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NOVEL MEMS APPARATUS FOR INSTIU THERMO-MECHANICAL TENSILE TESTING OF MATERIALS AT THE MICRO- AND NANO-SCALE (PREPRINT)

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14. ABSTRACT

We present, for the first time, a MEMS-based test methodology that potentially enables elevated-temperature mechanical tensile testing of nano- and micro-scale samples within a SEM or TEM (T > 500°C). Importantly, the test methodology allows for the samples to be fabricated separately from the MEMS-apparatus, a significant advancement from other test devices developed by some of the present authors [1]. Therefore the test methodology should be applicable to the study of a wide range of materials. Other advancements found in the methodology include a co-fabricated force calibration device, and a builtin thermocouple sensor to measure the stage temperature close to the sample.

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NOVEL MEMS APPARATUS FOR IN SITU THERMO-MECHANICAL TENSILE TESTING OF MATERIALS AT THE MICRO- AND NANO-SCALE

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ABSTRACT

We present, for the first time, a MEMS-based test methodology that potentially enables elevated-temperature mechanical tensile testing of nano- and micro-scale samples within a SEM or TEM (T > 500°C). Importantly, the test methodology allows for the samples to be fabricated separately from the MEMS-apparatus, a significant advancement from other test devices developed by some of the present authors [1]. Therefore the test methodology should be applicable to the study of a wide range of materials. Other advancements found in the methodology include a co-fabricated force calibration device, and a built-in thermocouple sensor to measure the stage temperature close to the sample.

INTRODUCTION

At the micro- and nano-scale, materials behavior can strongly depend on the scale of both internal microstructural features as well as the external dimensions of the structure in question. Microstructural length scales may include features such the grain size, the distance between impurities [2], and the layer thickness in a multilayered material, to name a few. With regards to the sample geometry, decreasing size results in an increase in the surface-to-volume ratio of the material. These surfaces and interfaces can affect the active deformation mechanisms and their dynamics, which may introduce new mechanisms of deformation. These surface- dominated properties may also be time and temperature dependent. As a result, bulk material properties cannot be extrapolated to the diminutive size-scales that are of interest to MEMS and NEMS, and thus the mechanical properties of materials with micro- or nano-scale dimensions need to be directly measured. A major challenge lies in measuring such properties, and also in understanding the mechanisms that control size-dependent mechanical properties.

In an earlier study, some of the present authors developed a MEMS stage that allowed for uniaxial tensile testing of ultra-thin free standing films (30 nm thick and larger) that was capable of operation within either a SEM or TEM [3]. This previously-developed MEMS-stage enabled the measurement of the stress-strain response of the films while also allowing for direct visualization of the films during testing. The force on the sample was determined by image-based measurement of the deflection of a force-sensing beam, while the sample deformation was determined by image-based measurement of the change of position of two gauges attached to the ends of the sample. A unique feature of the stage is that any misalignment error in loading is reduced by five orders of magnitude through the use of alignment flexures. This ensures precise uniaxial loading of the sample. The stage was employed to test aluminum and gold film samples with grain sizes ranging from 10-300 nm, and film thickness ranging from 30-300 nm. The experiments revealed unusual properties in nano-grained metal films, and helped uncover new fundamental mechanisms of deformation that occur in nanoscale microstructures; for example, the recovery of plastic strain with time [1], and non-linear elasticity in nanocrystalline metals [3].

However, the stage described above has some limitations because the sample and the stage are co-fabricated. First, this limits the choice of materials to be tested to those that can be deposited or grown on silicon and patterned. Also, the sample thickness is constrained to the typical range of thin films. Furthermore, each stage can only be used once. The new MEMS apparatus presented in this paper overcomes these limitations.

