Final Report for Period: 12/2010 - 05/2011 **Submitted on:** 08/26/2011
Principal Investigator: Pierron, Olivier N. **Award ID:** 0952641
Organization: Georgia Tech Research Corp
Submitted By:
Pierron, Olivier - Principal Investigator
Title:
EAGER: Investigation of Environmental Effects on the Fatigue Degradation Properties in Metallic Nanostructures

Project Participants

Senior Personnel

Name: Pierron, Olivier
Worked for more than 160 Hours: Yes
Contribution to Project:
Advisor for Ms Baumert.
Development of MEMS-based nanomechanics testing technique
Creation of summer enrichment program FrAMED

Post-doc

Graduate Student

Name: Baumert, Eva
Worked for more than 160 Hours: Yes
Contribution to Project:
Fabrication of metallic nanostructures. MEMS-based experimental nanomechanics

Undergraduate Student

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Organizational Partners

Other Collaborators or Contacts

An informal collaboration was started with Dr Marc Legros, Senior Staff Scientist at CEMES (Toulouse, France). Dr Legros is an expert in in-situ TEM studies. Dr Legros is interested in using the MEMS devices developed through this grant to performed quantitative in-situ TEM studies of nanomaterials. Dr Legros already acquired all the equipment necessary for these experiments, including our MEMS devices and an electrical biasing TEM holder.

Activities and Findings

Research and Education Activities:

The research activities of this EAGER grant focused on the development of an experimental technique dedicated to measuring the fatigue properties of nanomaterials, specifically nanocrystalline metallic nanobeams. Au and Ni nanobeams were successfully fabricated (see Year 1 report). A MEMS-based nanoscale testing system was successfully developed to perform fatigue tests of the nanobeams (see Findings section).

The developed experimental technique will be used in future research to investigate the surface fatigue crack initiation mechanisms in nanocrystalline metals. Particularly, the technique will allow the monitoring of cyclic plasticity and the measurement of initiation fatigue life on the nanocrystalline nanobeams, as a function of maximum plastic strain, frequency and environment. The technique will also be used to perform quantitative in-situ TEM fatigue experiments to identify the fatigue crack initiation mechanisms.

The educational activities include the mentoring of one graduate student (Ms. Eva Baumert) currently supported by this project, and the development of a summer enrichment program for high school students. The PI hosted two high school teachers, Mrs Martha Magana and Cherisse Campbell in summer 2010 to work on the curriculum development of FrAMED: Failure Analysis for Materials Engineering Detectives. Mrs Campbell was supported with a RET from this EAGER grant. During the one-week-long program, the students will learn about the fundamental science related to the failure of materials in the form of short lectures and hands-on demos, and will have a chance to act as failure analysis experts in litigation cases whose outcome depends on the correct analysis of a failed object.

Findings: (See PDF version submitted by PI at the end of the report)

Our MEMS-based nanotensile testing setup can be used to measure the fatigue properties of nanocrystalline nanobeams (fatigue life, cyclic stress-strain curves, transient behavior). Fatigue tests can be performed for a wide range of maximum applied stresses and strains, frequencies (up to ~0.25-1Hz with the thermal-actuator-based MEMS devices), and environment. While the fatigue test presented in this report was performed in laboratory air, future tests can be performed in an environmental chamber to assess the influence of temperature and humidity on the fatigue properties. Last but not least, the same experimental setup can be used to perform quantitative in-situ TEM tests, using an electrical biasing TEM holder.

Training and Development:

One graduate student (Ms. Eva Baumert, second year graduate student) currently supported by this project is advised by the PI (Pierron). As part of the training and development process, the PI meets weekly with Ms Baumert to discuss her research efforts. In addition, Ms Baumert presented her previous work on the fatigue of Si thin films at the 10th International Fatigue Congress in Prague (Czech Republic) in June 2010. One high school teacher, Mrs Cherisse Campbell, was also hired this summer to work on the development of the FrAMED program for high school student. Mrs Campbell learnt about the different failure modes in materials and prepared lectures for that program.

Outreach Activities:

The PI hosted two high school teachers, Mrs Martha Magana and Cherisse Campbell in summer 2010 to work on the curriculum development of FrAMED: Failure Analysis for Materials Engineering Detectives. Mrs Campbell was supported with a RET from this EAGER grant. During this seven-week period, Mrs Magana and Campbell learnt about the different failure modes of materials and the experimental techniques across several length scales to measure these properties. Then they prepared short lectures for the FrAMED program, as well as team projects for the high school students who will participate in that program.

