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Anelasticity in Freestanding Aluminum Thin Films

by

Paul A. El-Deiry

A Thesis Presented to the Graduate and Research Committee of Lehigh University in Candidacy for the
Degree of Doctor of Philosophy

in

Materials Science and Engineering

Lehigh University

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For my family

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Abstract

Anelastic deformation is present in all metals, but it is generally negligible in bulk form and at low homologous temperatures. In thin films, anelastic deformation contributes a great deal to the overall mechanical properties. Anelastic deformation is any portion of the total deformation of a body that occurs as a function of time when load is applied and which disappears completely after a period of time when the load is removed.¹ Two major anelastic mechanisms proposed for pure, metal thin films are reversible dislocation glide and grain boundary sliding. Dislocation glide is expected at low homologous temperatures while grain boundary sliding is expected at high homologous temperatures. With increasing grain boundary density (i.e. smaller grain sizes), grain boundary sliding is expected to dominate, and with larger grain sizes dislocation glide is expected to dominate.

To investigate this phenomenon in thin metal films, a custom-built microtensile system was designed and fabricated. Uniaxial tensile tests were performed on four different thicknesses (1.00 μm , 0.75 μm , 0.50 μm , and 0.25 μm) of freestanding Al thin films. Strain rate sensitivity and stress relaxation experiments were performed to investigate anelasticity. Film quality turned out to be a major factor in all of the mechanical measurements made, including the ability to observe anelasticity.

Uniaxial tensile experiments performed on Al and Cu micro-wires lead to the supposition that a dislocation gliding mechanism is responsible for the observed strain rate sensitivity. Stress relaxation experiments were performed at four different temperatures (38°C, 50°C, 60°C, and 70°C) on freestanding Al thin films. The results indicate that the freestanding films can be modeled by a linear anelastic solid. In addition, a time constant for fast relaxation (< 0.5

seconds) was determined at each temperature. From these values, an activation energy was calculated. The small value of the activation energy (approximately 14 kJ/mol) suggests that only one mechanism is operating over the time scale of 0.5 seconds: dislocation glide.

1 Introduction

Thin films of metals, semiconductors, oxides, and polymers are contained in all modern microelectronics devices: microchips in cell phones, computers, and O₂ sensors in cars; television and hand-held displays; fiber-optic switching systems; communication and surveillance satellites; medical equipment, and many more devices not mentioned. Thin films range in thickness from tens of nanometers to tens of micrometers, or about the diameter of a human hair. In order for the devices to reliably operate, the thin films must perform to the established design criteria throughout the lifetime of the device.

In 1967 with the design and fabrication of the resonant gate structure by Nathanson, Newell, Wickstrom, and Davis Jr. at Westinghouse Research Laboratories located in Pittsburgh, PA, MicroElectroMechanical Systems (MEMS) became a reality.² MEMS rely on the movement and interactions of extremely small three dimensional structures primarily constructed of deposited thin films. Common materials include silicon (Si), aluminum (Al), gold (Au), and copper (Cu). Reasons for using these materials are their great availability, ease of fabrication, and unique electronic and mechanical properties.

Comparable to their bulk counterparts, thin metal films (i.e. thin films) may be deformed elastically or plastically. If the strain is purely elastic, the thin film may behave as desired for billions of cycles. On the other hand, if the thin film undergoes plastic deformation in the first cycle of one hundred, then in the following ninety-nine cycles the thin film may not operate as desired. However, even if the strain is less than that required for the onset of plastic deformation, the metal may show signs of “anelasticity,” and the remaining cycles may not behave like the first. Anelastic deformation is any portion of the total deformation of a body that

occurs as a function of time when load is applied and which disappears completely after a period of time when the load is removed. In previous research, thin films have been shown to “relax” (i.e. show an increase in the amount of strain for the same applied stress, or a decrease in stress for the same applied strain) over time, conceivably to a sufficient degree to exceed the operating tolerances and cause the device to fail.³⁻⁷ Indications are that a large fraction of this relaxation is anelastic. If there is not enough “rest” time between cycles the repeatability and precision of the device will suffer. Therefore, research that examines the sources of anelastic behavior, and thereby suggests methods for modeling or reducing the effect, is imperative. This is the motivation for the work that follows.

1.1 *A brief history of anelasticity*

1.1.1 Elasticity and plasticity

Nowick and Berry define ideal elastic behavior for bulk materials as being implicit in these three conditions: “(1) that the strain response to each level of applied stress (or vice versa) has a unique equilibrium value; (2) that the equilibrium response is achieved instantaneously; (3) that the response is linear (e.g., doubling the stress doubles the strain).”⁸ Elasticity is therefore exclusively different from plasticity and anelasticity in that the equilibrium response between stress and strain is instantaneous and reversible. Plasticity is irreversible deformation sustained after removing the applied stress.

1.1.2 Anelasticity and viscoelasticity

Anelasticity deals with materials (mainly metals) that have an ordered and repeating crystallographic atomic structure. For relaxation to occur, atoms move back and forth along

well-defined paths by jumping or sliding, but they are not really “flowing.” On the other hand, viscoelasticity most often describes polymeric materials by combining both the mechanical properties of liquids (viscosity) and linear solids (elasticity). Also, most polymeric materials do not have an ordered crystallographic atomic structure, but instead are comprised of a myriad of tangled organic molecular chains. When a force is applied to the polymer, the viscous flow is introduced by an untangling of the chain and even possible reorientation of the molecules. Sometimes, the chains may be locked at pinning points causing them to untangle a specified amount and then return to their original configuration. It is this untangling and then returning process in polymers which is similar to the sliding and jumping of atoms in metals. Therefore, the stress-strain behavior of a viscoelastic polymer and an anelastic metal are similar. Many time dependent models describing viscoelasticity work very well for modeling the anelastic behavior of thin films.

1.1.3 The standard anelastic model

The standard anelastic model is shown in Figure 1-1. A linear spring and a linear viscous damper (i.e. dashpot) in parallel provide the time-dependent recovery; the arrow depicts the applied force on the system. A dashpot contains a viscous medium and has the property that its rate of displacement is proportional to the force across it, while a spring's displacement, not its rate of displacement, depends on the force across it. For the system in Figure 1-1, only one time constant for the recovery process can be derived because there is only one dashpot. Many real materials, however, exhibit at least two time constants. Therefore, two Voigt units, where the spring and dashpot are parallel to one another, in series with each other (Figure 1-2) would provide two time constants as long as the rate of displacement for each dashpot is distinct. This

simple anelastic model places three restrictions on the stress-strain relationship: 1) stress and strain must be linear to one another, 2) there are unique time constants associated with the relaxation, and 3) a unique equilibrium value between stress and strain must exist. The first, *linearity*, is exhibited if the stress doubles as the strain doubles (or vice versa). The second, a *unique time constant for relaxation*, means that for a specific physical event taking place in the material a certain amount of time lapses as the resulting stress returns to its original value. The third, a *unique equilibrium*, is when allowed sufficient time the system responds such that for every value of stress a unique equilibrium value of strain (or vice versa) exists to which the system will eventually settle.⁹ In other words, when enough time has passed the system will undergo complete recoverability and behave as if there was no prior strain or stress history.

Consider the model in Figure 1-1: when the initial force is applied, spring E_2 responds instantaneously. Spring E_1 , however, because it is in parallel with the dashpot, relies on the dashpot for response. The dashpot is now controlling the response of spring E_1 . This time response, τ associated with the dashpot is defined as μ (a measure of viscosity) divided by

$$E_1, \text{ where } \tau = \mu / E_1.$$

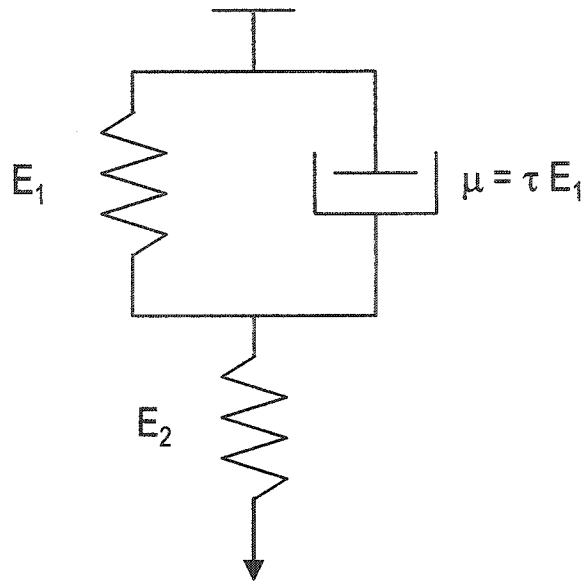


Figure 1-1: Mechanical analog for the standard anelastic solid containing one Voigt unit, which results in one time constant, τ .

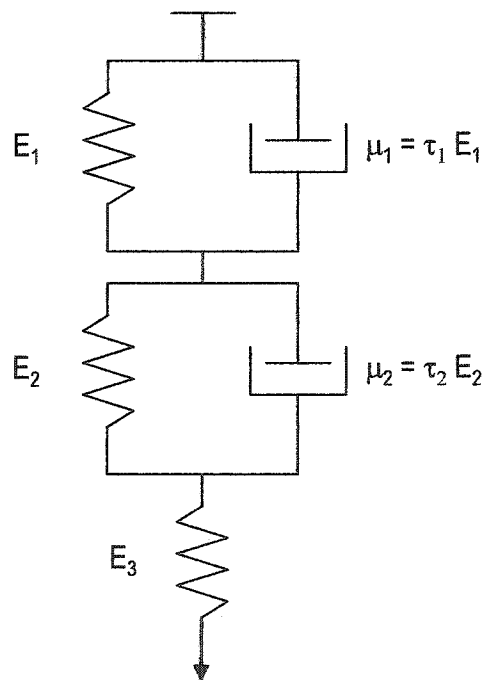


Figure 1-2: Mechanical analog for the standard anelastic solid containing two Voigt units, and therefore two time constants, τ_1 and τ_2 .

1.1.4 A constitutive equation for a 1-D response of a viscoelastic material

Consider the Maxwell model shown in Figure 1-3:

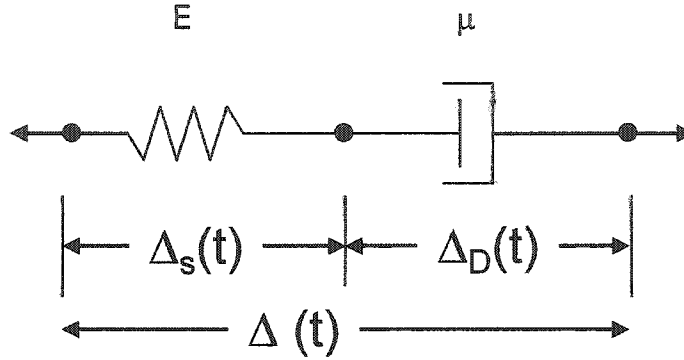


Figure 1-3: The Maxwell model for a 1-D viscoelastic material.

$$F(t) = F_s(t) = F_D(t)$$

Equation 1-1: Force balance for the 1-D Maxwell model.

In Equation 1-1, the force on the system, $F(t)$, as a function of time equals the force across the spring, $F_s(t)$, which equals the force across the dashpot, $F_D(t)$. The total displacement of the system, $\Delta(t)$, is equal to the displacement of the spring and the displacement of the dashpot (Equation 1-2).

$$\Delta(t) = \Delta_s(t) + \Delta_D(t)$$

Equation 1-2: The total displacement of the system.

The force across the spring (Equation 1-3) is equal to the spring constant, E , times the displacement; the displacement of the spring is instantaneous.

$$F_s(t) = E\Delta_s(t)$$

Equation 1-3: The force across the spring element.

The force across the dashpot (Equation 1-4) is the viscosity times the displacement, but the displacement of the dashpot is not instantaneous; it increases with increasing time.

$$F_D(t) = \mu \dot{\Delta}_D(t) = \mu \frac{d}{dt} \Delta_D(t)$$

Equation 1-4: The force across the dashpot element.

Because the displacement of the dashpot is not instantaneous, it is convenient to discuss the time derivative of Δ , $\dot{\Delta}$, rather than Δ .

$$\dot{\Delta}(t) = \dot{\Delta}_s(t) + \dot{\Delta}_D(t)$$

Equation 1-5: The response of the system.

Now, Equation 1-3 is differentiated with respect to time:

$$\dot{F}_s(t) = E\dot{\Delta}_s(t)$$

Equation 1-6: The force on the spring as a function of time.

Substitute Equation 1-4 and Equation 1-6 into Equation 1-5 to get:

$$\dot{\Delta} = \frac{\frac{d}{dt} F_s(t)}{E} + \frac{F_D(t)}{\mu} = \frac{\dot{F}}{E} + \frac{F}{\mu} = \left(\frac{\dot{\sigma}}{E} + \frac{\sigma}{\mu} \right) A;$$

Equation 1-7: Constitutive equation describing the time response of a viscoelastic material.

where A is the area of the material under stress.¹⁰ This constitutive equation is the building block for stress relaxation at constant strain observed in anelastic and viscoelastic materials. Through a numerical calculation, this model can correctly predict qualitatively the type of behavior that is expected for the freestanding Al thin films (Figure 1-4). By varying the value of the viscosity (i.e. 1000, 100, 10, and 1), the total range of stress relaxation behavior is covered and the modulus deficit (i.e. effective modulus) is observed. The effective modulus upon loading and unloading was measured between 0.1% and 0.2% strain, and 0.2% and 0.3% strain, respectively. A trapezoidal waveform run to a strain amplitude of 0.5% over a total time of 3 seconds was the control parameter in this calculation.

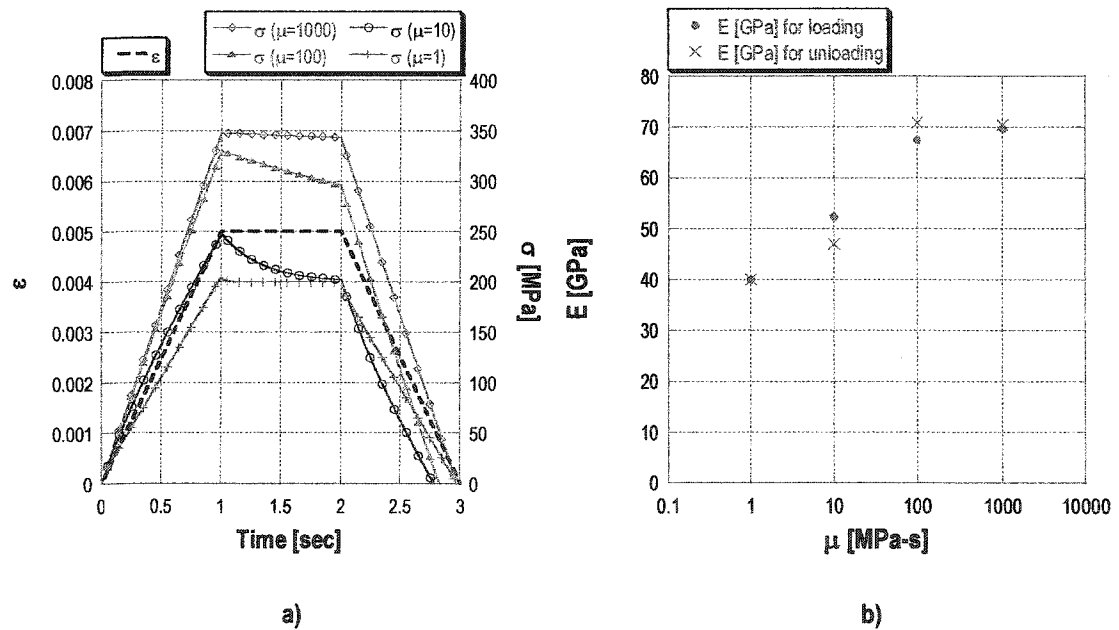


Figure 1-4: a) A plot of strain and stress vs. time displaying the stress relaxation behavior expected based on a material of which 57% (40/70 GPa) is elastic and 43% (30/70 GPa) is 1-D viscoelastic, and b) a plot of effective E vs. μ showing the expected modulus deficit; this can be related to effective E vs. $\dot{\epsilon}$.

1.1.5 Internal friction experiments

Clarence Zener, who performed internal friction experiments in the 1940's, is credited for establishing the field of anelasticity. However, Zener stated that anelastic behavior was first observed (or published) in 1835 by Weber; specimens were subjected to a constant load for a set amount of time followed by a quick removal of that load.^{11,12} Weber observed that most of the strain was returned almost immediately, but a residual strain remained which disappeared only after an extended period of time. Weber named this phenomenon "Elastische Nackwirkung", or elastic after-effect (Figure 1-5). Elastic after-effect experiments can provide enough time for the specimen to reach equilibrium following an initial applied force.