THE NEW TESTING APPARATUS

The test methodology described herein allows for testing of samples that are fabricated separately from the MEMS stage. The test apparatus consists of a MEMS tensile stage and a heating stage. Figures 1a and 1b show the Si MEMS tensile stage without a sample. The stage contains two grips with etched or focused ion beam (FIB)-fabricated wedges to hold a dog-bone shaped sample that is manufactured separately. One of the wedge grips is attached to the force sensing beam, while the other is attached to a set of beams and a U-beam that ensure alignment and uniaxial loading of the sample. The tensile sample is placed into the wedge grips using a micro-manipulator. The large etched holes on the outer ends of the MEMS-stage connect the stage to a macroscopic piezo-electric actuated test frame using rigid pins. In order to apply uniaxial tension to the sample, one end of the stage is held fixed while the other end is moved by the piezoelectric actuator. Force on the sample is measured from the deflection of the force sensing beam (Figure 2). There are two gauges in the stage. One provides the displacement of the force sensing beam, the other provides the relative displacement between the two wedge grips.

Procedure for calibration of spring constant of the force sensing beam

The spring constant of the force sensing beam is calibrated using a co-fabricated calibration device (Figure 1a). The calibrator consists of a leaf spring. Its spring constant can be readily determined using a commercial

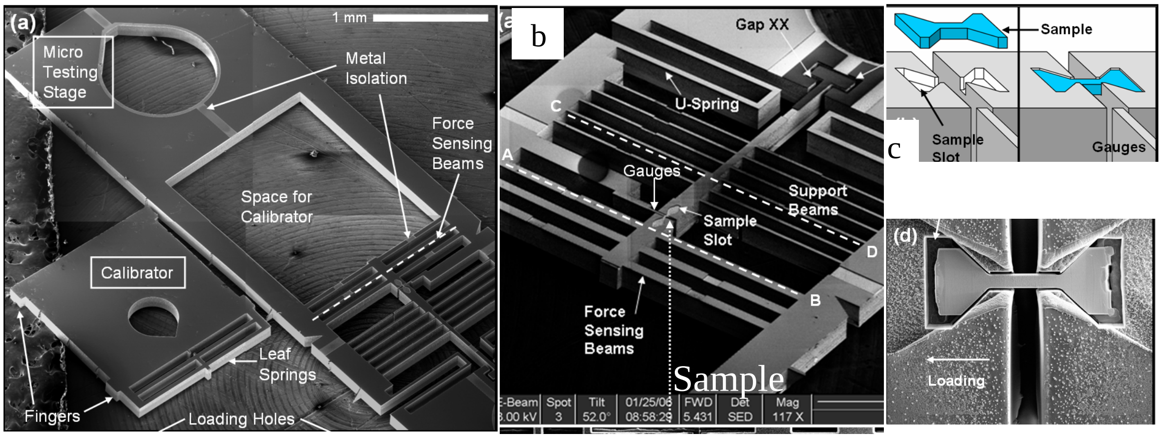

Figure 1. (a) SEM image of the MEMS tensile stage and the calibration device. The spring constant of the leaf spring of the calibrator is measured by a nano indenter. (b) Close-up image of the stage. (c) Schematic of sample loading. (d) Top view of the loading stage and the nickel-base superalloy sample fabricated by FIB.

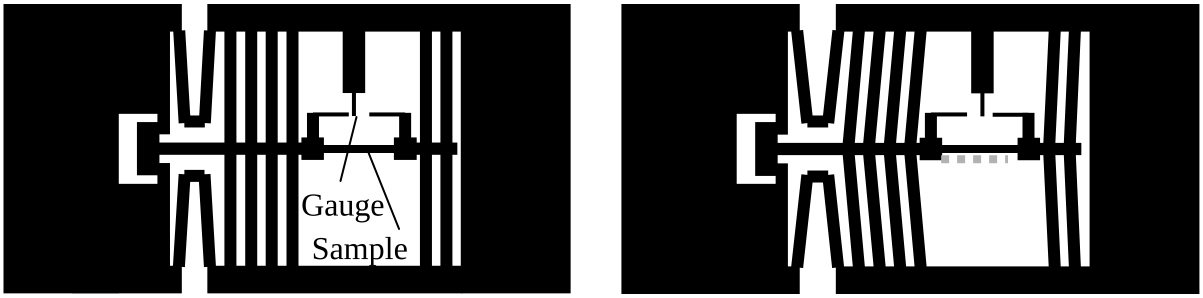

Figure 2. An unloaded (left) and a loaded (right) sample. The dotted line on the right diagram shows the original length of the sample. The force on the sample and the strain are measured from the change in the gauge positions.

nanoindentation system. Once the spring constant of the calibrator is determined, the calibrator is placed in the designated space of the MEMS stage where it can be placed in direct contact with the force sensing beam. Next, the calibrator is pushed by a micromanipulator to displace the force sensing beam. The spring constant of the force sensing beam is simply determined from the force imparted by the calibrator divided by the relative deflection of the force sensing beam.