Journal Publications

B. Pant, S. Choi, E.K. Baumert, B.L. Allen, S. Graham, K. Gall, O.N. Pierron, "MEMS-Based Nanomechanics: Influence of MEMS design on test temperature", Experimental Mechanics, p. , vol. , (2011). Published, 10.1007/s11340-011-9526-8

Pant, B.;Allen, B. L.;Zhu, T.;Gall, K.;Pierron, O. N.;, "A versatile microelectromechanical system for nanomechanical testing", Applied Physics Letters, p. 053506, vol. 98, (2011). Published,

Books or Other One-time Publications

Web/Internet Site

Other Specific Products

Contributions

Contributions within Discipline:

This project will contribute to the nanomechanics community through the development of a versatile MEMS device to measure the mechanical properties of nanomaterials. The MEMS device can be used for both ex-situ testing and in-situ testing. Ex-situ testing may be useful for measuring the environmental effects on the mechanical properties. In-situ testing, especially quantitative in-situ TEM testing may be very useful to observe nanometer-scale deformation mechanisms under the TEM while measuring the applied stress and strain. The versatility of the MEMS device is directly attributed to the way strains are measured, which does not require high magnification SEM or TEM images.

Contributions to Other Disciplines:

Contributions to Human Resource Development:

Contributions to Resources for Research and Education:

Contributions Beyond Science and Engineering:

Conference Proceedings

Categories for which nothing is reported:

Organizational Partners
Any Book
Any Web/Internet Site
Any Product
Contributions: To Any Other Disciplines
Contributions: To Any Human Resource Development
Contributions: To Any Resources for Research and Education
Contributions: To Any Beyond Science and Engineering
Any Conference

Demonstration of a fatigue test of a Ni nanobeam

The following illustrates the capability of our MEMS-based nanotensile testing setup to study the fatigue properties of nanomaterials. Specifically, the results of a fatigue test performed on a nanocrystalline Ni nanobeam are reported.

The specimen (length: 14.5μm, width: 690nm, thickness: ~250nm) was attached onto the MEMS device using Pt clamps (this procedure occurs in a dual beam microscope equipped with a nanomanipulator) [1]. The test was performed in laboratory air (*ex-situ* test). The voltage, V_{in}, applied to the thermal actuator (see Fig.1) was varied in a cyclic manner between 0 and 4V, resulting in cyclic loading of the nanobeam under near tension-tension conditions ($R_{\varepsilon}=\varepsilon_{min}/\varepsilon_{max}$~0). The stress-strain curve was calculated for each cycle until fatigue failure. The specimen was cycled at 0.006 Hz during the first 2500 cycles, then at a frequency of 0.25Hz between 2500 and 40000 cycles. The specimen failed at around N_f=40000 cycles.

During the test, the change in capacitance between the two capacitive sensors (CS) located on each side of the specimen, ΔCS_1-ΔCS_2, is measured as a function of V_{in}, using a MS3110 capacitive readout sensor (see Fig. 2). In addition, the change in capacitance for CS1 is measured as a function of V_{in} prior to placing the nanobeam onto the MEMS device (see Fig. 2). These two sets of data can be used to calculate the stress-strain curves [2]. The noise in the measure signal is less than 0.3 fF, resulting in nm resolution in strain and ~10-MPa resolution in stress. The strain is calculated assuming that the gap increase between CS1 and CS2 is equal to the specimen deformation (i.e., infinitely stiff Pt clamps).

Fig. 3 shows 5 stress-strain curves calculated over the first 2500 cycles. The stress-strain curve for the first cycle is linear up to 5% nominal strain, with an apparent elastic modulus of 40 GPa. This value is much lower than that previously calculated in similar nanocrystalline Ni nanobeams (~210 GPa) [2], the expected value for polycrystalline Ni. It is likely that, for this particular test, the Pt clamps were not strong enough, and therefore were deforming (elastically) along with the nanobeam. Fig. 4 shows the difference in clamps between the specimen used for this fatigue test, and a previous specimen for which the calculated elastic modulus was 208 GPa [2]. The calculated gap increase between CS1 and CS2 at V_{in} = 4V is ~700nm. To obtain an elastic modulus of~200GPa, the actual specimen elongation should be ~140nm, requiring ~280nm of deformation (possibly shear deformation) for each Pt clamp. This appears to be a likely scenario given the shape of the Pt clamps in Fig. 4(a). The manipulation and clamping of the nanospecimen is a delicate task, requiring extended training of the user. The specimen shown in Fig 4(a) was from a fairly novice user, while the specimen shown in Fig. 4(b) was from a proficient one. It is therefore expected that such issues with Pt clamping can be resolved with further training of the FIB user.

