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An apparatus for performing microtensile tests at elevated temperatures inside a scanning electron microscope

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Abstract

In this paper, we introduce an apparatus to perform microtensile tests at elevated temperatures inside a scanning electron microscope. The apparatus has a stroke of 250 μm with a displacement resolution of 10 nm and a load resolution of 9.7 μN. Measurements at elevated temperatures are performed through use of two silicon-based micromachined heaters that support the sample. Each heater consists of a tungsten heating element that also serves as a temperature gauge. To demonstrate the testing capabilities, tensile tests were performed on submicron Cu films at various temperatures up to 430 °C. Stress–strain curves show a significant decrease in yield strength and initial slope for the samples tested at elevated temperature, which we attribute to diffusion-facilitated grain boundary sliding and dislocation climb.
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Keywords: In situ tension test; High-temperature deformation; Thin film; Grain boundary sliding; Creep

1. Introduction

Thin metal films with thicknesses well below 1 μm are widely used as functional and structural elements in microelectronic devices and large-scale integrated circuits, thin-film solar cells [1], electrical sensors [2,3], and electronic textiles [4]. Decreasing dimensions of microelectronic and micromechanical devices have motivated research on the micro- and nanoscale mechanical behavior of materials. Much work has been dedicated to characterizing the mechanical behavior of thin metal films and it has been observed that the mechanical properties of thin films can be very different to those of their bulk counterparts. For example, experimental studies have shown that the flow stress, hardening rate and fatigue life exhibit a strong size-dependence [5–13]. With conventional mechanical testing methods used for bulk materials it is difficult to obtain precise stress–strain curves for thin films and capture this size-dependence. Much attention has consequently been directed toward experimentally measuring the mechanical properties of thin films by a variety of techniques [14–17]. Most of these studies successfully investigated the effects of microstructural length scales and sample size on the mechanical properties of thin films at room temperature. Length-scale effects at elevated temperatures remain an unexplored area.

Prior studies of the mechanical behavior of thin films at elevated temperatures focused on films deposited on substrates [18–22]. However, recent work has revealed that the tensile and fatigue behavior of metallic thin films on substrates may differ substantially from the behavior of

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freestanding thin films [6,23–26]. Thus, testing freestanding thin films at elevated temperature is a critical step towards understanding their intrinsic behavior.

Testing of freestanding thin films has historically been challenging due to the inherent difficulties associated with handling and gripping of the samples. The basic requirements for accurate elevated temperature mechanical testing, including a uniform temperature distribution throughout the sample, accurate measurement of the temperature, and a controlled environment to prevent sample oxidation, are further complicated by the small sample dimensions. As a result, only a few studies report mechanical testing of freestanding thin films at elevated temperatures [27–34]. These studies expanded our understanding, but were limited to films with thicknesses above several microns [30–33] or to temperatures lower than 200 °C [27–29,34].

In this paper, we present a tensile tester with 9.7 μN load resolution and 10 nm displacement resolution that can be used inside a scanning electron microscope (SEM). Measurements at elevated temperatures are performed through use of two silicon-based micromachined heaters that support the sample. Each heater consists of a tungsten heating element that also serves as a resistance thermometer. To demonstrate the testing capabilities, tensile tests were performed on submicron Cu films at various temperatures up to 430 °C.

2. Tester description and thermal modeling

2.1. In situ SEM tensile tester and calibration

The tensile tester was designed to cover a broad range of displacements (tens of nm to hundreds of μm) and loads (tens of μN to 10 N), and to work inside the chamber of a SEM. These design requirements were met through use of capacitive displacement gauges and a load cell that consisted of a compliant double leaf spring. The tester is shown schematically in Fig. 1. As illustrated in the figure, an *xyz*-axis microstage is used to align tester and sample. The sample is deformed using a piezo nanopositioning actuator (PI P-622K067) with an external amplifier/servocontroller for closed-loop control (PI E-665). This actuator is capable of imposing displacements as large as 250 μm with a resolution better than 10 nm. The actuator displacement, δ_1, scales with the voltage applied to the actuator and is measured using an internal displacement sensor. As the sample is loaded, the leaf spring deforms and the displacement of the spring, δ_2, is measured using a capacitive displacement sensor (Capacitec HPB-200A). This displacement sensor has a working range of 600 μm and a resolution of 10 nm. The load on the sample is determined from the displacement of the leaf spring and its spring constant k. To prevent interference by the electron beam inside the SEM, the displacement sensor is shielded by an aluminum enclosure.

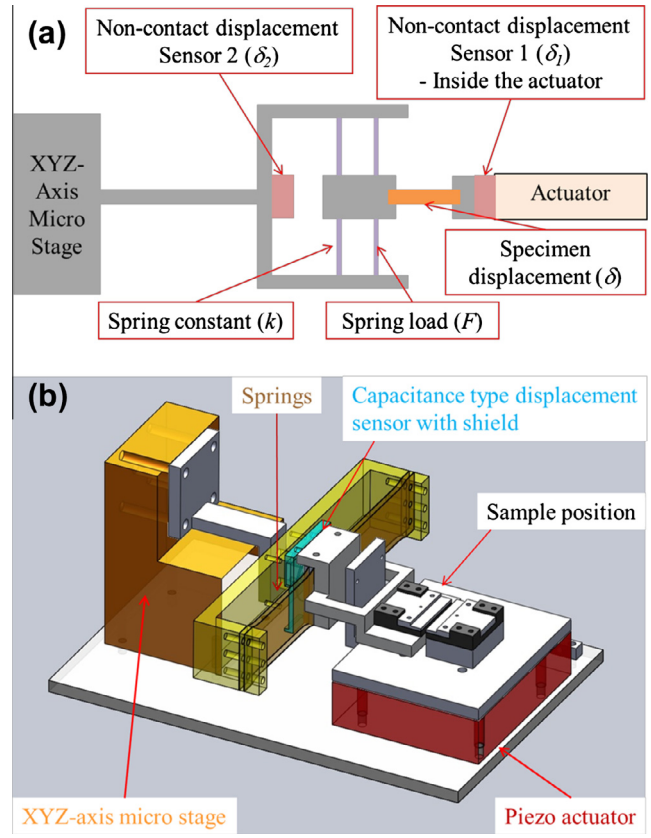

Fig. 1. Schematic representation of the in situ SEM tensile tester: (a) top view showing how the load and displacement of the specimen are measured; and (b) isometric view.

The displacement imposed on the sample is given by the difference between the displacement of the actuator and that of the leaf spring:

$$\delta = \delta_2 - \delta_1, \tag{1}$$

while the load on the sample is given by:

$$F = k.\delta_2. \tag{2}$$

To calibrate the leaf spring, a 98 mN load cell (Cooper Instruments & Systems LFS 270 10G) was attached to the tester and the load–displacement characteristic of the leaf spring was measured. The spring was linear to within 99.7% for loads up to 0.098 N and had a spring constant of 966.6 ± 0.5 N m^{-1}. Given a displacement resolution of 10 nm, this translates to a load resolution of 9.7 μN. The resolution of the load cell can be further improved through use of a more compliant leaf spring, although any gain in resolution would decrease the maximum load that can be imposed. Conversely, the maximum load can be increased to 10 N, the maximum value allowed by the piezo actuator, if a stiffer leaf spring is used. The various components of the tester were controlled and all signals were recorded with a personal computer equipped with a National Instruments USB-6281 data acquisition card (DAQ) and running the program Labview.

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