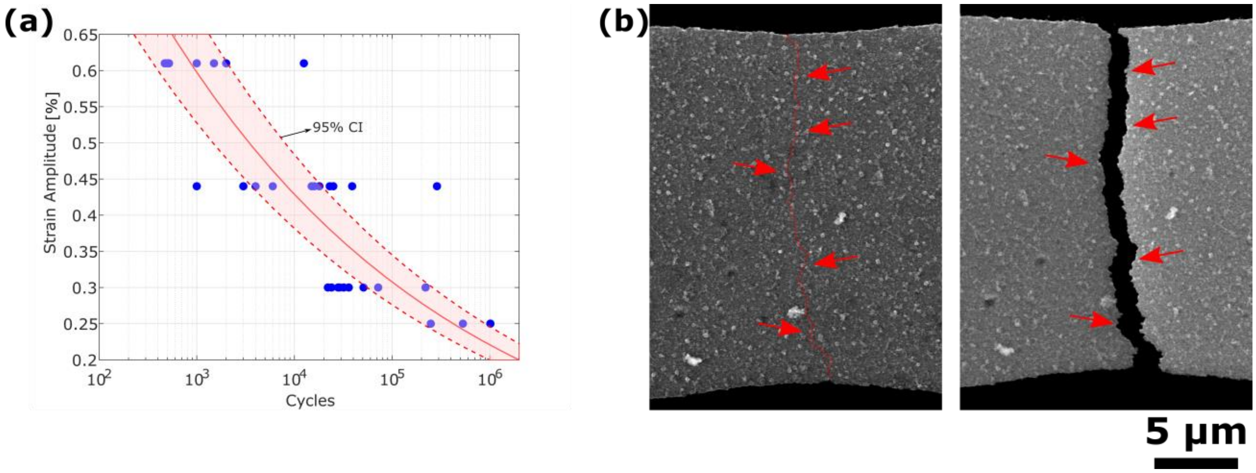

Fig. 2 – (a) Strain-life curve of the fatigue behavior for nanocrystalline Al. The solid red line represent the average fatigue behavior, while the shaded region represents the 95% Confidence Interval; (b) Crack propagation path in an Al dogbone in its original condition and after failure.

The *in situ* SEM high-throughput technique also allowed for the investigation of fatigue crack nucleation propagation with statistically significant data. Fig. 2b shows a representative case of fatigue crack behavior considering all ~30 samples tested. The crack path was added in red to its untested condition. The crack follows a very tortuous path, reminiscent of intergranular fracture, and tends to follow grains protruding from the Al thin film, highlighted by the red arrows. This grains serve as stress concentration sites and are the main mechanism for crack nucleation and propagation as no other salient features (extrusion, slip bands) were observed at the surface of the thin films.

3. Conclusions

In this work, a new methodology for the high-throughput cyclic testing of freestanding materials at the microscale is presented. Using a microfabrication process and an automated *in situ* SEM procedure, it was possible to efficiently characterize the fatigue behavior of nanocrystalline Al. Results highlight the stochasticity in fatigue failure and the collection of statistically significant data regarding the evolution of fatigue cracks in these thin films.

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