Based on the above paragraph, it is likely that the specimen be initially loaded mainly under elastic loading (based on the nearly linear slope up to 2 GPa, and the little difference in stress-strain curves between loading and unloading) with an actual strain of ~1% to obtain a more reasonable value of the elastic modulus. This behavior is consistent with ultra-strong nanocrystalline materials [2]. Fig. 3 shows the stress-strain curves after 50, 700, 1400 and 2500 cycles. The plastic deformation for each cycle is small (see loading versus unloading for cycle 1), implying that there is no large scale plasticity occurring within one cycle. However, plastic deformation did accumulate over these first 2500 cycles, as evidenced by the increasing strain at which the applied force is zero (see Fig. 5). In addition, the decrease in the slope of the stress-strain curve for small strain values is also consistent with plastic strains: as the specimen is elongated due to plastic strains, it undergoes buckling near the end of the unloading portion of the cycle, resulting in a lower apparent elastic strain. This result is further confirmed with SEM images of the specimen taken after 2500 cycles (see Fig. 6). No significant further evolution in stress-strain curve was observed after 2500 cycles, until failure at 40,000 cycles, which is consistent with the plateau regime observed after 1500 cycles in Fig. 5. Fig. 7 shows the SEM images of the specimen after failure, along with a high magnification of the fracture surface. The fracture surface occurs away from the Pt clamps. In addition, the specimen overlaps by ~370nm at the location of fracture, confirming the accumulation of plastic strains throughout the fatigue test.

At 2500 cycles, the fatigue test was interrupted and the effect of frequency on the stress-strain curve was investigated. Fig. 8 shows that the stress-strain curves are nearly identical for cycle duration varying between 4s (f=0.25Hz) and 170s (f=0.006Hz). It will therefore be possible with this testing setup to study the effect of frequency on fatigue failure over this range of values.

Conclusions:

Our MEMS-based nanotensile testing setup can be used to measure the fatigue properties of nanocrystalline nanobeams (fatigue life, cyclic stress-strain curves, transient behavior). Fatigue tests can be performed for a wide range of maximum applied stresses and strains, frequencies (up to ~0.25-1Hz with the thermal-actuator-based MEMS devices), and environment. While the fatigue test presented in this report was performed in laboratory air, future tests can be performed in an environmental chamber to assess the influence of temperature and humidity on the fatigue properties. Last but not least, the same experimental setup can be used to perform quantitative in-situ TEM tests, using an electrical biasing TEM holder.

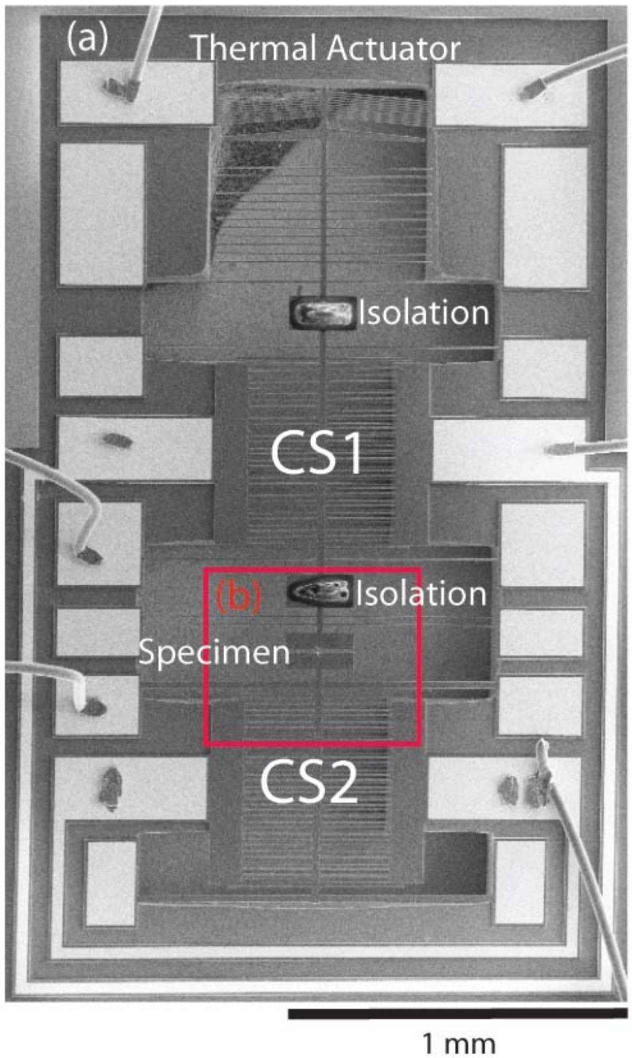

Fig. 1: MEMS device used for fatigue test. See [1] and [2] for details.