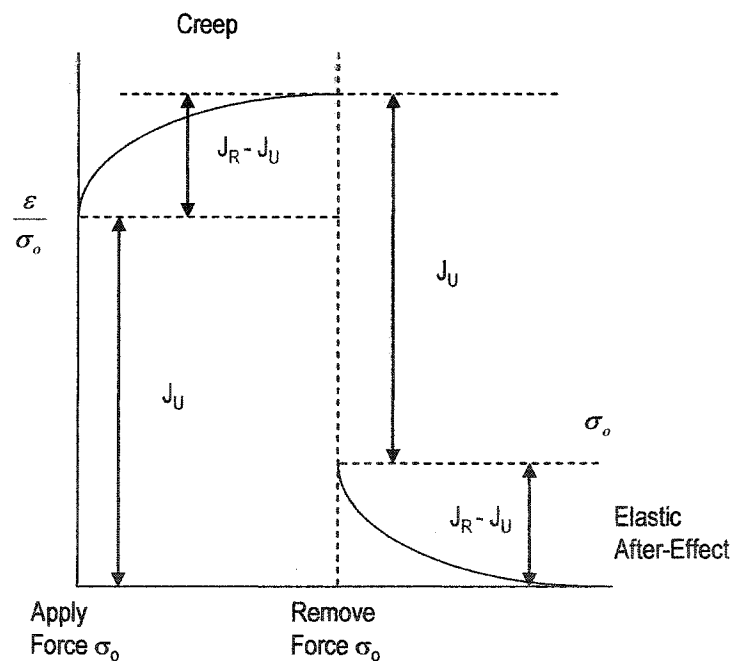


Figure 1-5: A plot of compliance vs. time showing the elastic after-effect observed by Weber in 1835.

When an instantaneous stress of σ_o is applied at t_o , the sample responds with the unrelaxed modulus, $1/J_u$, because no time is allowed for the sample to relax. When the sample

is held at the constant stress, the modulus relaxes from $1/J_u$ to $1/J_R$, where $1/J_R$ is the relaxed modulus. In Figure 1-5 this modulus relaxation is reflected by an increasing sample strain.

Historically, elastic after-effect experiments have been useful for measuring relaxation times on the order of greater than one second and usually for a total duration of many hours and sometimes days.

To measure relaxation times on the order of less than a second, internal friction experiments were performed. In 1947, Kê performed internal friction experiments on both polycrystalline and single crystal aluminum. He found that anelastic mechanisms present in the polycrystalline aluminum were almost completely absent in single crystal aluminum suggesting that the observed behavior was a result of the grain boundaries. Furthermore, he calculated an activation energy of 34,500 cal/mol (or approximately 144 kJ/mol, or 1.4 eV) associated with "viscous slip along the grain boundaries."¹³ This behavior of viscous grain boundaries is present at either low deformation rates and/or high temperatures.

1.2 Anelastic mechanisms

Common anelastic mechanisms in bulk materials include interstitial diffusion (i.e. Snoek effect), dislocation glide, and grain boundary sliding (GBS). Diffusion-based mechanisms, such as the Snoek effect and GBS, are usually present in bulk materials with high solute concentrations. These diffusion-based mechanisms are anelastic because there is some finite time associated with the solute atoms moving back to their original locations in the lattice once the applied stress is removed. A dislocation glide mechanism is anelastic when a dislocation runs into a grain boundary. If the dislocation is not held secure at the grain boundary, once the applied stress is removed a finite time is needed for the dislocation to move back to its original

location. However, because the thin films in this study are pure, any mechanism based on solute concentration is not expected.

Therefore, two major anelastic mechanisms are suggested to be active in thin films: (1) dislocation glide on specific slip systems including their interactions with other dislocations, free surfaces, grain boundaries, etc., and (2) GBS which involves atomic diffusion processes along the grain boundaries. It is thought that as the grain size of a bulk material approaches nanocrystalline dimensions (i.e. an average grain size smaller than 100 nm), the dominating deformation behavior transitions from a dislocation mechanism to a GBS mechanism. The mechanism that dominates the entire relaxation process can depend upon average grain size, d ; films with larger grain sizes are expected to have dislocation glide dominate, while those with smaller grain sizes are expected to exhibit diffusion-controlled GBS processes. The manner in which the mechanisms operate is influenced by microstructural parameters (i.e. film thickness, grain size, grain geometry, film texture), material properties (i.e. atomic structure, absolute temperature), and whether the film is on a substrate or freestanding.

Studies carried out with film/substrate systems suggest the substrate, acting as both an inhibitor to dislocation glide and a nucleation site for dislocation creation, has a significant role in dislocation processes.^{14,15} In-situ transmission electron microscopy deformation of thin films in combination with computer simulations of dislocations gliding and dislocation-dislocation interactions are making clearer the dynamic behavior of dislocations.¹⁶⁻²⁵ Thin films on substrates at low temperatures generally support much higher stresses than their bulk counterparts and these stress levels are very sensitive to the film thickness and the presence of a hard passivation layer.²⁶⁻³⁴ The overall film strength varies inversely with the film thickness.³⁵

Both dislocation and GBS mechanisms are present in thin films on substrates. Freund and Nix

have formulated specific dislocation models dealing with the movement of single dislocations for face centered cubic (FCC) metallic thin film/substrate systems.^{14;36} At the film/substrate interface, dislocations can nucleate and propagate into the film or, conversely dislocations in the film can meet at the interface and remain sessile. In addition, dislocation mechanisms have been shown to operate differently in Cu (a FCC metal not shown to oxidize readily) and Al (a FCC metal on which it is almost impossible to avoid oxidation). A film with this passivation layer present may behave similarly to a film on a substrate.

Previous research has revealed that relaxation occurring in thin films may either be irreversible or reversible and time-dependent; this reversibility is the foundation for anelasticity.³⁷⁻⁴¹ When the applied stress is removed and the deformation completely recovers, the relaxation process is anelastic. The recovery may occur quickly or it may take many hours, but as long as the recovery is complete, the process is anelastic. For instance, if a thin film is tested a second time after allowing "sufficient" rest time for recovery following the first relaxation, it will behave as if unaware of the first relaxation.

1.3 *Evidence of anelasticity in thin films*

1.3.1 Internal friction experiments

Internal friction experiments have also been performed on thin films. Relaxation processes occurring over extremely short time intervals (typically 10^{-6} to 10^{-9} seconds) can be measured. This time scale is solely dependent on the frequency used; typically on the order of kHz to MHz. Also, internal friction measurements are dependent on temperature. At one temperature, the thin film has one resonance; when the temperature is increased or decreased, the resonance

changes. However, it should be noted that internal friction experiments have been performed at longer time intervals of 1 to 10 seconds, but not on thin films. Performing internal friction experiments at these time scales on thin films are difficult. By observing the change in resonance with temperature, activation energies for a specific relaxation mechanism can be calculated. From measurements performed on Al/substrate composites, an activation energy of approximately 0.6 eV was calculated, which was associated with a GBS relaxation mechanism.⁴² Bohn et al. suggest that a higher number of impurities are present in bulk Al resulting in a retardation of grain boundary migration, which leads to the higher activation energy of 1.4 eV.

For freestanding Al thin films, the relaxation mechanism may be different. It has proven difficult to understand the role of the substrate on the relaxation mechanism. With freestanding Al thin films, the substrate effect may still be present because of the native oxide. Also, the activation energy for freestanding Al thin films may vary from that of Al films on substrates. If this is true, the role of the free surface may be influencing the specific anelastic mechanism, whether it is diffusion-based or dislocation glide. Microtensile experiments are capable of measuring freestanding thin films.

1.3.2 Microtensile experiments

When films are freestanding, the role of a thick substrate is removed; however, a thin layer of Al₂O₃ is present on both Al surfaces (at a thickness of a few nanometers) and may act similarly to a substrate but will almost certainly have a lesser impact in impeding dislocation glide.⁴³ In freestanding thin films at ambient temperatures and pressures, it is generally noted that the overall yield strength and Young's modulus (i.e. effective modulus) compared to films

on substrates and their bulk counterparts is greatly reduced.^{37,44-61} Reasons for a reduced modulus could be the result of other effects, such as the presence of voids or microcracks, more compliant grain boundaries, or a highly topographic (i.e. “wrinkled”) film.^{48,62} If the film is not flat when a stress is applied, certain regions of the film may start yielding while others may not even be taut. Furthermore, it has been shown that grains exhibit a sliding behavior as seen by a dynamic creep response (resulting in a lower effective modulus) in the stress-strain plot. However, in contrast to documented GBS mechanisms, Haque et al. report an unexpected low ductility and a linear elastic behavior up to a brittle fracture in nanocrystalline (average grain size = 20nm) Al thin films.⁶³ This nanocrystalline grain size may have caused the Al to behave in a brittle manner by impeding dislocation glide. In general, the dominant stress relaxation mechanism in freestanding thin films is expected to be dependent on the number of grain boundaries. For films having only a few large grains (i.e. low grain boundary density but long grain boundaries), dislocation mechanisms are expected to dominate. On the other hand, for films with small grains and a high grain boundary density, GBS mechanisms are expected to dominate.

1.4 Experimental motivation and plans

Traditional microtensile experiments are carried out at a single temperature and can successfully be performed on freestanding thin films at time scales between 10 seconds and 30 minutes. Microtensile experiments mainly concentrate on strain rate sensitivity and stress relaxation behavior in freestanding thin films. Strain rates typically range from 10^{-6} to 10^{-4} s⁻¹ while stress relaxations are observed anywhere from 10 seconds to 10 minutes after applying the initial stress. Strain rate sensitivity experiments are intended to show a dependence of

effective modulus on strain rate, where high strain rates are expected to result in an increased effective modulus. Direct experimental measurements of anelasticity can be perceived using strain rate sensitivity experiments and stress relaxation experiments.

Al ($T_m = 933.15$ K) has a FCC atomic structure. Because the majority of mechanical testing will be performed at ambient temperature, the ratio of $\frac{T}{T_m}$ is important (i.e. ambient temperature ≈ 298.15 K; therefore, $\frac{T}{T_m} = 0.32$ for Al). Dislocations are mobile on specific slip systems (i.e. a closed packed atomic plane in conjunction with a close packed direction in that plane). These atomic planes and directions have the highest atomic planar density and the highest atomic linear density, respectively. In FCC materials, the close-packed planes are $\langle 111 \rangle$ and the close-packed directions are $[110]$ (i.e. usually written as $\langle 111 \rangle [110]$). This $\langle 111 \rangle [110]$ combination is the FCC slip system.

1.4.1 Strain rate sensitivity experiments

A strain rate sensitivity experiment is typically used for measuring the relaxation taking place while the specimen is being subjected to an applied stress. At low strain rates, the value of the effective modulus is expected to approach the value of the relaxed modulus; in other words, the sample is given sufficient time to relax. On the other hand, a high strain rate would not give the sample as much time to relax thereby resulting in a higher effective modulus. Consequently, for different strain rates approaching the same strain amplitude, the peak stress may not be the same for both rates. The lower strain rate will result in a lower peak stress for the same strain amplitude because of the relaxation taking place during straining. This type of

experiment is called a strain rate sensitivity experiment where an exponent (i.e. the strain rate sensitivity exponent) can be calculated.

The strain rate sensitivity equation is $\sigma = K\dot{\epsilon}^m$; where m is the strain rate sensitivity exponent. Taking the logarithm of both sides of the equation gives $\log(\sigma) = \log(K) + m\log(\dot{\epsilon})$. When plotting $\log(\sigma)$ vs. $\log(\dot{\epsilon})$, the slope of the line is the strain rate sensitivity exponent. Because $E = \sigma/\epsilon$, then the $\log(\sigma) = \log(E) = \log\left(\frac{K}{\epsilon}\right) + m\log(\dot{\epsilon})$ by association.⁶⁴ Therefore, the experimentalist can plot $\log(E)$ vs. $\log(\dot{\epsilon})$ instead, where E is the effective modulus measured from the stress-strain plots.

1.4.2 Stress relaxation experiments

A similar measurement technique to the elastic after-effect experiment is the stress relaxation experiment. A sample is strained to a specific amplitude where the strain is then held constant; during the hold time at constant strain, the stress is observed to relax. The microtensile experiments performed in this research are unique because they use the “elastic after-effect” method, but are capable of measuring relaxation times less than one second.

To measure the time constants for the relaxation mechanism during the hold periods at maximum strain, a fit using a double exponential decay was performed (Equation 1-8) where σ_{∞} is the stress that the relaxation eventually reaches if allowed infinite time, σ_1 is the amount of stress relaxation taking place with the first time constant, τ_1 , and σ_2 is the amount of stress relaxation taking place with the second time constant, τ_2 . These terms defined in Equation 1-8

are displayed in Figure 1-6 which shows a schematic of the stress vs. time plot typical of a microtensile stress relaxation experiment performed on freestanding thin films.

$$\sigma(t) = \sigma_{\infty} + \Delta\sigma_1 * \left(\exp\left(\frac{-t}{\tau_1}\right) \right) + \Delta\sigma_2 * \left(\exp\left(\frac{-t}{\tau_2}\right) \right)$$

Equation 1-8: A double exponential decay used to model the observed stress relaxation behavior in freestanding Al thin films.

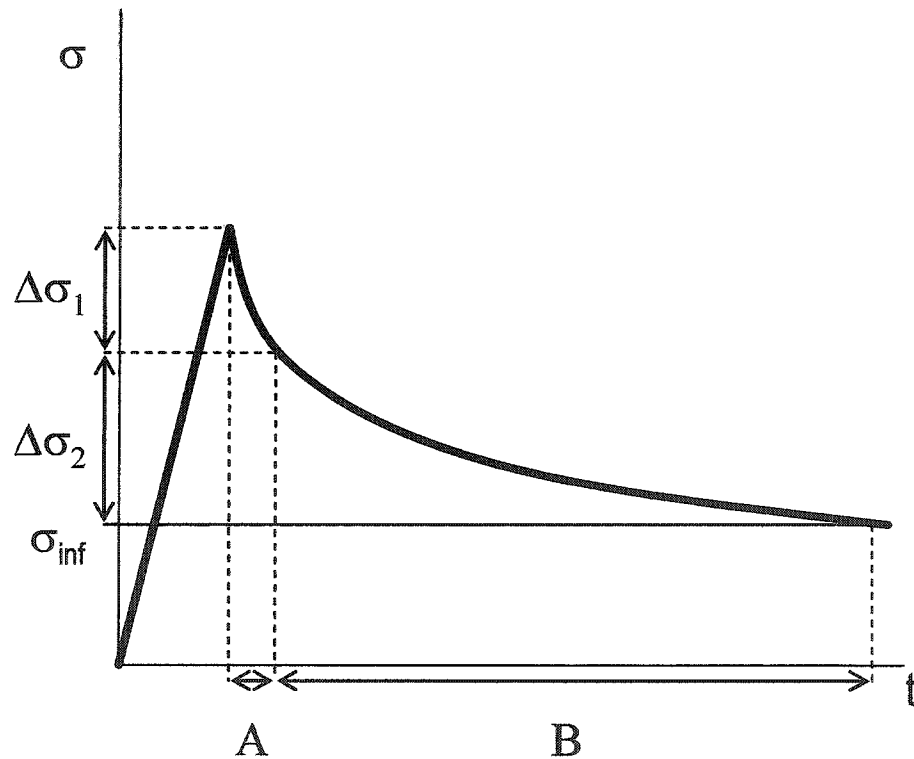


Figure 1-6: A schematic of the stress vs. time plot for a stress relaxation process observed during a microtensile experiment on a freestanding Al thin film; in time interval A, τ_1 , controls the stress relaxation, and in time interval B, τ_2 , controls the stress relaxation.

This research intends to answer the following question: Is this observed relaxation caused by a GBS or a dislocation glide mechanism? By measuring an activation energy, this question

may be answered. Creep experiments have been performed on single crystal aluminum thereby eliminating the effect of grain boundaries. Activation energies have been found for a dislocation climb mechanism (149,000 J/mol), a dislocation cross-slip mechanism (117,000 J/mol), and a dislocation glide mechanism (14,000 J/mol).⁶⁵ By comparing experimentally determined activation energies derived from stress relaxation experiments to these from creep, a specific mechanism responsible for the observed relaxation may become clear.

This research concentrates on characterizing rapid anelastic mechanisms in freestanding Al thin films by performing strain rate sensitivity and stress relaxation experiments. A custom-built microtensile apparatus was used to perform displacement-controlled experiments over the following range of strain rates: $8.33 \times 10^{-5} \text{ s}^{-1}$ to $8.33 \times 10^{-1} \text{ s}^{-1}$. High strain rates were chosen to investigate quick relaxation behavior (transition times < 0.5 second) associated with a fast-acting microstructural mechanism. Until now, successful and understandable microtensile experiments have been performed at strain rates ranging from 10^{-6} to 10^{-3} s^{-1} , but no stress relaxation information is currently published for times less than 1 second. Additionally, numerical models are developed in order to better classify which microstructural mechanisms (dislocations or GBS) are the basis of the observed anelastic behavior. By performing stress relaxation experiments at four different temperatures, activation energies for a specific mechanism were also determined.

1.5 Summary

The objective of this research is to understand the dependence of anelastic mechanisms on film thickness, grain size, strain rate, and strain amplitude in freestanding Al thin films. As thickness increases, a transition of anelastic mechanisms, from primarily diffusion-controlled

GBS to primarily dislocation glide, is anticipated. The goal was to confirm or refute this anticipation with quantitative experiments and published models of relaxation effects. Furthermore, by experimentally measuring activation energies, the specific anelastic mechanism can be determined. This study is not intended to be comprehensive, but instead provides a step toward a deeper understanding of grain size and film thickness effects on the mechanical properties of thin films.

2 Fabrication and characterization of the Al microtensile beams

In this chapter, the fabrication techniques used for making the Al microtensile beams are reviewed. Correction factors needed for accurate calculations of the strain are discussed. The techniques used for microstructural and compositional characterization are briefly described, and the key findings are assessed. Finally, the specimen naming scheme used throughout the remainder of this document is explained.