Heating stage

In order to provide a capability to measure material properties at elevated temperatures, a micro-heater has been designed and tested that works with the MEMS testing stage (Figure 3). The micro-heater consists of a Si substrate with a patterned Cr micro-coil for Joule heating. For an elevated temperature test, the MEMS-stage is placed on top of the heating stage, and both the stage and sample are heated primarily by conduction. The temperature of the MEMS stage is measured by a built-in thermocouple. It consists of a silicon cantilever coated on one side by Cr. The mismatch between the thermal expansion coefficients of Si and Cr results in bending of the cantilever with increasing temperature, and the temperature is calculated from the relative deflection of the cantilever tip. The thermocouple is calibrated by measuring the tip deflection when the stage is heated to known temperatures. For calibration experiments, the stage temperature can be easily varied by using a hot plate or a convection oven. In a separate experiment, we have qualitatively verified the accuracy of the cantilever-based temperature measurement by melting materials with known melting temperatures. These materials include solder wire and aluminum, and the results from these experiments are shown in Figure 3. In order to further push the temperature capabilities of this type of micro-heater device, a silicon carbide substrate was substituted for the Si substrate. Here silicon carbide also served as the resistor for Joule heating. This heater was used to melt glass (Soda lime glass, softening temperature: 720C, Gold Seal Microslide) (placed on the heater), indicating that temperature can exceed 700°C.

Fabrication

The stage is fabricated from single crystal silicon by deep reactive ion etching. The process flow is given in Figure 4 with the details listed in the figure caption.

EXPERIMENTS WITH THE NEW STAGE

As a demonstration of the applicability of the stage, a nickel superalloy sample was tested under uniaxial loading. The specimen was made by FIB milling from a bulk crystal using techniques similar to those shown in [4]. The dimensions of the specimen gage section were approximately 25 μm in length, 10 μm in thickness, and 5 μm in width. The tensile axis of the sample was oriented parallel to the <123> direction, which corresponds to a single-slip orientation at the beginning of the experiment. Once the specimen was separated from the bulk crystal, it was transferred onto the wedge grips of the MEMS stage via micromanipulation within the FIB. The tension experiment was carried out in an SEM. The specimen and the displacement gauges were captured in the same SEM image so that both the stress-strain data and images of the deforming sample were captured together.

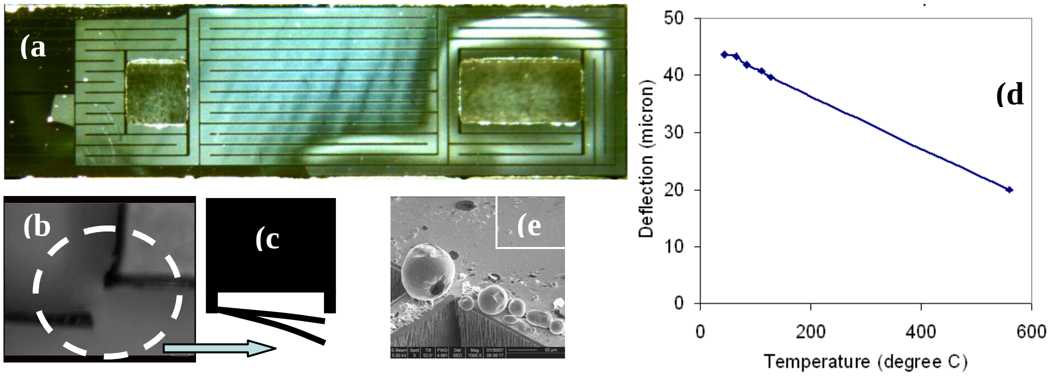

Figure 3. (a) The micro heater with Cr coils on a silicon substrate. The micro tensile stage is placed on top of the heater for uniaxial experiment. (b, c) Bimaterial (Si/Cr) temperature sensor built- in to the loading stage. The temperature is measured from the deflection of the beam (d). Temperature calibration of the sensor is shown (e) Al droplets produced by melting.