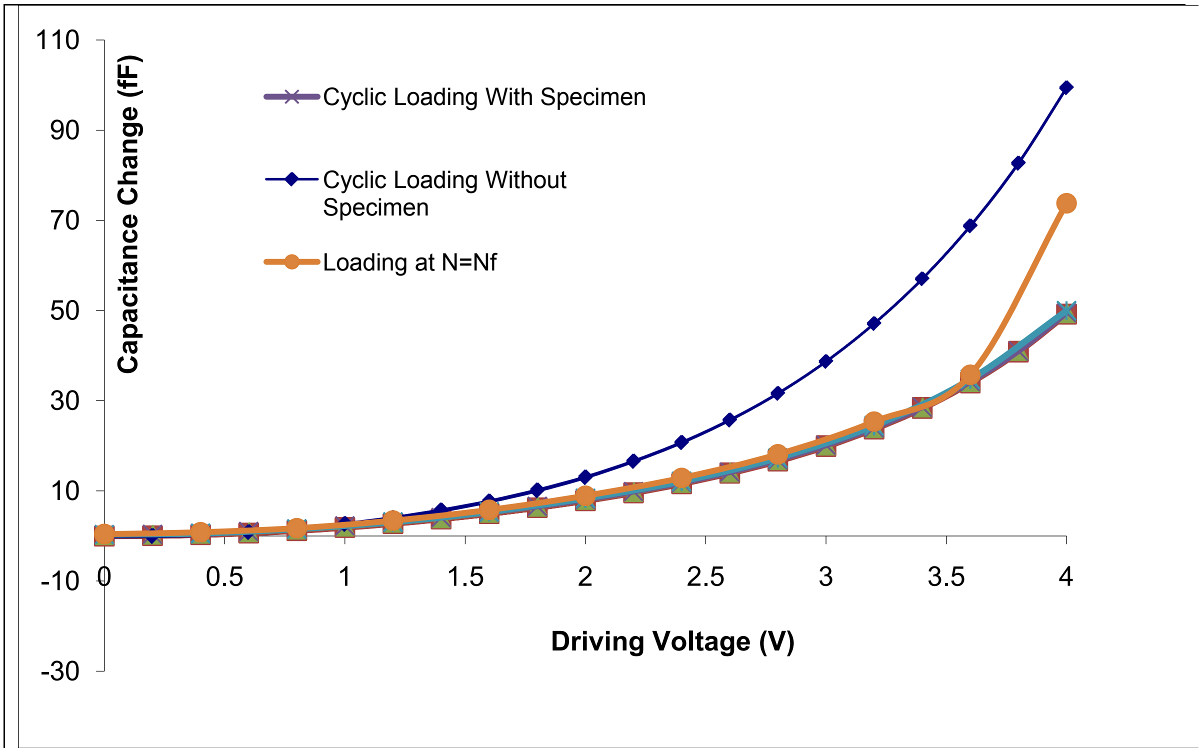

Fig. 2: Capacitance change, ΔCS_1-ΔCS_2, versus driving voltage (V_{in}). At N=N_f, failure occurred at 4V.

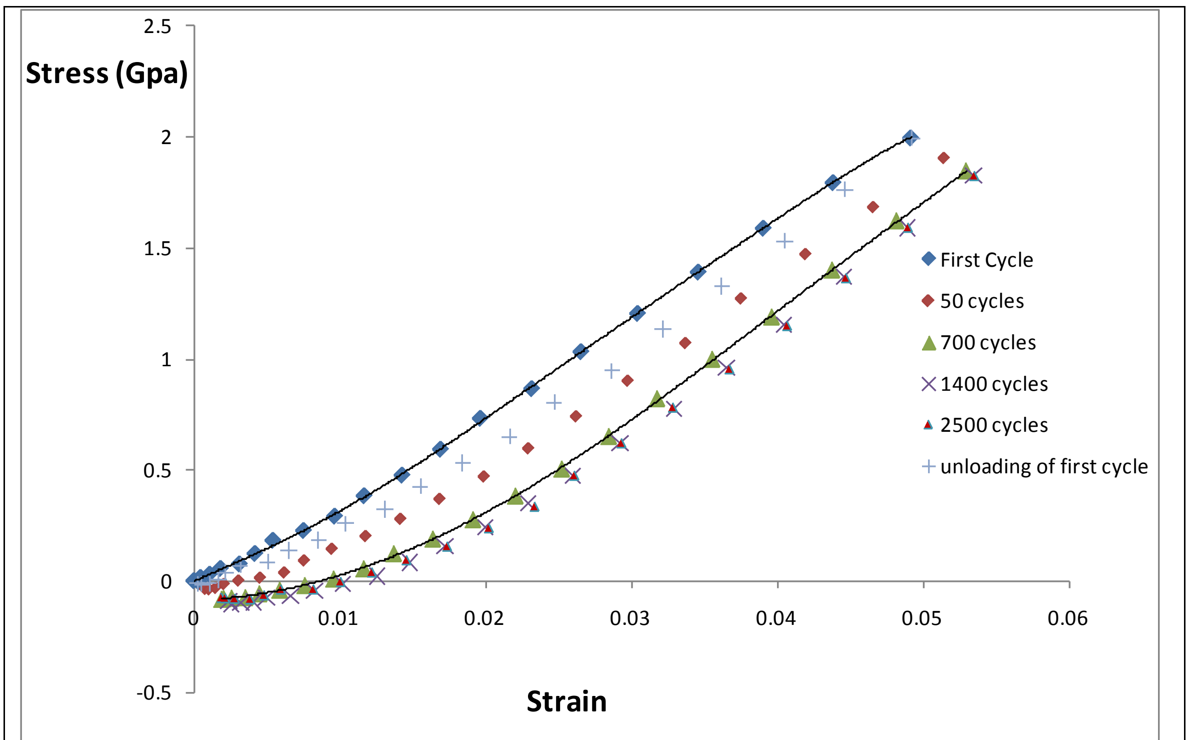

Fig. 3: Stress-strain curves after different number of cycles.