2.1 Fabrication

The sample fabrication sequence (Figure 2-1) was designed collaboratively and has already been discussed in detail in the Ph.D. dissertation of Nick Barbosa III.⁶⁶ Therefore, the sequence will be summarized here. Pure Al films were thermally evaporated onto a LPCVD silicon-rich SiN_x layer grown on 3" (100) silicon wafers. After deposition, the microtensile beams were patterned using front-side and back-side lithographic techniques; the non-protected Al was removed with a solution of phosphoric, acetic, and nitric acids (commonly known as a PAN etch). The protected Al defined the microtensile beam. After a reactive ion etch (RIE) to remove the patterned back-side SiN_x layer, the silicon directly underneath the front-side patterned Al microtensile beam was then removed using an 85°C potassium hydroxide (KOH) wet-etch; the front-side SiN_x layer beneath the deposited Al layer provided an etch stop. Once the silicon was etched, the front-side SiN_x layer was removed with the same RIE procedure used on the back-side SiN_x. The silicon frames containing the microtensile beams were then cleaved from the wafer (Figure 2-2). Each 3" wafer provided a total of ten microtensile beams, assuming a 100% yield.

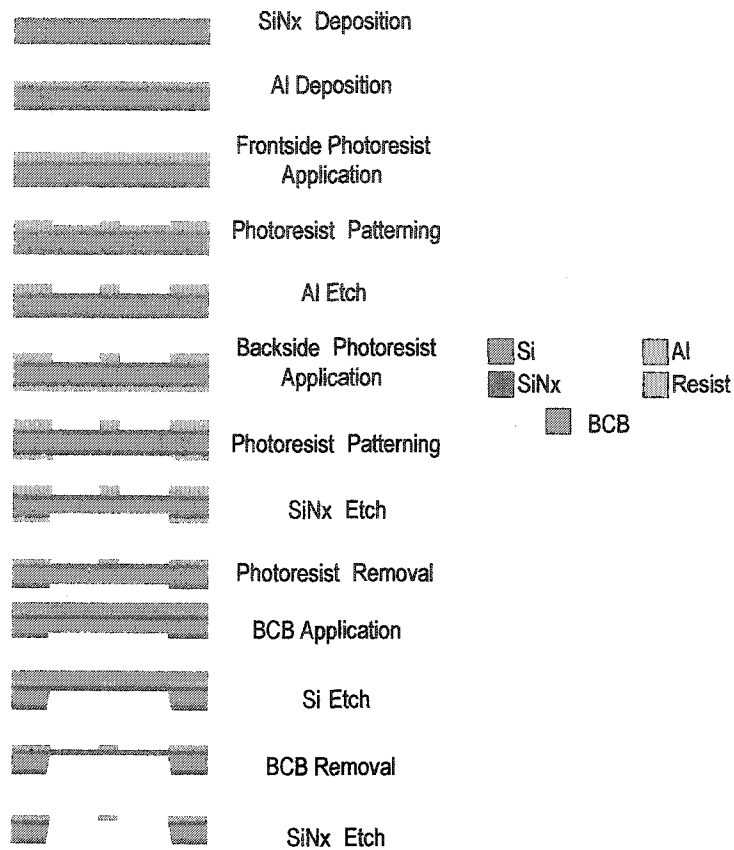


Figure 2-1: The fabrication procedure for producing freestanding Al microtensile beams.

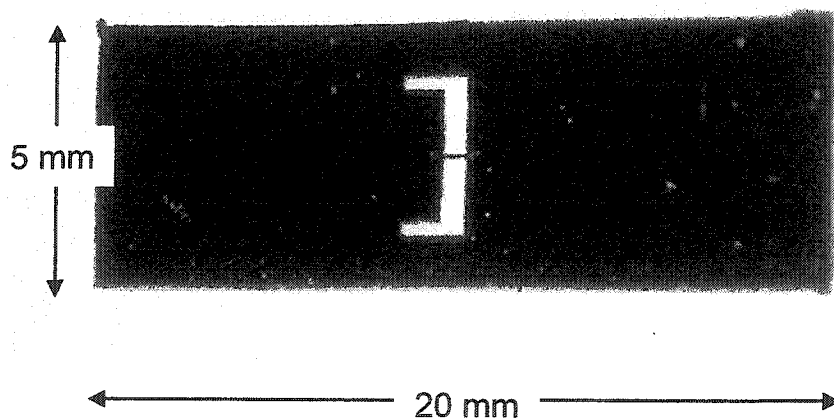


Figure 2-2: A Si frame containing a 1.00 μm thick Al microtensile beam.

2.2 Gage length correction factor

Fillets were designed at both ends of the microtensile beams to reduce the stress concentration at the point where the freestanding beam met the silicon support. For an ideal beam, the total length was 600 μm , and the uniform gage section was 500 μm long. The fillet geometry is designated in Figure 2-3 where the black line indicates the upper right fillet on the microtensile beam. Because the beam is symmetric, only one half of the right side is shown. At 600 μm , the edge of the Si starts. From 50 μm to 550 μm , the gage length is 100 μm wide. From 0 μm to 50 μm , the left fillet is present.

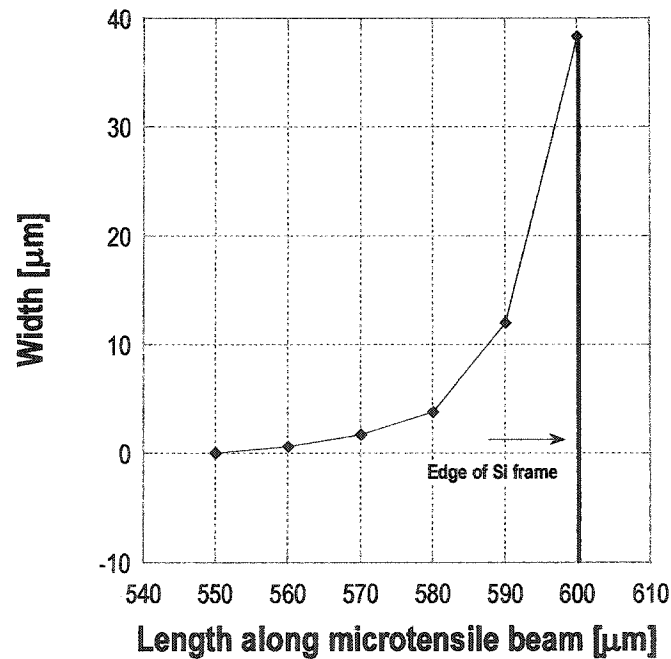


Figure 2-3: A plot of width vs. the length along the microtensile beam; the dots outline the shape of the upper right fillet on the microtensile beam.

During the Si-etching step (i.e. with the KOH) the Si was sometimes over- or under-etched (Figure 2-4). Because of this, the unsupported length of the microtensile beam was then no

longer the intended length of 600 μm . Therefore, when determining the effective elastic modulus, the real gage length needed to be determined. A correction factor was used when calculating the effective elastic modulus for instances when the under- or over-etching was uniform. Experiments were not carried out on microtensile beams having a non-uniform gage length (i.e. under- or over-etching only occurred on one end of the beam).

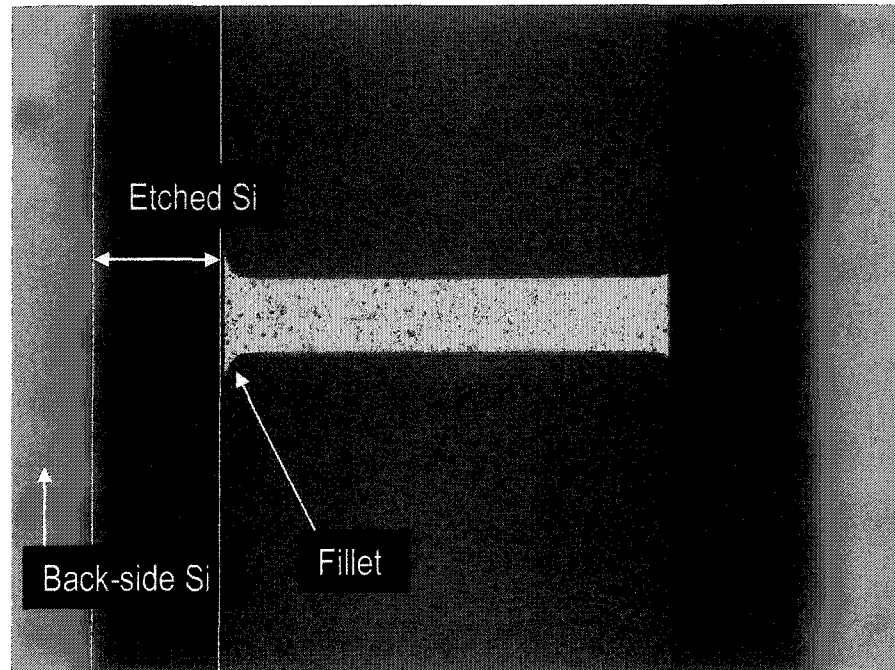


Figure 2-4: Light optical microscope image of the backside of a microtensile beam; notice the under-etching of the silicon on the right side of the microtensile beam (Mag. 80x).

Microtensile beams were sometimes produced having no fillets on either end; therefore, the gage length used was approximately 500 μm (the actual value was measured in a calibrated light optical microscope image). If no fillets were present on both ends, then this gage length used to determine the effective modulus did not need any additional correction factor.

However, if the full fillets were present, then a correction factor was necessary because the stress in the fillets was not uniform. The effective modulus could be multiplied by one of two

correction factors depending on which value of ideal gage length was assumed (i.e. if 500 μm was used, then the effective modulus was multiplied by 1.18; if 600 μm was used, then the effective modulus value was multiplied by 0.98). The reasoning for these two factors is explained using the following derivations.

The stiffness calculation is: $k \left[\frac{\text{mN}}{\mu\text{m}} \right] = \frac{E[\text{GPa}] \times t[\mu\text{m}] \times w[\mu\text{m}]}{l_o[\mu\text{m}]}$, where $E = 70[\text{GPa}]$,

$t = 1.00[\mu\text{m}]$, and $l_o = 1.00[\mu\text{m}]$. Each 10 μm long section along the fillet was divided into ten 1 μm long segments to obtain a detailed representation of the fillet. The compliance,

$\frac{1}{k \left[\frac{\text{mN}}{\mu\text{m}} \right]}$, was determined for all elements and was then totaled for each 10 μm long section

(i.e. 550 μm to 560 μm). The sum of the compliances for each 10 μm long section provided the compliance for the entire half fillet, which was 0.0063 $[\mu\text{m}/\text{mN}]$. The compliance of the 500 μm long microtensile beams was calculated to be 0.0714 $[\mu\text{m}/\text{mN}]$. The effective compliance of the gage length and the fillet is then $2 * 0.0063 [\mu\text{m}/\text{mN}] + 0.0714 [\mu\text{m}/\text{mN}] = 0.084 [\mu\text{m}/\text{mN}]$. To determine the correction factor when using a gage length of 500 μm , divide the compliance of

the gage length and by the effective compliance: $\frac{0.0714 \left[\frac{\mu\text{m}}{\text{mN}} \right]}{0.084 \left[\frac{\mu\text{m}}{\text{mN}} \right]} = 0.85$. This results in a

correction factor of $\frac{1}{0.85} = 1.18$. A similar calculation can be carried out for a gage length of 600 μm giving a correction factor of 0.98. The effective modulus is multiplied by 0.98 because it is an overestimate of the real value. For a 500 μm gage length used in the calculation,

$E_{corrected} = E_{500\mu m} \times 1.18$; and for a 600 μm gage length used in the calculation,

$$E_{corrected} = E_{600\mu m} \times 0.98 .$$

2.3 Characterization

Many different techniques were used to characterize the Al films in this research. Identical films were used in a parallel study of fatigue behavior of thin film Al, and many of the techniques and results have already been described in detail.⁶⁶ Therefore, they will only be summarized here. The exception is x-ray photoelectron spectroscopy (XPS), which will be discussed in more detail in Chapter 5. Table 2-1 lists the techniques and their results.

A cross-section analysis performed on all film thicknesses illustrates the columnar grain structure (Figure 2-5) that was typical of all films. In addition, film thickness measurements were performed from these cross-section images. Supplementary film thickness measurements were determined using quartz rate monitors during deposition and atomic force microscopy (AFM) post-deposition. The values of film thicknesses for all films tested were within a small percentage (approximately 1%) of the target values of 1.00 μm , 0.75 μm , 0.50 μm , and 0.25 μm .

Grain size analyses from plan-view transmission electron microscope (TEM) images showed the 0.75 μm and the 0.25 μm thick films to have an average grain size of 369 nm and 402 nm, respectively. The 0.50 μm thick films had an average grain size of 316 nm and the 1.00 μm thick films have an estimated grain size similar to that of the 0.50 μm thick films. These grain sizes were not expected based on the well-known rule for vapor-deposited films (i.e. "grain size $\propto$ film thickness"). It is not understood why the grain size trend did not scale

with film thickness, but one possibility may have been the presence of impurity elements. XPS spectra taken 400 Å below the top surface showed a higher N and O content for the 1.00 µm and the 0.75 µm thick film, respectively. This technique was performed on four different microtensile beams after being tested and the results are contained in Chapter 5 section 5.1.5. Grain size was the only observable difference among the films revealed using standard TEM techniques.⁶⁶ No voids were present, and no impurities at the grain boundaries were present (Figure 2-6).

Table 2-1: Summary of the characterization techniques performed on the microtensile beams.

Technique	Results for all four Al film thicknesses
Scanning electron microscopy	Similar surface topography; similar general film quality
X-ray diffraction	⟨111⟩ in-plane texture and no measurable impurities
Atomic force microscopy	As-deposited surface roughness similar to post-test surface roughness
Focused-ion beam microscopy	Columnar cross-sectional grain geometry
Transmission electron microscopy	Planar grain size varies for two sets of films
X-ray photoelectron spectroscopy	Impurity concentration at a depth of 400 Å below the top surface

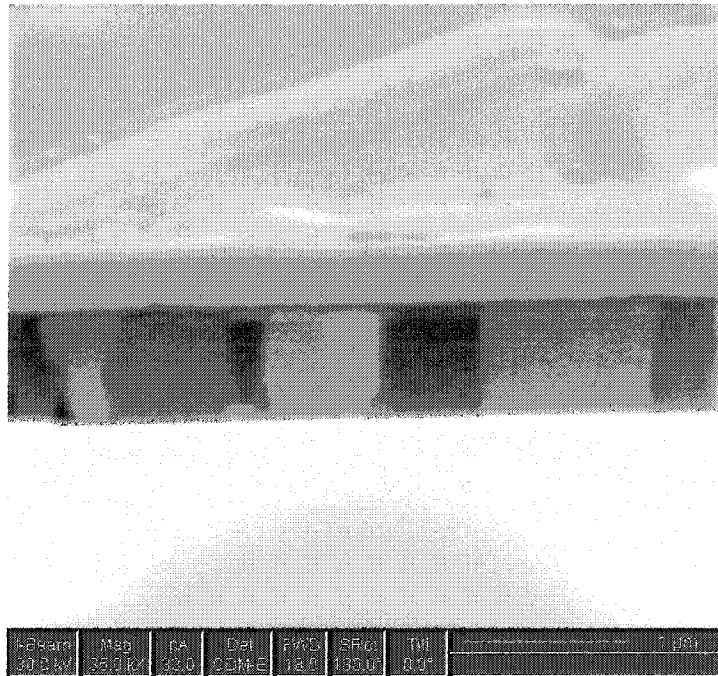


Figure 2-5: An I-beam image illustrating the columnar grain structure present in all the microtensile beams, this is a 1.00 μm thick Al microtensile beam (image taken by Nick Barbosa III).

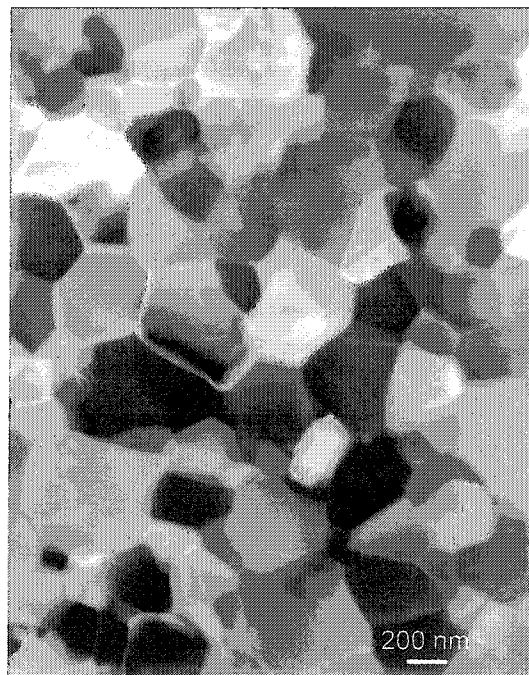


Figure 2-6: TEM image of a microtensile beam (image taken by Nick Barbosa III).

2.4 Naming the microtensile beams

A numerical naming system is used to identify the individual samples throughout the following chapters. It contains critical information about the specific fabrication batch, wafer number, die number, and film thickness. For example, consider microtensile beam 1A-1L-3-9; this sample is a 1.00 μm thick Al film (1A) deposited in the 1st batch at Lehigh University (1L) from the 3rd wafer in the 1st batch (3), and it is die number nine (9) on the 3rd wafer. Consider a second example; microtensile beam 0.5A-1L-2-4 is die #4 (4) from the 2nd (2) wafer of 0.50 μm thick Al films (0.5A) deposited in the 1st batch (1L) at Lehigh University.