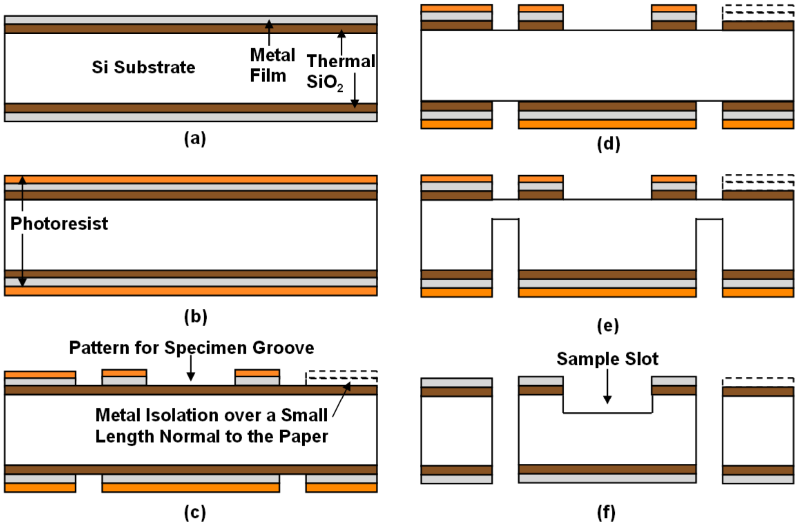

Figure 4. Schematic of process flow of the tensile stage: (a) Thermal growth of SiO_2 and deposition of aluminum layers on both sides of Si wafer; (b) Photoresist spin-coating on both sides of Si wafer; (c) Patterning of both sides by photolithography and liquid etching of metal layer (Here, the pattern of the bottom surface is a mirror image of the top pattern except for the patterns of the sample slot and the center gauge on top.); (d) Dry etching of SiO_2 layer on top and bottom by RIE; (e) Deep Si etching from bottom by ICP DRIE; (f) Shallow Si etching from top to form the sample slot and the center gauge by ICP DRIE and removal of Photoresist on top and bottom by O_2 plasma. Note that the metal layer is intended to serve as conductors for possible resistivity measurement of the sample.

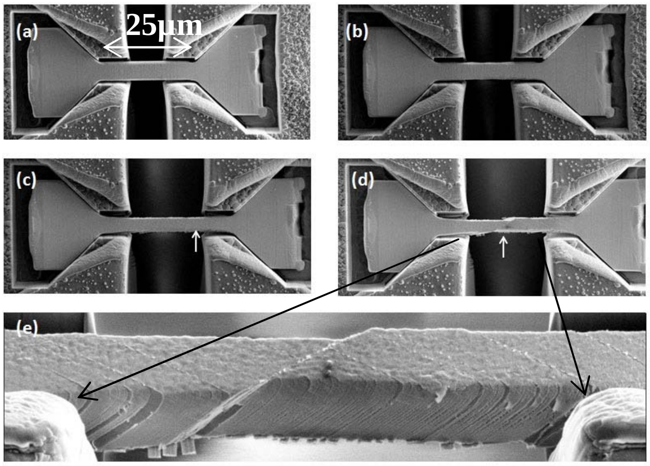

Figure 5. In situ, room temperature uniaxial test of a nickel-base superalloy sample using the MEMS-stage.

Figure 5(a-e) show images of the sample under increasing strains. After yielding, there is significant plastic flow with little strain hardening up to about 25% strain. In-situ SEM observation reveals that many slip lines are formed along the gage section that are consistent with activity from the primary slip system (white arrows). The sample then begins to strain harden until about 40% strain, at which time the stress begins to drop. The peak load coincides with the initiation of a second slip system (Figure 5e).

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