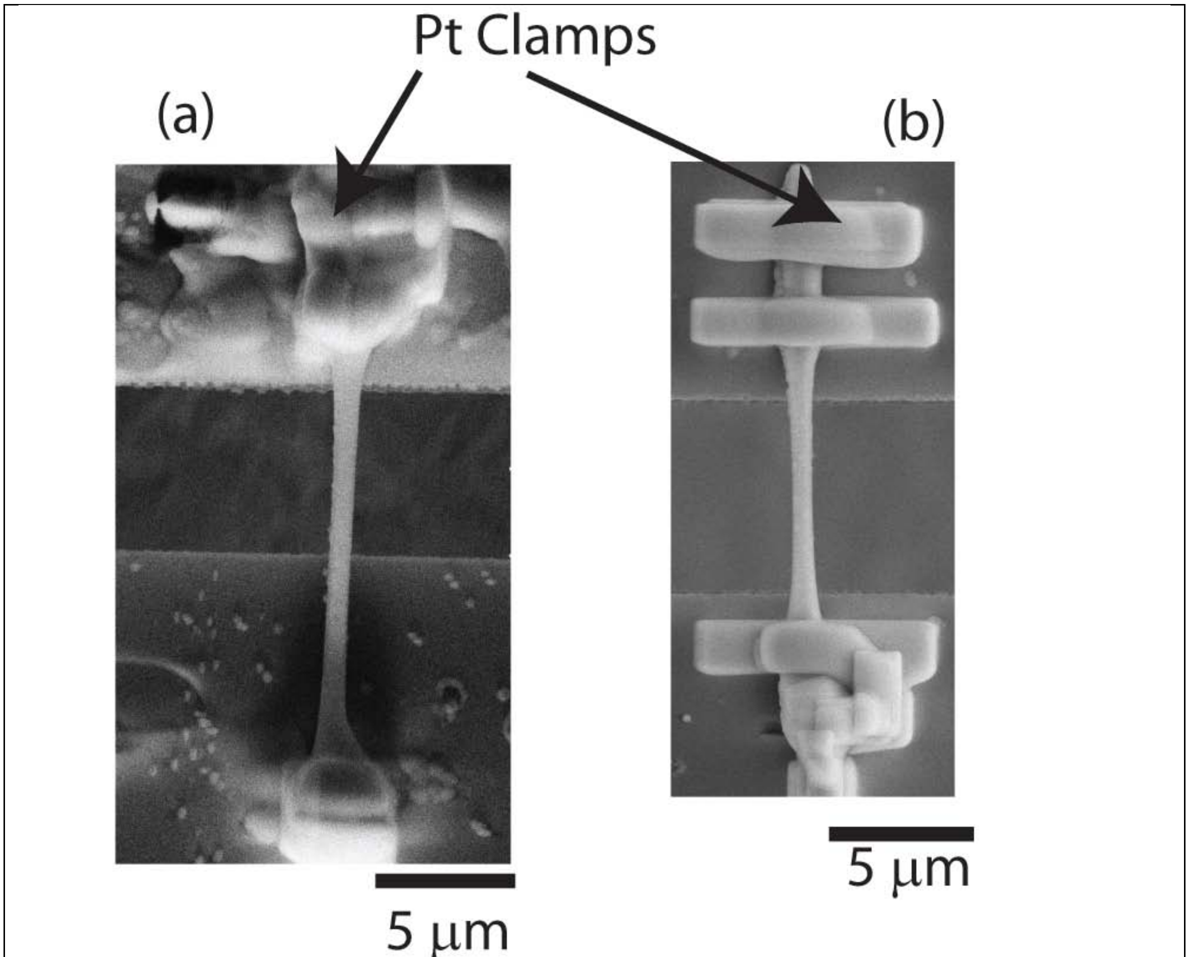

Fig. 4: (a): Pt clamps for this test. (b): Pt clamps for a previous test [1].