3 Microtensile system design and operation

This chapter summarizes a brief history of the microtensile system designs built at Lehigh University. Four of the five main components of the microtensile system are discussed; the electronic circuits and LabVIEW® program flow, the piezo translator, the capacitance grip sensor, and the gripping and alignment devices. The load cell is the 5th and final component but will be discussed in detail in Chapter 4. Also, the procedure for aligning the grips is outlined.

3.1 System design overview

A custom microtensile system (Figure 3-1) was designed and built at Lehigh University. The design of this system was based on the microtensile systems built at Stanford University and NIST (Boulder, CO).^{37;67} Both the Stanford and NIST systems are horizontal; one disadvantage of the horizontal design gives rise to "sagging" in the grips, which may reduce the precision of measurements. The system built at Lehigh is vertical. Because it is vertical, the grips do not have a tendency to sag.

A total of four microtensile system designs were carried out with each new design improving upon the previous design. The five main elements of all system versions are control and data acquisition (DAQ) electronics, a piezo translator (which provides the crosshead displacement, or piezo displacement), gripping and aligning devices, a capacitance sensor (which is imbedded in the top grip and provides a measure of distance between the lower surface of the top grip and the upper surface of the bottom grip, or the grip displacement), and a load cell (which measures the applied stress on the microtensile beam). The piezo translator is the drive and the load cell is the response.

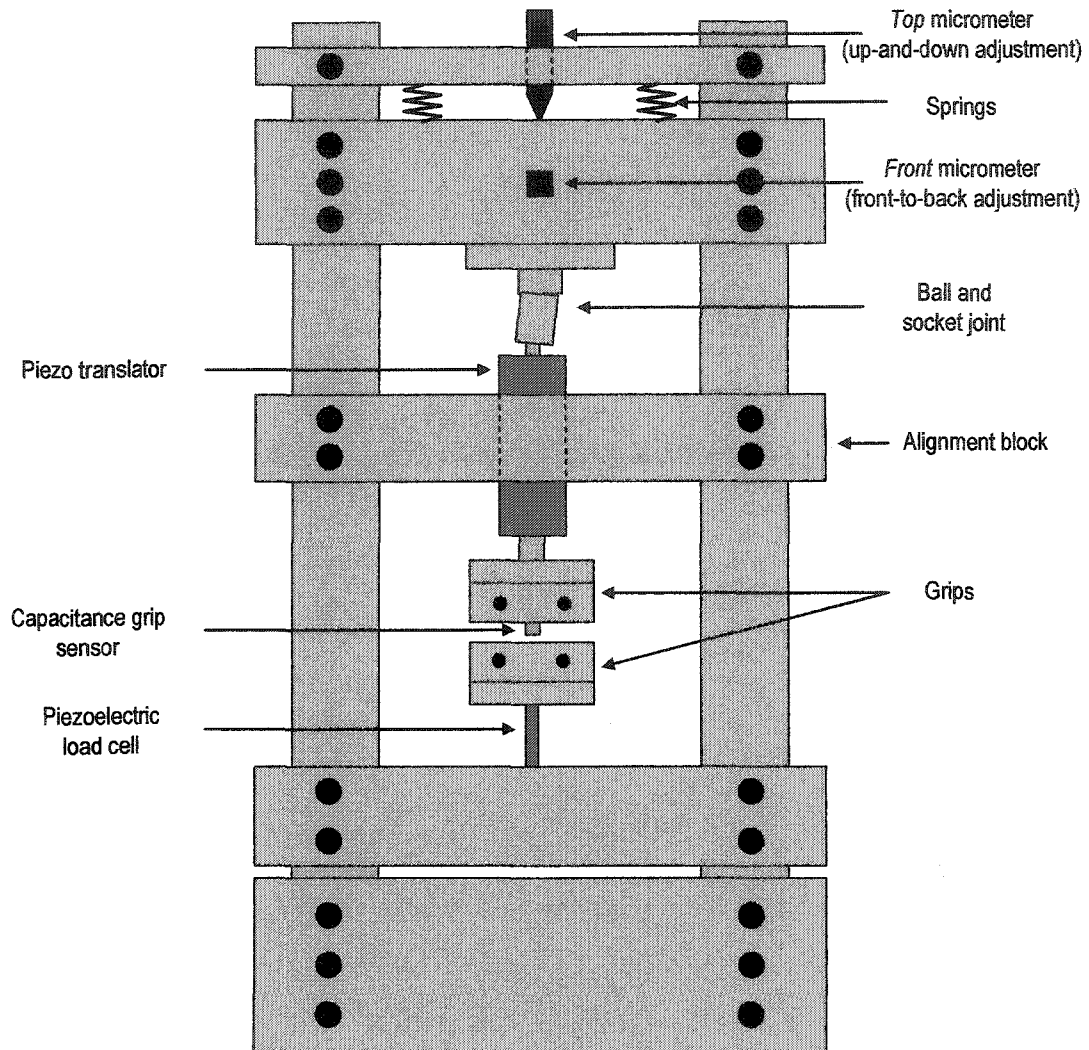


Figure 3-1: A schematic of microtensile system #4.

The control and DAQ electronics, the piezo translator, the gripping and aligning devices, and the capacitance grip sensor are consistent across all four systems. The piezo translator (Polytec PI model P239.47) has a total displacement range of 60 μm with a resolution of 10 nm. The capacitance grip sensor (Capacitec model HPT – 40) has a total displacement range of 50 μm and a resolution of 10 nm.

Similar to the Stanford system, the Lehigh system has two micrometers (at *front* and *top*) that allow front-to-back adjustments as well as up-and-down adjustments for the piezo translator (and the top grip, since it is attached to the piezo translator). For the Lehigh system, however, it was determined that these two adjustments were not adequate to provide proper alignment of the grips. Therefore, a grip alignment block was also added. The grip alignment block has three screws situated 120° from one another, and the purpose of these screws is to set the position of the piezo translator; the screws tighten down onto the stainless steel casing of the piezo translator.

Unlike the remainder of the system, the load cell underwent a series of four designs. The first load cell configuration (microtensile system #1) based on the microtensile system designed at NIST consisted of a single spring steel beam and a pair of capacitor plate sensors; the top capacitor plate was located on the underside of the steel beam. The bottom grip, located on the topside of the steel beam, was then attached to the top capacitor plate thereby sandwiching the steel beam. The bottom capacitor plate was mounted on a series of metal shims, which were eventually replaced by a z-stage. The z-stage allowed for height adjustment of the bottom capacitor plate, which was subsequently raised in order to move the two capacitor plates into the measuring range of 100 μm . One advantage of the force-sensing beam setup was its high sensitivity resulting in a load resolution of 0.1 mN. Drawbacks to this load cell configuration were its poor response to high strain rates and its poor front-to-back mechanical rigidity (i.e. many samples broke during the clamping and cutting process).

The second load cell configuration (microtensile system #2) consisted of a double titanium force-sensing beam (this one being more rigid than the single beam) where on the lower beam, the capacitor plate sensors were arranged the same way as on the single spring steel beam in

system #1. System #2 was originally designed and built for high temperature microtensile testing. Supplementary features included a longer load train necessary for the addition of a furnace to the center of the microtensile system. Therefore, in order to increase the front-to-back rigidity of this long load train, this double titanium force-sensing beam load cell was proposed and built. It turned out that other issues associated with high temperature testing gave rise to building an entirely separate system.⁶⁸ However, even when using this double titanium force-sensing beam load cell, the long load train was still mechanically unstable resulting in a low success rate for samples surviving the clamping and cutting process; the double titanium force-sensing beam load cell configuration was also tried with a short load train and proved to be more mechanically rigid resulting in a higher yield of samples surviving the clamping and cutting process. Unfortunately, similarly to the single spring steel force-sensing beam, the double titanium force-sensing beam load cell suffered from slow response times that did not allow measurement under high strain rate conditions.

The third load cell configuration (microtensile system #3) used a commercial semiconductor load cell (Honeywell Sensotec miniature load cell model 31). A Wheatstone bridge measures the resistance change in a piece of Si as it bends from applied loads. This load cell also was incapable of performing measurements at high strain rates; furthermore, it was affected by humidity making it unstable over long time periods. When analyzing experimental results, there was some difficulty in positively identifying the observed behavior as only originating with the load cell and nothing else in the load train. To ensure the observed behavior was associated only with the load cell and to examine how the rest of the microtensile system responded, a “solid” load cell (this was a solid piece of aluminum constructed to have the same stiffness as the commercial model 31 load cell) was exchanged for the commercial

load cell. The results from these experiments offered insight for the employment of the fourth and final load cell configuration.

The fourth load cell configuration (microtensile system #4) consisted of a commercial piezoelectric load cell (Kistler model 9203). This load cell has a very fast response behavior that permits precise stress measurements at high strain rates. Furthermore, it has a very high stiffness ($40 \text{ N}/\mu\text{m}$) allowing the sample strain to be measured from the piezo displacement. The resolution is 1 mN; for a $1 \mu\text{m}$ thick and $100 \mu\text{m}$ wide Al microtensile beam, this corresponds to a stress resolution of 10 MPa. Most of this resolution, however, is limited by vibrations throughout the entire microtensile system created by the 60 Hz feedback loop for the piezo translator. When the servo controller on the piezo translator is turned off (i.e. no feedback loop), the resolution of the piezoelectric load cell is increased by 10 making it 0.1 mN. Because of its unique design and high stiffness, the load cell-to-bottom grip connection is rigid making the clamping and cutting process very successful. All experiments performed on each microtensile system (#1, #2, #3, and #4) were displacement controlled (i.e. the piezo translator applied the strain while the load cell registered the response of the sample).

In the following sections, the design and operation of the common system elements will be described in more detail. Characterization of the four different system configurations will be presented in Chapter 4.

3.2 *Electronic circuits and LabVIEW® program flow*

All of the stress-strain results shown in this dissertation were recorded using a National Instruments data acquisition card (DAQ) and LabVIEW® programs. The DAQ card is version PCI-MIO-16XE-10, and is capable of recording both analog and digital voltages, as well as

temperature signals. It is a 16 bit card, or there are a total of 2^{16} bytes (65,536 bytes). The number of bytes determines the voltage resolution of the incoming analog signal; if the total voltage range is 20 volts, then the voltage resolution is 0.305 mV, calculated in the following

manner: $\frac{20[\text{volts}]}{65536[\text{bytes}]} = 0.000305 \left[\frac{\text{volts}}{\text{byte}} \right] = 0.305[\text{mV}]$. If the voltage range is

reduced by a factor of 2, or from 20 volts to 10 volts, then the voltage resolution is increased to 0.15 mV. A connector-block (model SCB-68) containing the voltage leads for all the input and output signals is connected to the DAQ card. For all the incoming signals (piezo transducer, capacitance grip sensor, piezoelectric load cell, box temperature, and voltage input to the piezo transducer), the voltage range on the DAQ card was set to 20 volts. The “box temperature” signal is coming from a thermocouple taped on the side of the top grip and measures the temperature inside the thermally-insulated box in which microtensile system #4 is located. The “voltage input to the piezo transducer” is the voltage that controls the position of the piezo transducer. Nick Barbosa III's Ph.D. dissertation covers the specifics of this signal as well as the LabVIEW® program created for controlling microtensile system #4.

This specific DAQ card is capable of 100,000 samples/second (100 kS/sec). This means that if nothing else is slowing down the acquisition rate, then the card can capture 100,000 data points in one second, or one data point every 10 μs . However, it was realized that with the current setup, the maximum allowable data acquisition rate was approximately 18,000 samples/sec. Above 18,000 samples/sec, a significant amount of backlog was occurring, which means that data points were being lost. The acquisition rate depends on the speed of the processor as well as the number of extraneous programs running during the acquisition. When

the number of programs is drastically reduced, the acquisition rate can be increased. For the results in this dissertation, 5,000 samples/sec was deemed sufficient.

By using binary data loggers in LabVIEW®, a large amount of data could be stored over small time intervals. However, a second program (i.e. “data conversion”) was necessary to convert the binary data to scaled data (or something that is recognizable as integers). After the data was converted, it was realized that the files were too large for many spreadsheet programs or graphing programs to handle. Therefore, a third program (i.e. “data picker”) was designed that allowed a specific area of data to be “cut” from the original data. These two programs were originally thought-up by Nick Barbosa III. They were especially helpful for looking at the fatigue data, which sometimes could run for 120 hours. Other programs were designed for specifically dealing with the fatigue data, but are not covered here.

3.3 *Piezo translator*

The piezo translator (Figure 3-2) provides crosshead displacement. A piezo translator is constructed in the following manner: a stack of ceramic piezoelectric crystal disks are aligned, glued together, and then housed in a stainless steel casing. A piezo crystal (usually referred to as PZT, where PZT is an acronym for plumbum, or lead, zirconium titanate) has the property that when a voltage is applied to the crystal, the crystal expands; or vice versa, if pressure is applied to the crystal, a charge is produced. The expansion of the crystal is measured by a strain gage. For more detailed information on piezo translators, suggested reading is the section titled “Tutorial: Piezoelectrics in Positioning” in the Polytec PI catalog, or online at www.polytecpi.com. The top grip is connected to the piezo translator by a 6 mm (M6) screw threaded into the piezo translator and a 5 mm (M5) screw threaded into the top grip. To ensure

flatness of the top grip to the piezo translator, a two-sided shoulder bolt was used (Figure 3-3). Therefore, the piezo translator and top grip are both tightened until the shoulder bolt is flat against the stainless steel housing of the piezo translator and the upper surface of the top grip.

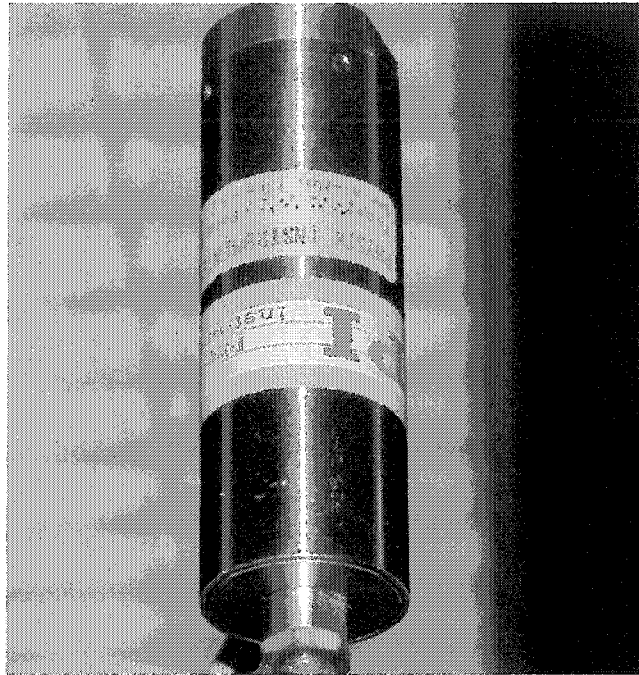


Figure 3-2: A digital image of the piezo translator mounted upside-down in the microtensile system.

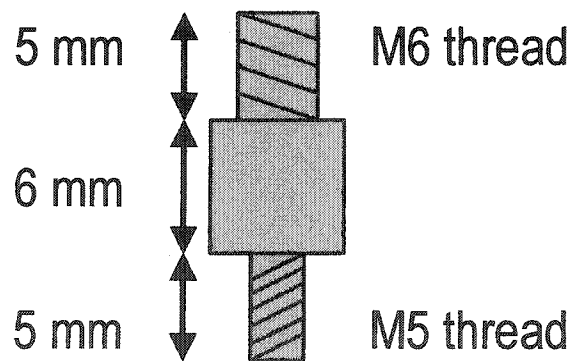


Figure 3-3: A schematic of the two-sided shoulder bolt used to connect the top grip to the piezo translator and the bottom grip to the piezoelectric load cell in microtensile system #4.

3.4 Capacitance grip sensor

For microtensile systems #1, #2, and #3 the grip displacement provides a more accurate measurement of sample strain than the piezo displacement due to the high compliance of these load cells. Figure 3-4 shows a schematic of the capacitance grip sensor, the grips and clamps, and the microtensile beam during experiments. The capacitance grip sensor protrudes from the lower surface of the top grip about 2.8 mm, as seen in Figure 3-5. This distance is adequate for allowing enough clearance between the two grips when cutting the side supports of the Si frame. The capacitance grip sensor measures the capacitance through the air between the sensor tip and the upper surface of the bottom stainless steel grip and relates that to a voltage and a physical distance through calibration techniques. At a nominal distance of 100 μm from the bottom grip, the capacitance grip sensor reads 10 volts. At 50 μm , it reads 0 volts and less than 50 μm it reads negative voltage values (starting from 0 and going to negative 10 as it approaches the upper surface of the bottom grip). Negative voltage readings are not good because the capacitance grip sensor is in danger of crashing into the bottom grip. Furthermore, the capacitance grip sensor is only calibrated over the range of 50 μm to 100 μm ; a total range of 50 μm .