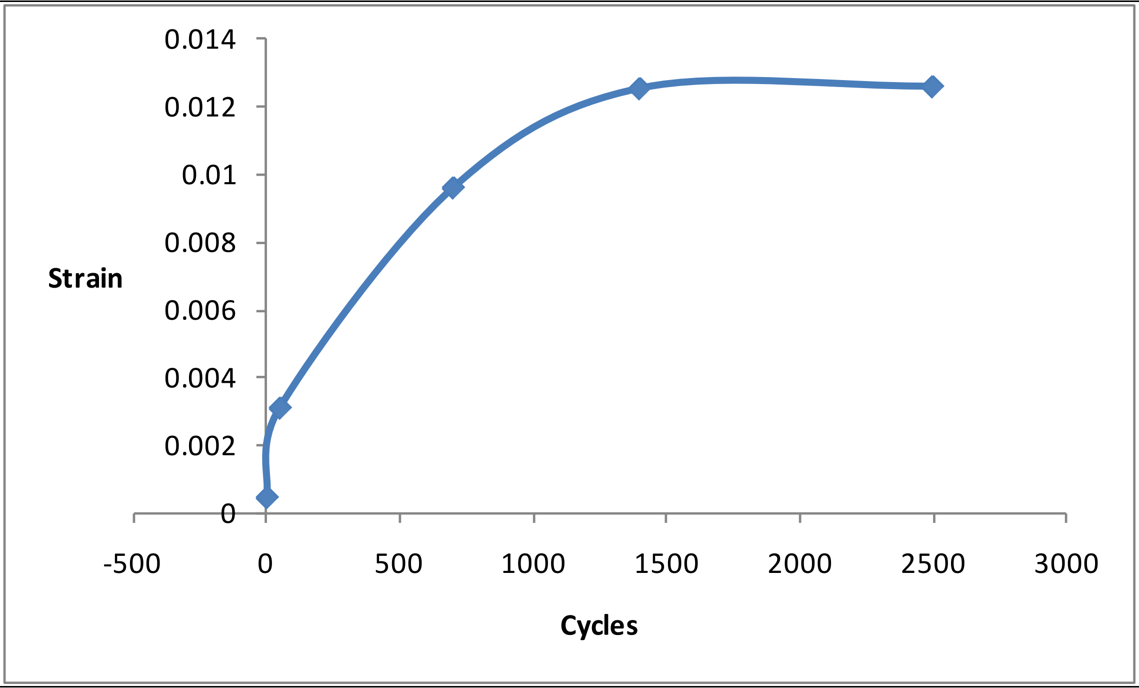

Fig. 5: Accumulated plastic strain as a function of cycles.

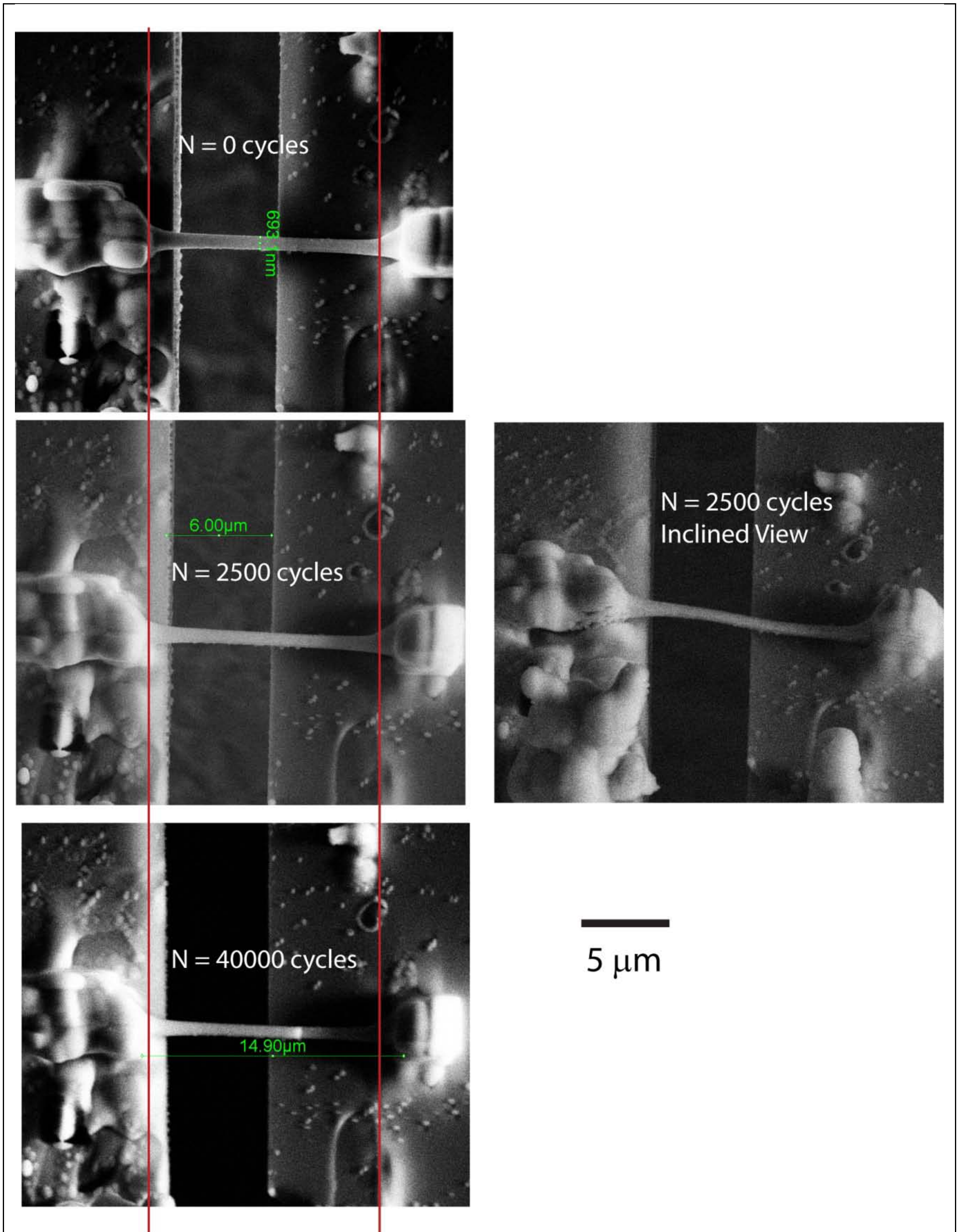

Fig. 6: SEM images of the specimen before test, after 2500 cycle (including inclined view to show buckling effect due to accumulated plastic deformation), and after failure.

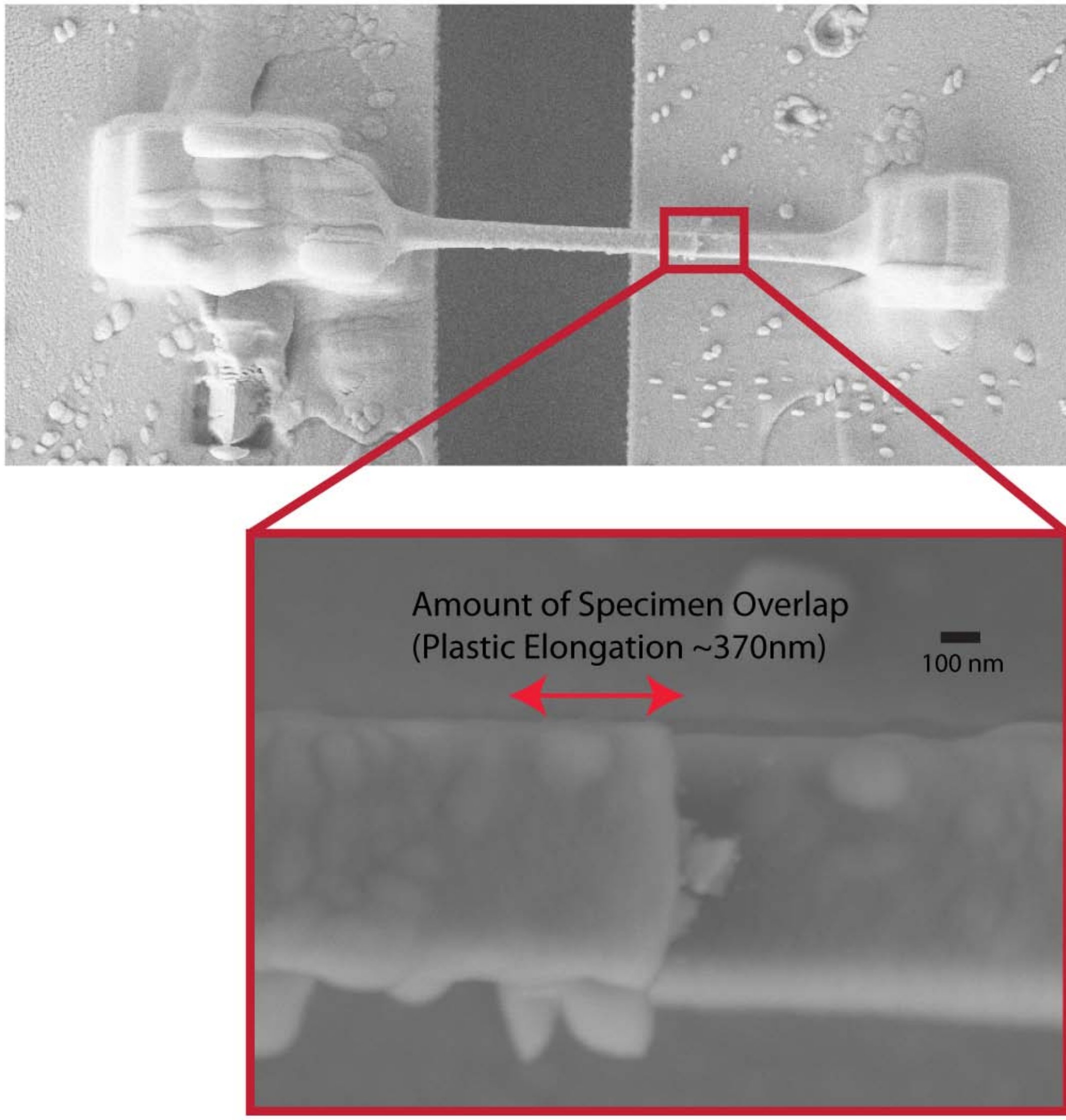
Amount of Specimen Overlap
(Plastic Elongation ~370nm)
100 nm