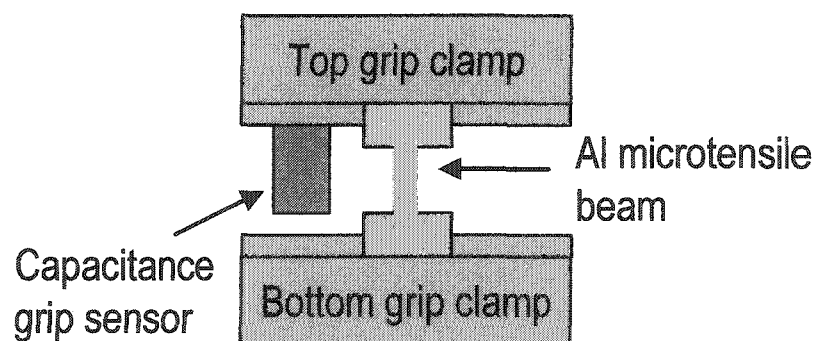


Figure 3-4: A schematic of capacitance grip sensor and grips during a microtensile beam experiment.

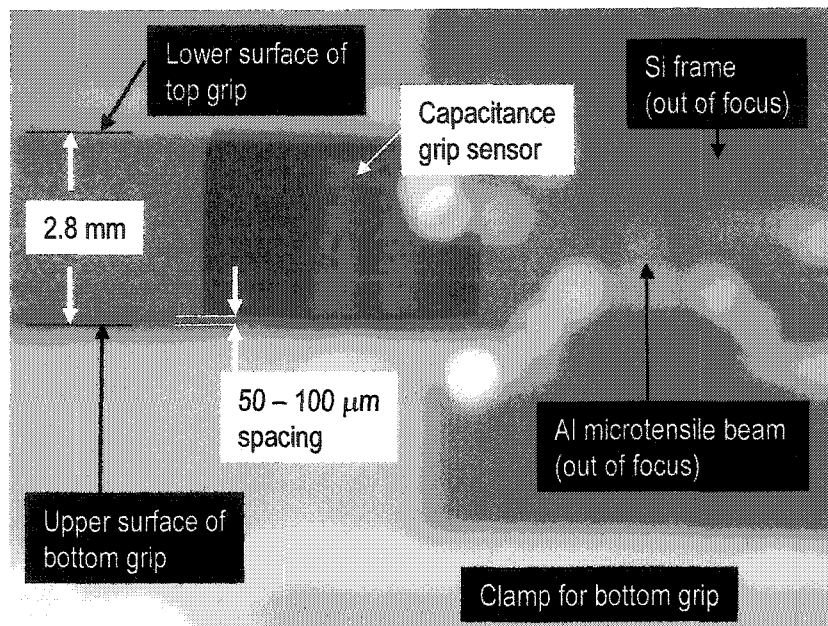


Figure 3-5: A stereomicroscope image showing the capacitance grip sensor, the spacing between it and the upper surface of the bottom grip, and the Al microtensile beam which is out of focus.

For all the load cells used (except the piezoelectric load cell on microtensile system #4), the grip displacement performed a sample strain measurement superior to the piezo displacement. This is due to the fact that the bottom grip moves during straining of the sample because of the low stiffness of the load cells; systems #1, #2, and #3 all had very compliant load cells. Therefore, just using the piezo displacement of the piezo as the sample strain overestimated the actual sample strain. However, the high strain rates being used on system #4 revealed additional problems associated with the electronics of the capacitance grip sensor. Therefore, on system #4 the piezo displacement was used for determining the sample strain. Because of the piezoelectric load cell's extremely high stiffness, a negligible amount of

movement is taking place in the bottom grip. Consequently, using the piezo displacement turned out to be an accurate measure for sample strain on microtensile system #4.

3.5 Gripping and aligning devices

When clamping the Si frame (with the suspended freestanding microtensile beam in the center) into the grips (Figure 3-6, Figure 3-7, Figure 3-8, and Figure 3-9), the side supports can support a very small amount of force. If the grips are misaligned, then most likely when clamping the Si frame the side supports will immediately fail rendering that sample useless for testing. Also, if the grips are not machined flat (i.e. if bumps or burrs are present), then there is a high probability the sample will break when clamping. For the sample to be successfully clamped, the grips need to be smooth, aligned, and flat relative to one another.

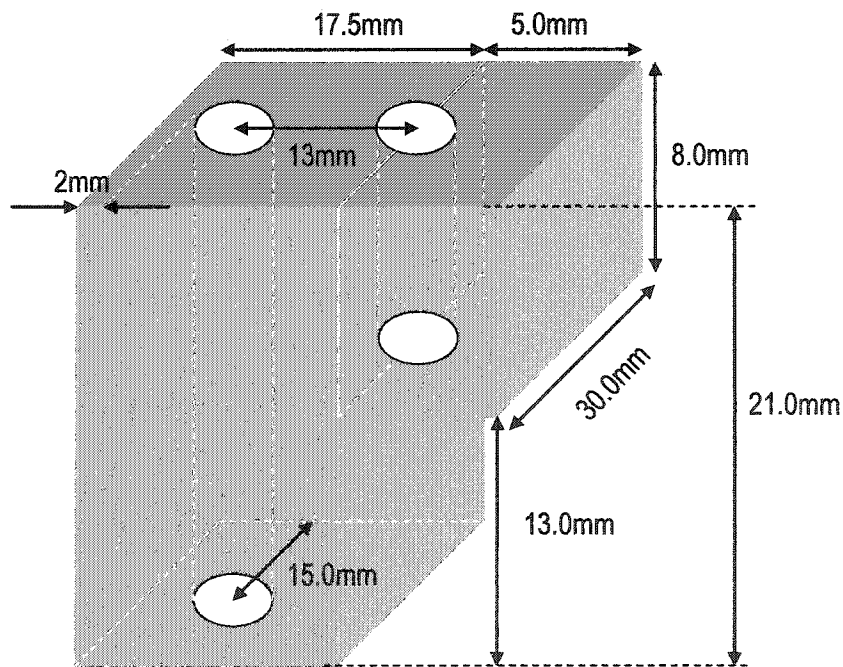


Figure 3-6: A schematic of the top grip; side view (not drawn to scale).

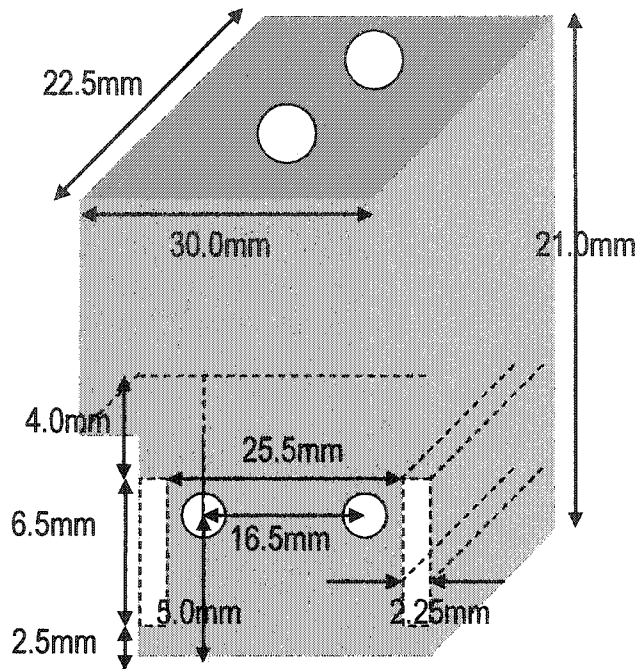


Figure 3-7: A schematic of the top grip; front view (not drawn to scale).

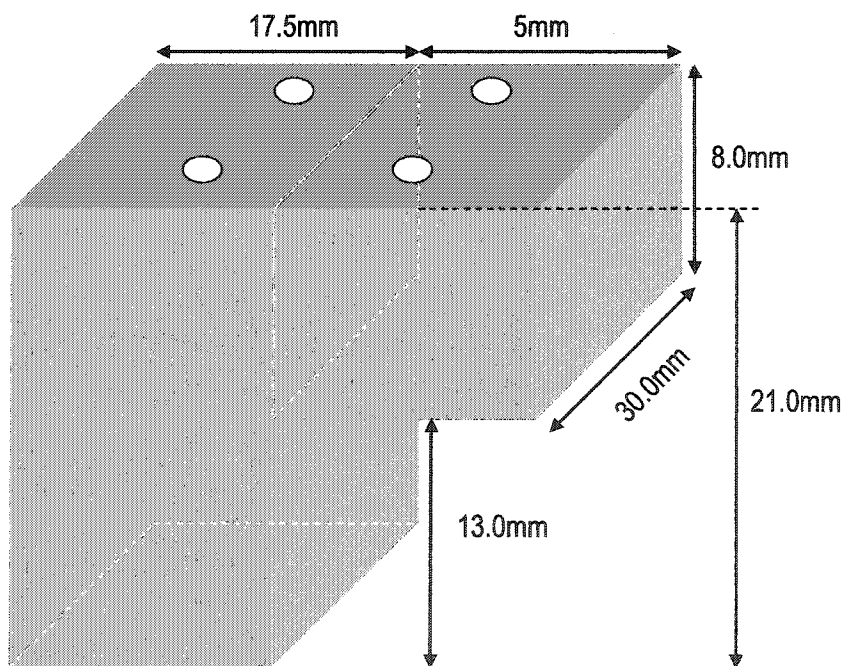


Figure 3-8: A schematic of the bottom grip; side view (not drawn to scale).

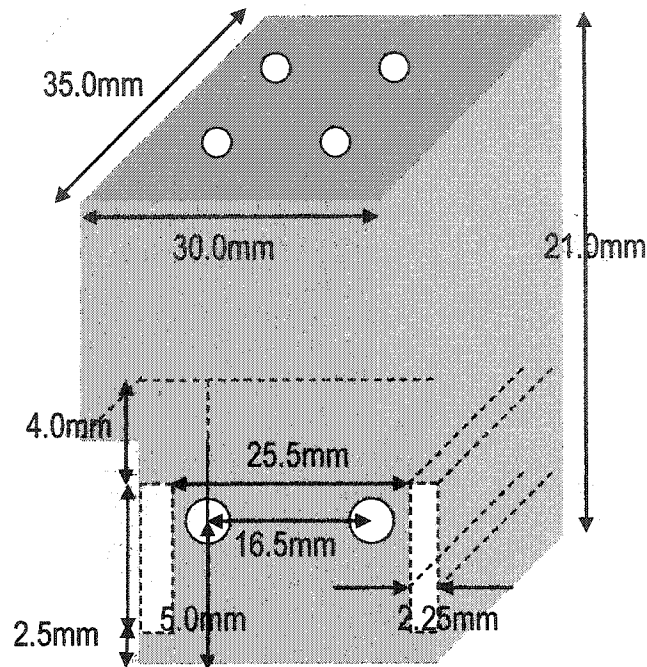


Figure 3-9: A schematic of the bottom grip; front view (not drawn to scale).

For system alignment, connecting the bottom grip to the load cell is a crucial step. For microtensile system #4, a two-sided shoulder bolt (similar to the connection between the piezo translator and top grip) was used for this purpose. This assured proper alignment of the grip with respect to the load cell. However, this presumes that the load cell is aligned to the remaining parts of the microtensile system. Alignment of the load cell to the stainless steel side rails of the microtensile system depends on the straightness of the tapped holes as well as the straightness of the stainless steel side rails. Once the bottom grip is connected to the load cell, the top grip is aligned to the bottom grip by making adjustments with the alignment block.

3.5.1 Clamps

The clamps (Figure 3-10) are necessary for securely holding the Si frame against the middle surface of the grip; if the clamp is tight, then slipping of the Si frame is prevented

resulting in a more controlled experiment. The screws originally used for fastening the clamp to the grip were spring-loaded single-sided shoulder bolts; the idea was to pull the clamp forward thereby compressing the springs, place the Si frame between the clamp and the middle surface of the grip, and then release the clamp. However, this process proved to be too delicate and very awkward; instead two ¼" 4-40 stainless steel type 316 screws were used to attach the clamp to the grip. They are magnetic and can easily be picked up with a magnetic screw driver. Furthermore, to ensure no slipping of the Si frame in the clamp, the holes were offset toward the surfaces of the grips; the bottom surface of the top grip and top surface of the bottom grip.

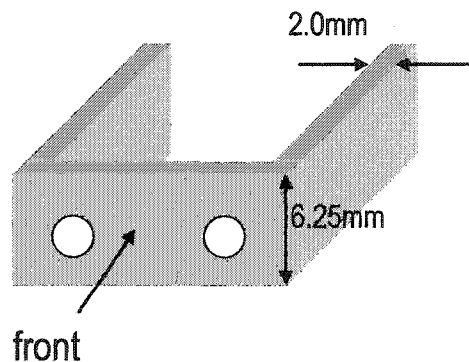


Figure 3-10: A schematic of one clamp; front view (not drawn to scale).

3.5.2 Grip alignment

Grip alignment is perhaps the most vital aspect of microtensile testing. The Si frame will not survive the clamping process when the grips are misaligned. However, if the misalignment is small and the Si frame happens to survive the clamping and cutting process, then most likely the sample is misaligned; and if the sample is misaligned then the stress measured at a specific strain will be lower than if the sample was perfectly aligned. For Al microtensile beams,

crystallographic planes would experience an increasing shear stress as the sample becomes more misaligned, and since the shear modulus of Al is about three times smaller than the Young's modulus, the applied load on the sample would be lower.

Surrounding the piezo translator is an alignment block (Figure 3-11) which keeps the positioning of the piezo translator stable while providing adjustments. In the alignment block are three screws positioned 120° apart from one another. The purpose of the alignment block is to align the top grip to the fixed bottom grip. The piezo translator is mounted near the top of the system on a ball and socket joint that allows free rotation and front-to-back as well as side-to-side adjustments.

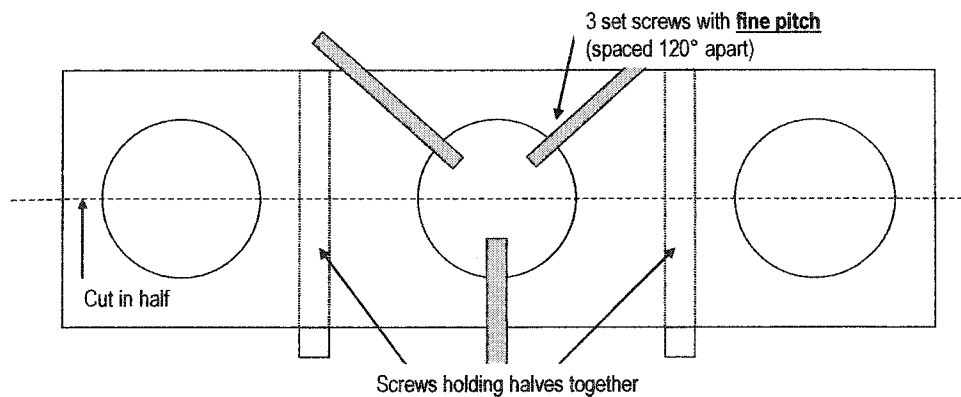


Figure 3-11: A schematic of the alignment block; top view.

3.5.2.1 Grip alignment procedure

The grip alignment procedure requires many steps, but is not difficult; therefore this section provides a set of instructions for completing this task. Once all the hard connections are made (i.e. piezo translator-to-top grip and bottom grip-to-load cell) the screws in the alignment block are withdrawn (so as not to make contact with the casing of the piezo translator) and the top

grip is roughly aligned to the bottom grip by hand. Before tightening the alignment screws, the top grip should be moved down toward the bottom grip so that the grip displacement reads between 40 μm and 50 μm (use the *top* micrometer and closely monitor the capacitance sensor for this adjustment: BE CAREFUL so as not to crash the capacitance grip sensor into the bottom grip). After the rough adjustments, the alignment screws are gently tightened onto the casing of the piezo translator, and a piece of polished, reflective Si is clamped into the grips (this does not have to be Si, but it must be flat and reflective). The Si piece is then cut in half (Figure 3-12) using a 3" Norton[®] diamond saw blade and a high speed Dremel[®] tool (Figure 3-13); the end result is that one half is clamped into the top grip and the other half in the bottom grip with the cut edges approximately parallel to each other.

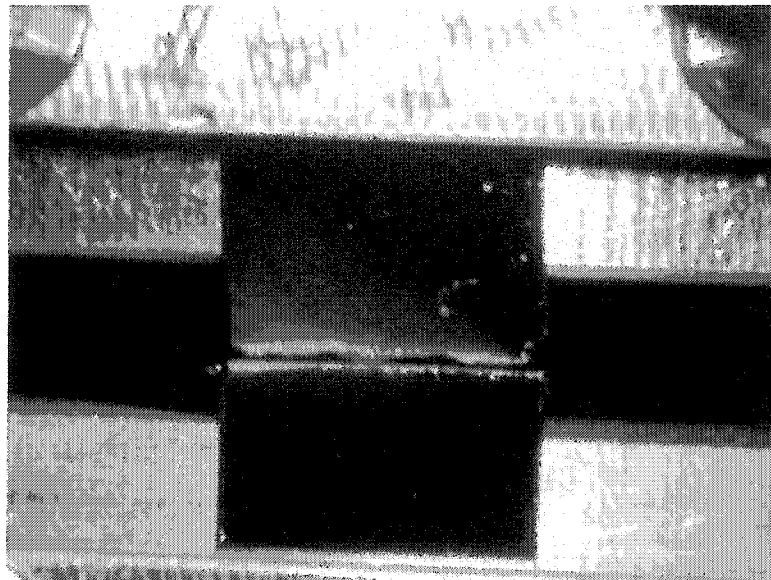


Figure 3-12: A digital image showing the two halves of the Si piece after cutting. The red dot is the incident laser beam; the laser beam is split so that half is on both the top and bottom pieces of Si.