Fig. 7:

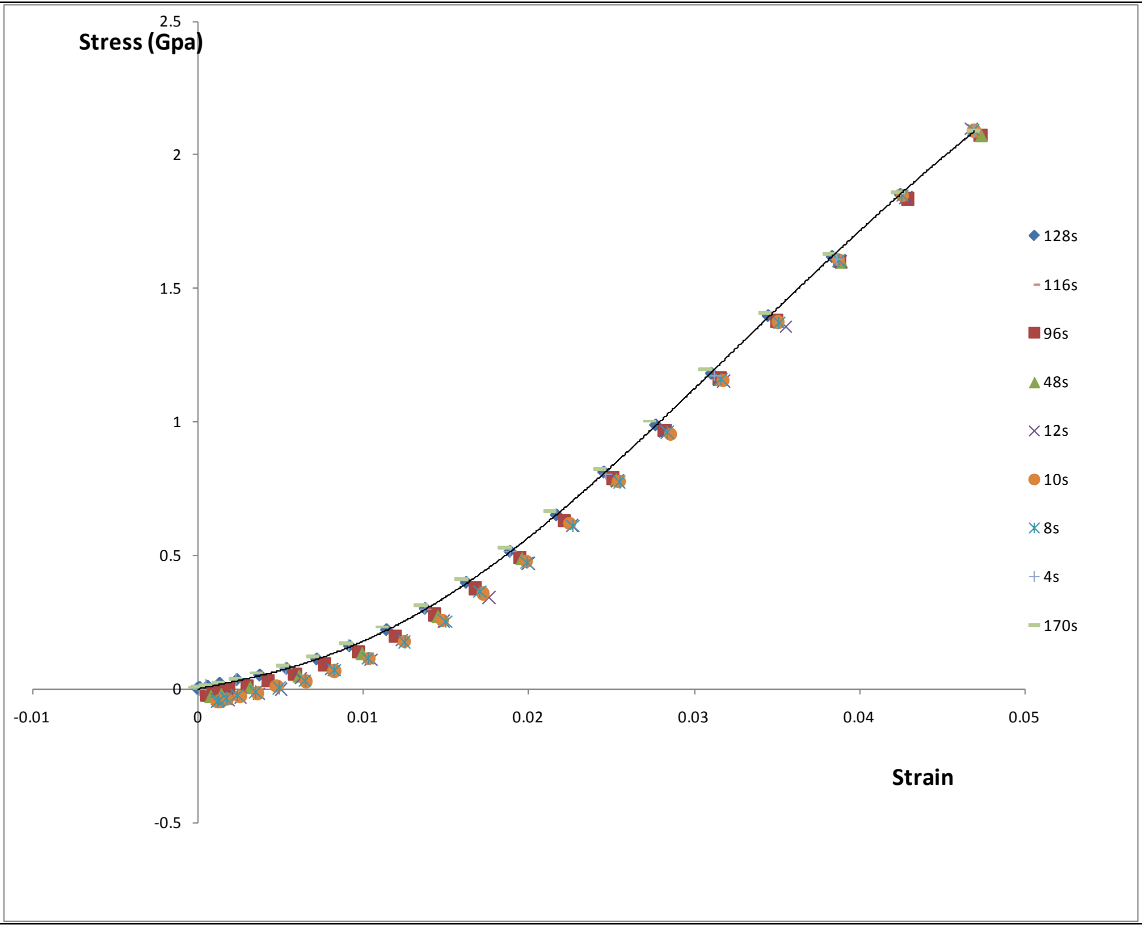

Fig. 8: Stress-strain curves for various cycle times, ranging from 4s (f=0.25Hz) to 170s (f=0.006Hz)

References

[1] B. Pant, S. Choi, E. Baumert, B. Allen, S. Graham, K. Gall, and O. Pierron, MEMS-Based Nanomechanics: Influence of MEMS Design on Test Temperature, Exper. Mech. (2011) 1-11.

[2] B. Pant, B.L. Allen, T. Zhu, K. Gall, and O.N. Pierron, A versatile microelectromechanical system for nanomechanical testing, Appl. Phys. Let. 98 (5) (2011) 053506.