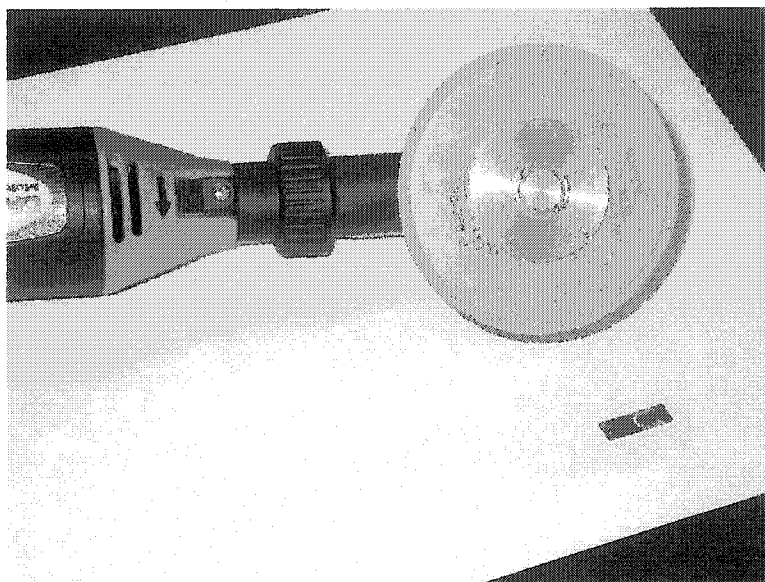


Figure 3-13: A digital image of the high speed Dremel® tool and Norton® diamond saw blade; note the Si frame containing an Al microtensile beam in the lower right hand corner.

The piezo translator is then extended until the grip displacement reads approximately 30 μm . It is good practice to operate both the piezo translator and capacitance grip sensor in the middle of their total ranges (30 μm is a good distance for both). Use the *top* micrometer to make additional adjustments. A laser pointer (which is on an x-y-z stage) is then set up so that the incident beam illuminates both the top and bottom Si pieces (Figure 3-12). Additionally, a stereomicroscope is set up so that the Si pieces can be viewed from about a 70° angle off center (Figure 3-14). Figure 3-15 outlines the complete grip alignment procedure.

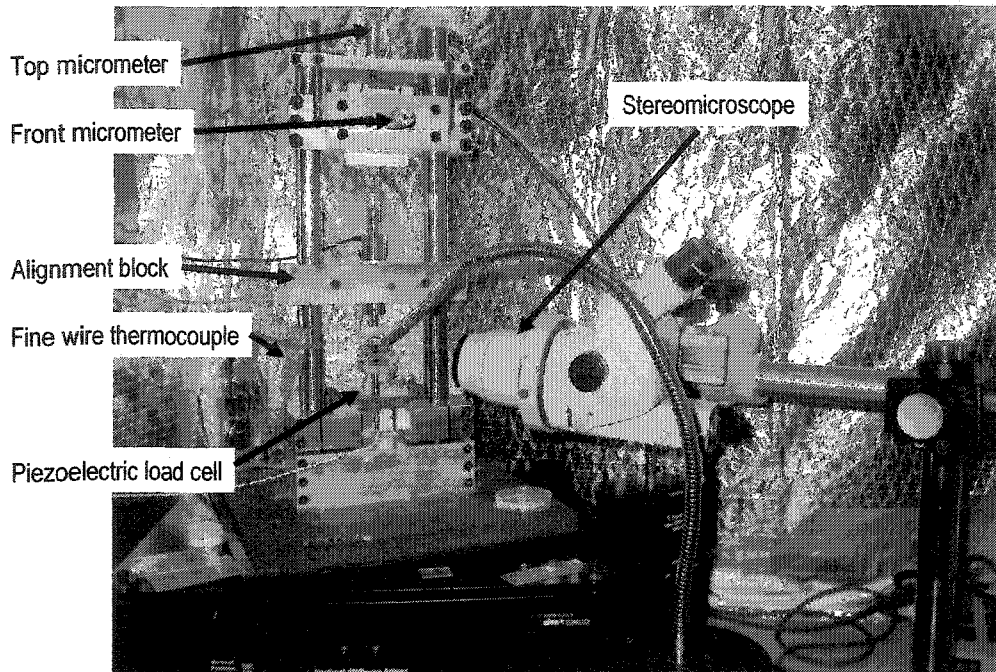


Figure 3-14: The alignment setup showing microtensile system #4 and the adjacent stereomicroscope (both are contained inside the temperature-controlled thermal insulating box).

Consider the reflected laser beams: if the grips are not aligned in rotation, the reflected beam (spot) from the top grip will not match the position of the reflected beam from the bottom grip. Both reflected laser beams should be lined up so that the spots are on top of one another; however, adjustments can be made only to the beam reflecting off the Si piece in the top grip because the bottom grip has a fixed position. During the alignment procedure extra care is necessary with constant monitoring of the grip displacement. Rotating the top grip (i.e. hold the piezo translator and rotate CW or CCW) moves the reflected spot left-and-right (Figure 3-16). This enables the reflected spots to be aligned onto the same vertical line. The next step is to move the reflected spots onto one another (Figure 3-17). Once the reflected spots are aligned onto one another, look in the stereomicroscope to see how far one piece is in front (or behind) the other. Keeping the reflected beams together in one spot, translate the top grip backward (or

forward) using the *front* micrometer and the alignment screws while periodically looking in the stereomicroscope to see when the two pieces of Si are aligned with one another. If the top piece of Si is in front of the bottom piece, then loosen the two rear alignment screws and translate the front grip backward by turning the *front* micrometer clockwise. On the other hand, if the top piece of Si is behind the bottom piece, then loosen the front alignment screw and translate the front grip forward by turning the *front* micrometer counter clockwise. Look in the stereomicroscope; the two pieces of Si should appear only vertically separated and the reflected beams should produce one spot (Figure 3-18).

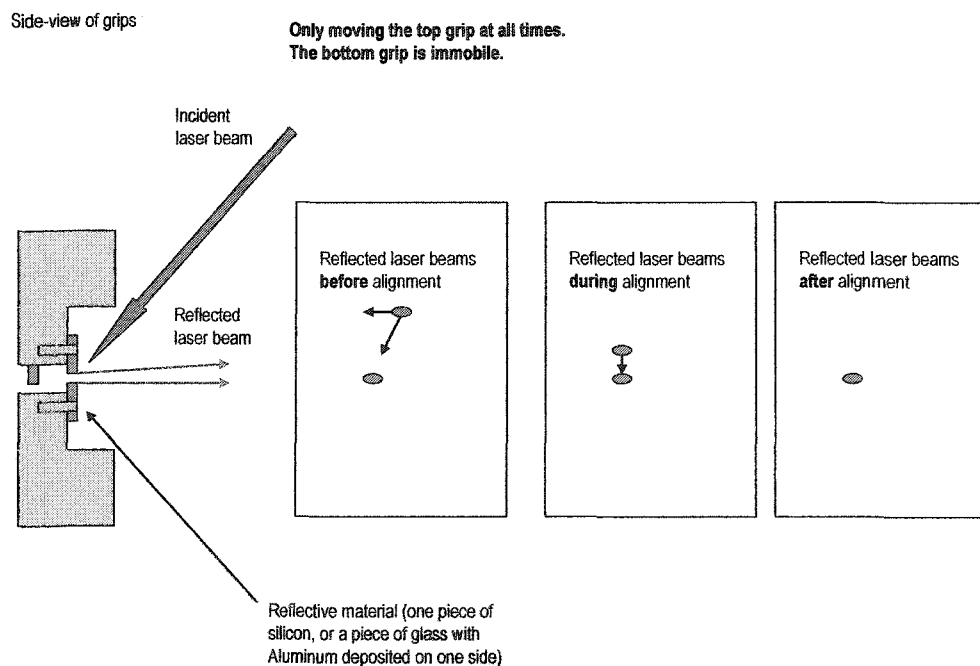


Figure 3-15: Grip alignment procedure; reflected laser beam spots before, during, and after alignment.

Side-view of grips

Only moving the top grip at all times.
The bottom grip is immobile.

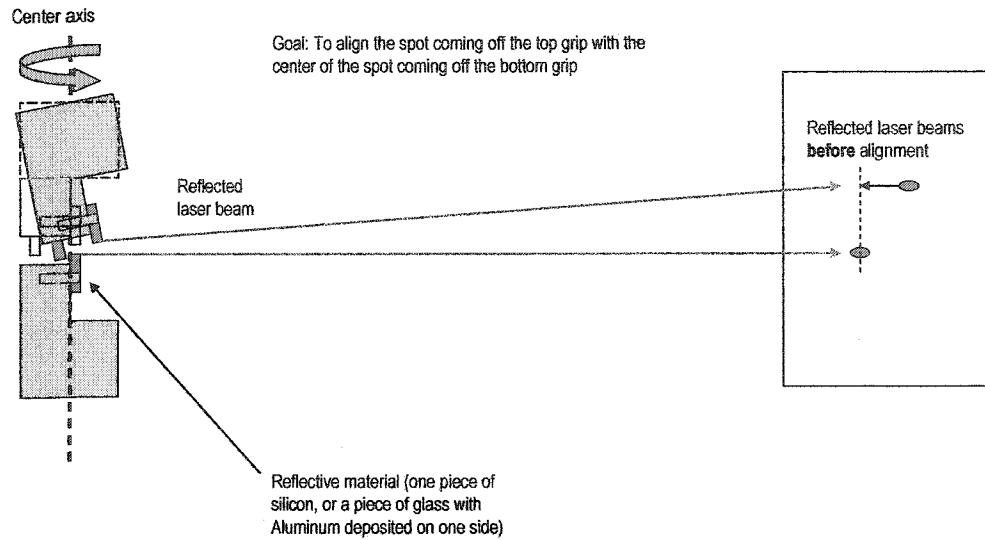


Figure 3-16: Grip alignment procedure; aligning reflected beam spots onto one vertical line.

Side-view of grips

Only moving the top grip at all times.
The bottom grip is immobile.

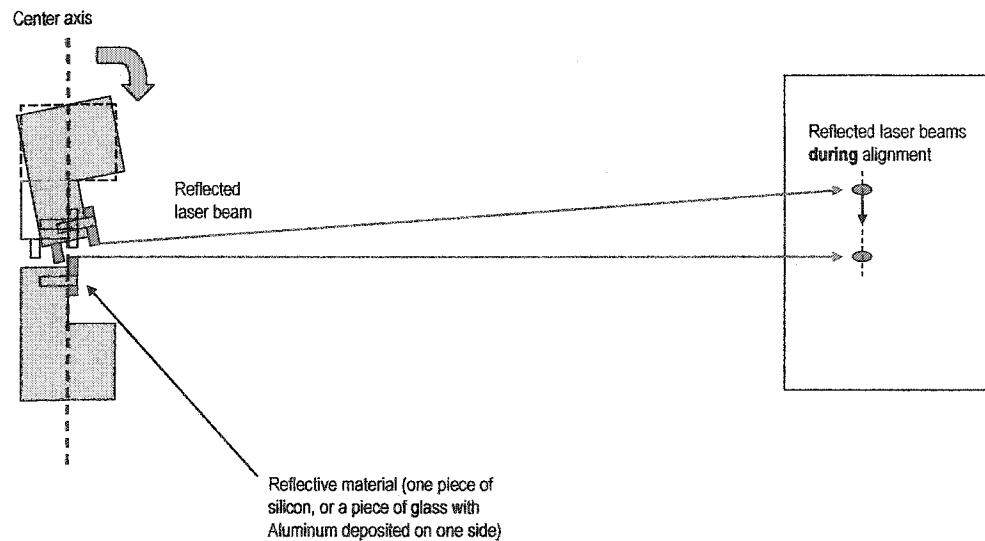


Figure 3-17: Grip alignment procedure; moving the reflected beam spots onto one another.

The top grip is aligned to the bottom grip using the screws in the alignment block as well as the *front* micrometer for the front-and-back alignment. This method aligns the middle surfaces of the grips to one another; the Si frame is clamped against the middle surface of the grips during testing. As an extra alignment inspection, the backs of the grips should be parallel to one another; this is accomplished by placing a glass microscope slide against one grip and looking for a gap between the other grip and the glass slide. Alignment adjustments with the *front* micrometer and alignment screws can also be done using this technique.

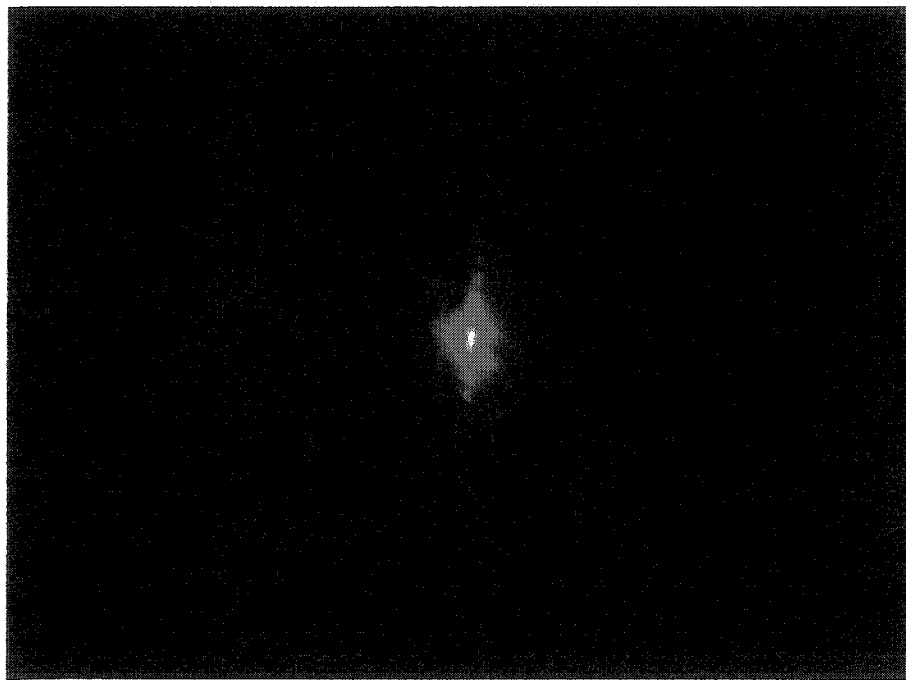


Figure 3-18: A digital image showing the single reflected beam spot from the aligned grips.

4 Microtensile system characterization and initial experiments

This chapter begins by characterizing the time response of the piezo translator and the capacitance grip sensor, which is essential to understand when designing feasible experiments. This section is followed by a complete characterization of the load measurement techniques used for each of the four different system configurations. Included are preliminary experiments that were performed using each configuration. These experiments were tremendously beneficial for understanding the behavior of each specific system configuration and provided the necessary information for moving ahead with the next configuration. Finally, experiments performed on the AI microtensile beams using the 3rd and 4th system configurations are discussed.

4.1 Time response of the piezo translator and capacitance grip sensor

A square wave, using the lowest frequency setting on the waveform generator (1 Hz) for a “pulse” wave function, was input to the piezo translator in order to determine the time response of the piezo displacement to the voltage input. High strain rate and 100 Hz frequency experiments will use this piezo translator, so characterizing the response as a function of time is necessary when designing achievable experiments. For Figure 4-1 through Figure 4-3, the piezo displacement and grip displacement [μm] are on the left axis (blue & red curves) and the drive input [volts] to the piezo translator are on the right axis (green curve). Also, both the piezo displacement and grip displacement were normalized; the grip displacement was multiplied by -1 to visually match the piezo displacement. Figure 4-1 shows the complete square wave; the second plot (Figure 4-2) shows the first transient, and the third plot (Figure 4-3) shows the

second transient. Both transients exhibit the same behavior. Figure 4-4 shows a simple exponential simulation (Equation 4-1) of the first transient for the piezo translator displacement:

$$y = -2.05 \cdot \left(1 - \exp\left(-\frac{t - 0.1458[\text{sec}]}{\tau[\text{sec}]}\right) \right)$$

Equation 4-1: Exponential fit to square wave.

where $\tau = 0.003, 0.002, 0.0015, \text{ and } 0.001$ [sec]. As τ approaches zero, the exponential fit more closely approximates the square wave. As seen in Figure 4-4 the piezo translator response to the square wave does not track this simple exponential, but instead has some initial acceleration followed by some constant speed and then deceleration.

For this measurement, the scan rate of the LabVIEW® data acquisition (DAQ) software was set to 5000 scans/second, which corresponds to recording a data point every 0.0002 seconds (for more details about DAQ electronics and LabVIEW® setup, consult section 3.2). The linear region of the piezo response is between $0.25 \mu\text{m}$ and $1.5 \mu\text{m}$. The displacement rate in this region is approximately $\left(\frac{1.50 - 0.25[\mu\text{m}]}{0.149 - 0.147[\text{sec}]} \right) = 600 \left[\frac{\mu\text{m}}{\text{sec}} \right]$; this value is slightly higher than the fastest displacement rate of $500 \mu\text{m/s}$ called for in the experiments. Instead of using the linear range of the curve between $1.5 \mu\text{m}$ and $0.25 \mu\text{m}$, the total range of $2 \mu\text{m}$ was traveled in 0.005 seconds ($0.151 - 0.146$ [sec]); this total range of $2 \mu\text{m}$ includes the acceleration and deceleration regions of the curve. The average displacement rate over this interval is $400 \mu\text{m/s}$.

Figure 4-3 shows a time delay of 0.002 seconds ($0.650 - 0.648$) for the grip sensor lagging behind the piezo translator. This delay is present in all experiments but is only apparent when

operating under faster data acquisition rates than the delay time (i.e. faster than 500 scans/sec). The 200 Hz filter in the Capacitec® circuit is not responsible for this time delay because the numerical calculation for the time delay of a 200 Hz filter gives:

$$\frac{1}{2\pi \cdot f} = \frac{1}{2\pi \cdot 200[\text{Hz}]} = 0.000795[\text{sec}].$$

This value of 0.000795 seconds is about 2.5 times

smaller than the experimentally measured value of 0.002 seconds. Presently, it is still not known what is causing this delay of 2 milliseconds in the grip displacement signal, but most likely it is somewhere in the Capacitec® circuit and not a physical reason. Because of this time delay in the grip displacement, the piezo displacement was used for measuring sample strain when running at high strain rates. System #4 was especially important for these measurements because the high stiffness of the piezoelectric load cell allowed the strain in the microtensile beam to be accounted for by the piezo displacement.

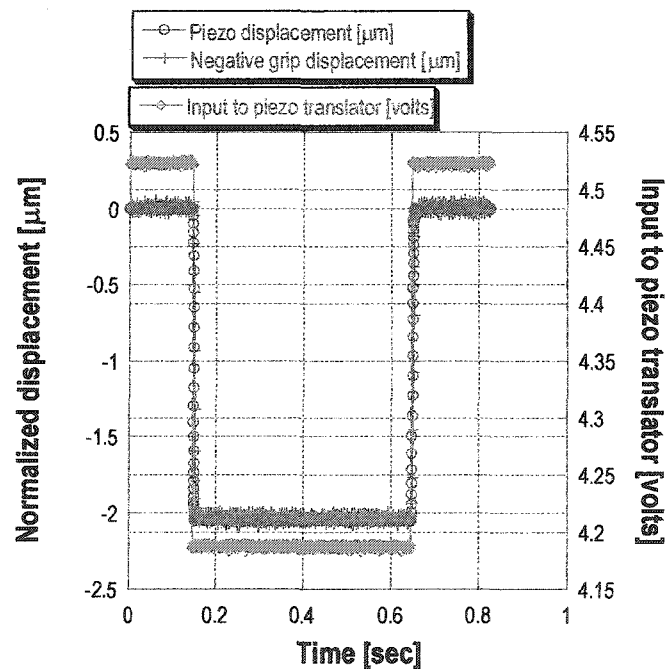


Figure 4-1: A plot of normalized displacement and voltage input vs. time showing the response of the piezo displacement and grip displacement to a square wave voltage input.

Created by Paul El-Deiry

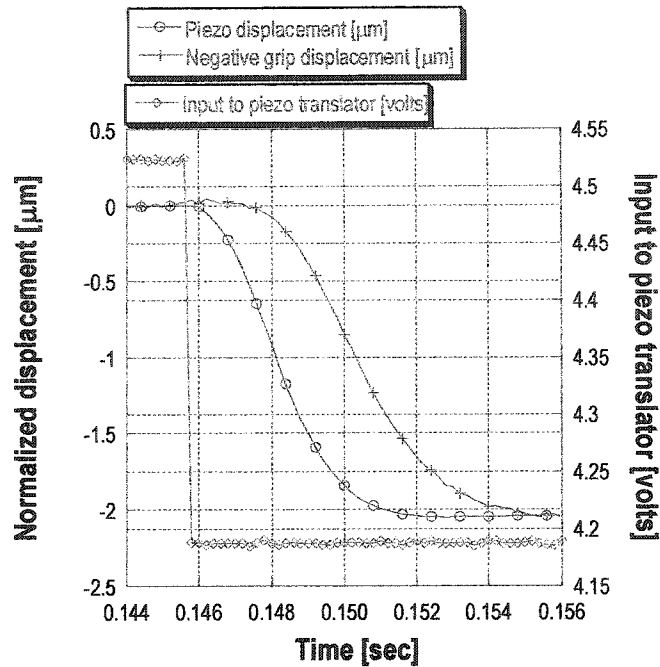


Figure 4-2: A plot of normalized displacement and voltage input vs. time showing the first transient response to a square wave voltage input.

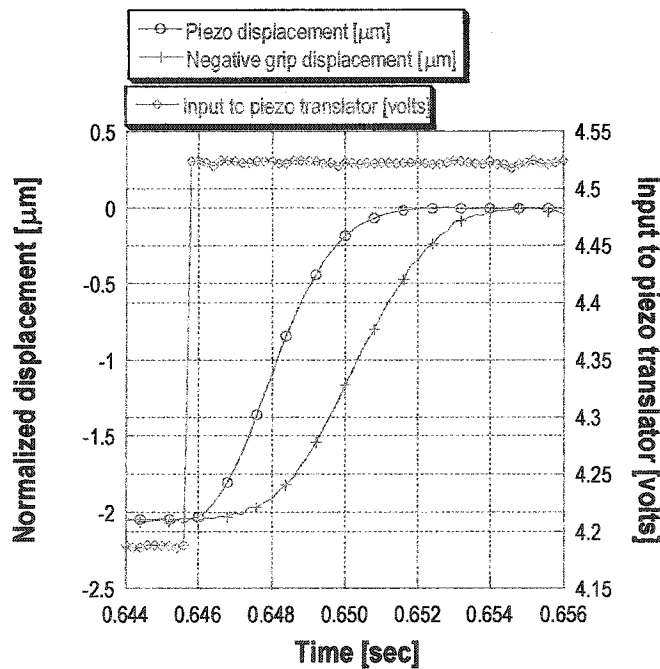


Figure 4-3: A plot of normalized displacement and voltage input vs. time showing the second transient response to a square wave voltage input.

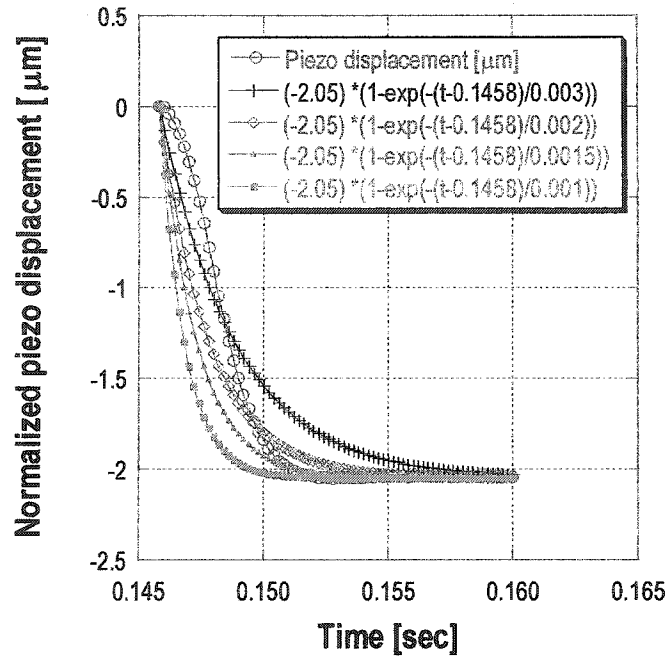


Figure 4-4: A plot of normalized piezo displacement vs. time showing the single exponential fit to the normalized piezo displacement.

4.2 Load measurement techniques

There were four distinct load measurement setups: microtensile systems #1, #2, #3, and #4. Microtensile system #1 consisted of a single spring steel beam and a pair of capacitor plate sensors from Polytec PI. The distance between the two plates was monitored and calibrated to a load applied on the steel beam. Microtensile system #2 had two titanium beams with the same capacitor plate sensors; a different calibration constant was determined. Microtensile system #3 used a Honeywell Sensotec precision miniature model 31 tension/compression semiconductor load cell, and microtensile system #4 currently uses a Kistler model 9203 piezoelectric load cell.

With each of these system configurations, experiments and system tests were run in order to investigate the behavior of the materials that were being tested, and more importantly, the performance of the microtensile system. Attempts were made at understanding each of these experiments, both as to what was happening with the sample as well as the microtensile system. At the time of writing this report, there was no standard thin film sample that was mechanically well understood. Experiments included testing thin films fabricated at Stanford University to testing pieces cut from extra Si wafers here at Lehigh University. The Si pieces at least provided a very rigid sample which allowed a more thorough analysis of the microtensile system.

4.2.1 Microtensile system #1: single spring steel force-sensing beam

The load cell configuration for system #1 was based on a design implemented by Read et al. at the National Institutes of Standards and Technology (NIST) and suggestions from Cornella et al. at Stanford University.^{37,67} A single spring steel force-sensing beam load cell configuration was designed using a single beam of spring steel and two parallel plates of a capacitor which measured the deflection of the steel beam. A spreadsheet (Figure 4-5) was created for selecting beam geometry and beam material based on material properties and the mechanics for the point-load bending of a clamped-clamped beam. The spring steel beam was sandwiched between the lower surface of the bottom grip and the top capacitor plate. The bottom capacitor plate was positioned below the top plate using metal shims (Figure 4-6). Setting this up was a tedious process and did not ensure the capacitor plates being parallel to one another. Information on how the capacitor plates operate can be found in the Polytec PI catalog or online at www.polytecpi.com. An output voltage of -5.0 volts is registered when the

This load cell configuration resulted in a high signal-to-noise ratio because of the high sensitivity and excellent thermal stability of the capacitor plate sensors. With this setup, the load resolution was 0.1 mN. After the parallelism of the sensors was determined to be critical for measurements, a z-stage was purchased to which the bottom capacitor plate sensor was then attached (Figure 4-7). The z-stage offered three advantages: 1) the capacitor plate sensors were parallel to one another, 2) the bottom capacitor plate could be moved into range when an experiment was being performed (this offered a safety feature so the sensors did not crash into one another), and 3) shim stock did not have to be used anymore. Once the capacitor plates were within their measuring range of 100 μm , the beam deflection was then calibrated to a value of applied load using a series of weights. Figure 4-8 shows a plot of the capacitor plate sensors (i.e. load sensors) displacement vs. time. Each plateau and valley is associated with placing a weight on and then removing it from the bottom grip, respectively. A calibration curve (Figure 4-9) was created by measuring the difference between the plateaus and valleys, and then plotting it against the associated weight. Calibration runs were performed each time the system was taken apart and put back together or if there was a long time between tests when the system was not being used.

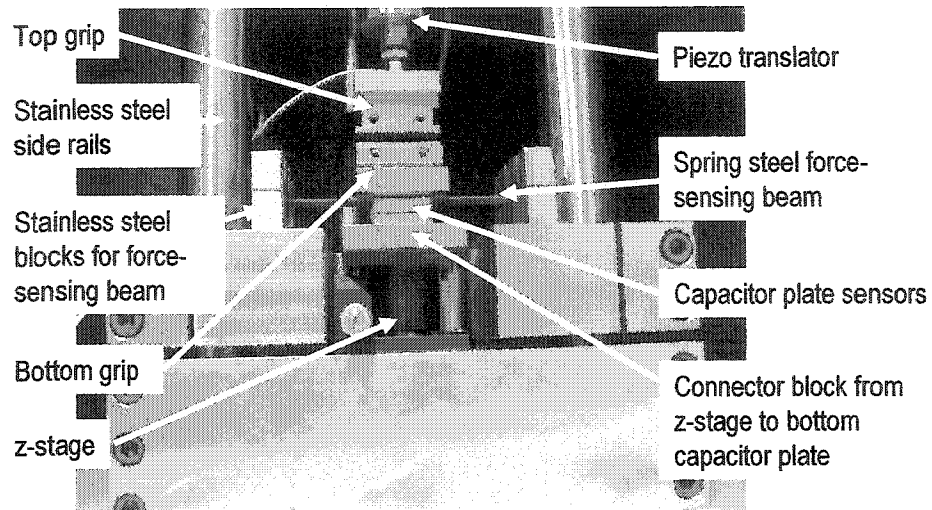


Figure 4-7: A digital image of microtensile system #1 with the single spring steel force-sensing beam load cell.

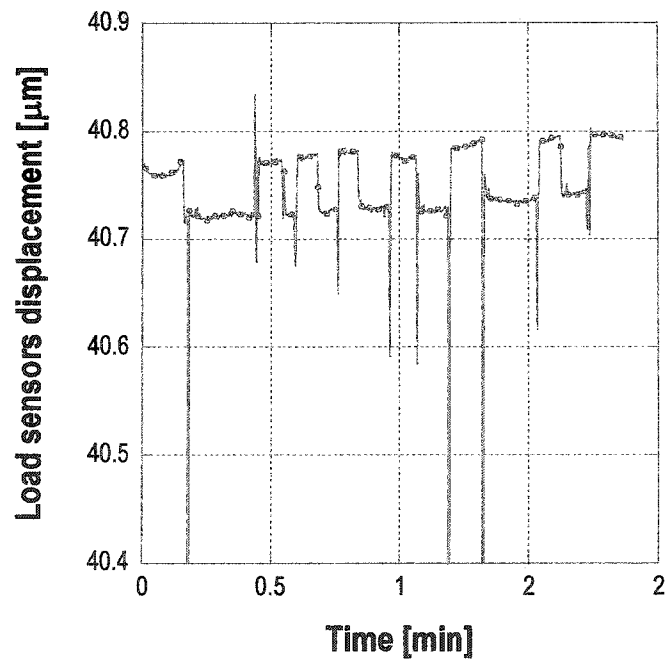


Figure 4-8: A plot of the load sensors displacement vs. time showing when a weight is placed onto the bottom grip multiple times.

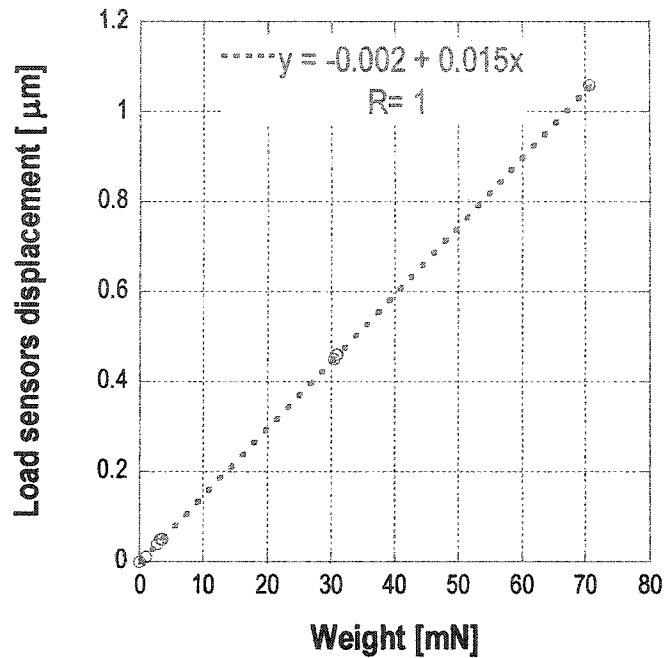


Figure 4-9: A plot of the load sensors displacement vs. weight showing the calibration curve for the single spring steel force-sensing beam load cell; the calibration constant = 0.0015 $\mu\text{m}/\text{mN}$.

4.2.1.1 Microtensile system #1 experiments

For microtensile systems #1 and #2, Al microtensile beams were not yet fabricated at Lehigh University. Therefore, the test samples were extra AlTi microtensile beams of various thicknesses that were available courtesy of Stanford University, 1 μm thick SiO_2 microtensile beams fabricated at Lehigh University (by Ming Tzer-Lin), commercial 75 μm diameter Al_2O_3 fibers, and commercial 10 μm diameter Al and Cu wires.

4.2.1.1.1 AlTi microtensile beam

An AlTi microtensile beam fabricated at Stanford University was tested on microtensile system #1. The dimensions of this microtensile beam were similar to the microtensile beams

later fabricated at Lehigh University except that the AlTi microtensile beam was 50 μm wide instead of 100 μm wide. The gage length (600 μm) is the same and the fillet geometry is similar, but not exactly the same. Figure 4-10 shows the stress-strain plot from a 1 μm thick AlTi microtensile beam. This experiment confirmed that microtensile system #1 was capable of testing a microtensile beam, and proved that the single spring steel force-sensing beam load cell was capable of measuring the applied force on the microtensile beam. However, this system did not have the integrated alignment block, which may have resulted in lower measured loads than if the system was correctly aligned. Also, the quality of this microtensile beam was not determined; if the quality was poor, then this may have resulted in the rollercoaster effect observed in the stress-strain plot. This rollercoaster effect could also have been a result of the Si frame slipping in the clamps. The displacement rate of the piezo translator was a constant 0.068 $\mu\text{m/s}$, which corresponded to a strain rate of $1.13 \times 10^{-4} \text{ s}^{-1}$,

calculated as:
$$\left(\frac{0.068 \left[\frac{\mu\text{m}}{\text{sec}} \right]}{600 \mu\text{m}} \right) = 1.13 \times 10^{-4} [\text{sec}]^{-1}.$$
 The data acquisition scan time was set

at approximately 2 scans per second; two data points were recorded every second (for more details about DAQ electronics and LabVIEW® setup, consult section 3.2).

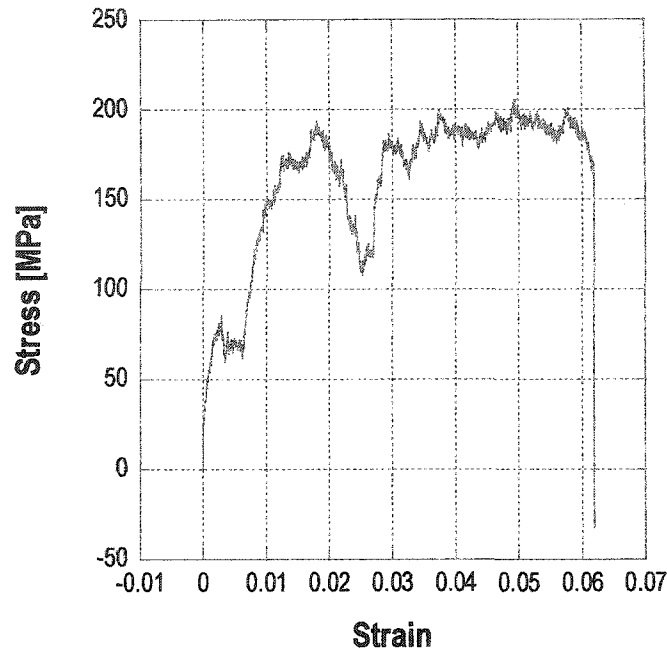


Figure 4-10: The stress-strain plot for an AlTi microtensile beam tested on microtensile system #1.

4.2.1.1.2 *SiO₂ microtensile beams*

Additional experiments were performed on SiO₂ microtensile beams. The SiO₂ microtensile beams were 100 μm wide, 600 μm long and 1 μm thick. Figure 4-11 is a light optical microscope (LOM) image of a SiO₂ microtensile beam that cracked during fabrication, and Figure 4-12 is an image of a SiO₂ microtensile beam clamped in the grips and being tested. Despite being a difficult task, a Si frame containing a SiO₂ microtensile beam was eventually clamped, cut and the freestanding SiO₂ microtensile beam tested. Figure 4-13 shows the stress-strain plot. The strain rate was $8.0 \times 10^{-5} \text{ s}^{-1}$. The measured elastic modulus was 64 GPa which compared well to the reported elastic modulus of the bulk material, 70 GPa. This was a promising measurement considering the expected linearity and brittle failure characteristic displayed in the stress-strain plot. These two measurements performed on the AlTi and SiO₂

microtensile beams demonstrate that microtensile system #1 was capable of differentiating between two different types of material behavior; ductile and brittle.

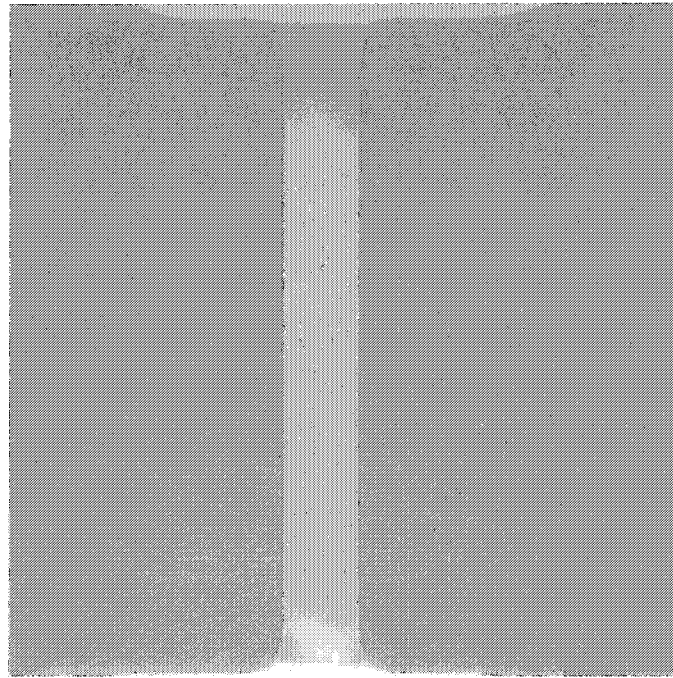


Figure 4-11: A LOM image of a cracked 1 μm thick SiO_2 microtensile beam (Mag. 75x).

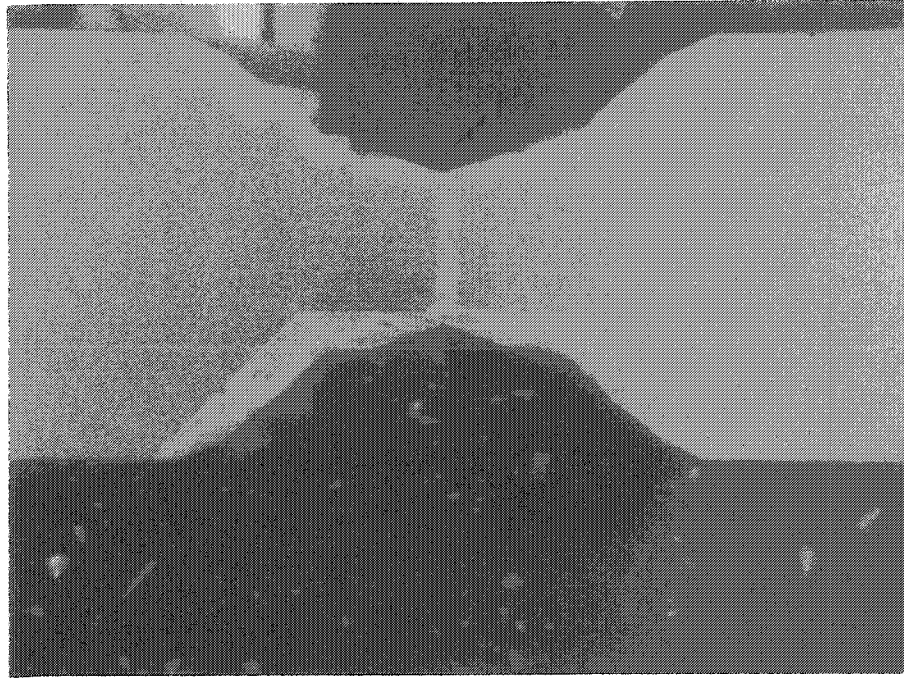


Figure 4-12: A stereomicroscope image of a SiO₂ microtensile beam during an experiment (Mag. 17x).

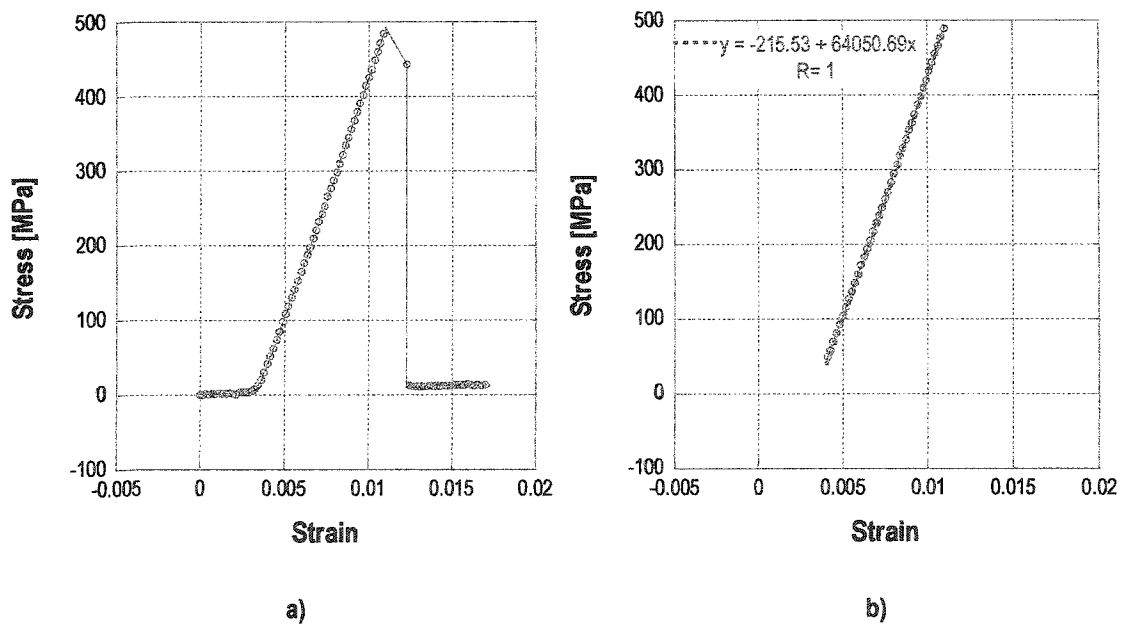


Figure 4-13: a) The stress-strain plot for a 1 μm thick SiO₂ microtensile beam tested on microtensile system #1 resulting in b) an elastic modulus of 64 GPa.

4.2.1.1.3 75 μm diameter Al_2O_3 fibers and 10 μm diameter Al and Cu wires

Because of the small diameter of the fibers and wires, it was thought to be nearly impossible to directly clamp them into the grips especially after attempting many times and not succeeding. Therefore, they were glued (using Krazy Glue[®]) to a Si frame; the whole assembly of the Si frame and wire was then clamped. Excess Si frames without microtensile beams were available from both the SiO_2 and Al fabrication processes. Once the Si frame was clamped in the grips, the side supports of the Si frame were cut using the diamond saw thereby allowing the freestanding fiber or wire to be tested. Figure 4-14 shows a schematic of the sample setup and Figure 4-15 shows a LOM image of a Cu wire. The purpose behind testing the Al_2O_3 ceramic fibers was to ascertain if system #1 was capable of measuring the reported elastic modulus of bulk materials. Additionally, these fibers would serve as microtensile standards to which other values of measured moduli could be compared. All microtensile experiments performed on thin films report a measured elastic modulus value less than the elastic modulus of bulk materials. Arguments are proposed as to whether these low measured values are a result of the sample itself or the testing system. If a standard was available, then measured values reported on thin films can be true values and not a consequence of the system. The reported modulus for the Al_2O_3 fibers was 434 GPa.

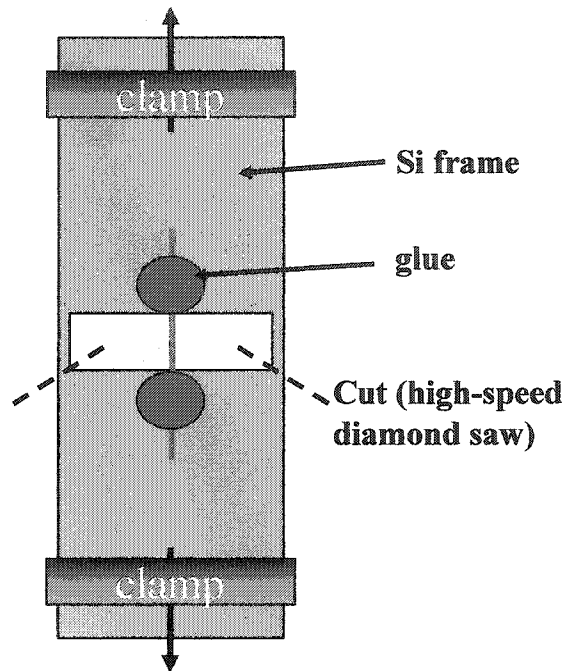


Figure 4-14: A schematic of the sample arrangement for testing 75 μm diameter Al_2O_3 fibers and 10 μm diameter Al and Cu wires.

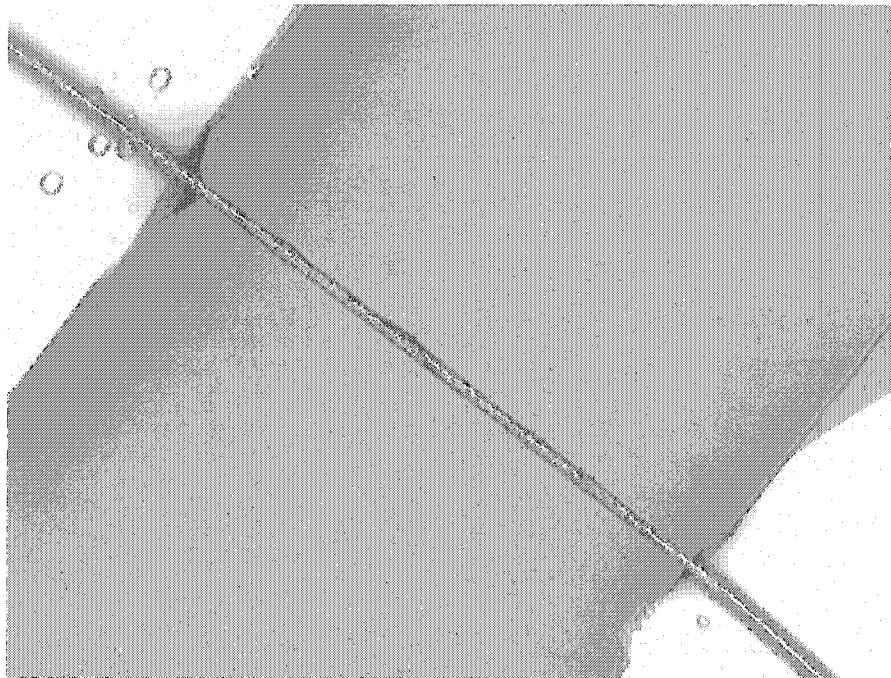


Figure 4-15: A LOM image of a 10 μm diameter Cu wire glued onto the Si frame (Mag. 100x).

The fiber was tested at a strain rate of $3.33 \times 10^{-4} \text{ s}^{-1}$. The gage length was approximately 600 μm which is the distance spanning the open section of the Si frame. The elastic modulus upon unloading was 13 GPa (Figure 4-16), which is 97% lower than the reported elastic modulus of bulk materials. One reason for this large difference in moduli was poor adhesion of the glue to the fiber. This gave rise to extra compliance in the sample thereby resulting in a very low elastic modulus. Multiple fibers were tested using this approach with each resulting in a very low elastic modulus. Some fibers were glued to pieces of plastic shim stock instead of Si frames, but the resulting elastic modulus was the same and even lower in some cases. The plastic shim stock was obviously more compliant than the Si, hence giving lower modulus values. By doing these experiments with the plastic shim stock, the adhesion problem was pinpointed to the fiber and not the Si. These experiments carried out on Al_2O_3 fibers provided a foundation for testing the 10 μm diameter Al and Cu wires which are discussed in Chapter 6 section 6.1.

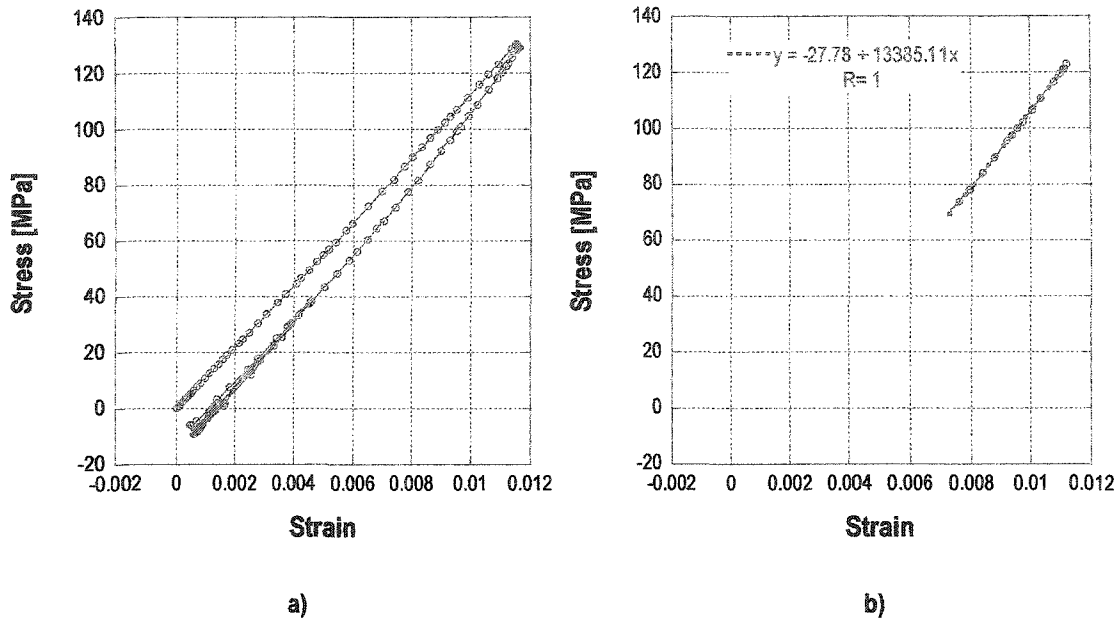


Figure 4-16: a) The stress-strain plot for a 75 μm diameter Al_2O_3 fiber tested on microtensile system #1; b) the elastic modulus upon unloading was 13 GPa.

4.2.2 Microtensile system #2: double titanium force-sensing beams

The load cell configuration for microtensile system #2 consisted of a double titanium force-sensing beam (Figure 4-17). This design change from a single spring steel beam to double titanium beams was made because of the instability of a long load train; the long load train was necessary in order for the microtensile system to accommodate a furnace (Figure 4-18). The purpose of the furnace was to provide the microtensile system with the capability of measuring stress relaxation of 1 μm thick SiO_2 microtensile beams at temperatures between 800°C and 1000°C.⁶⁸

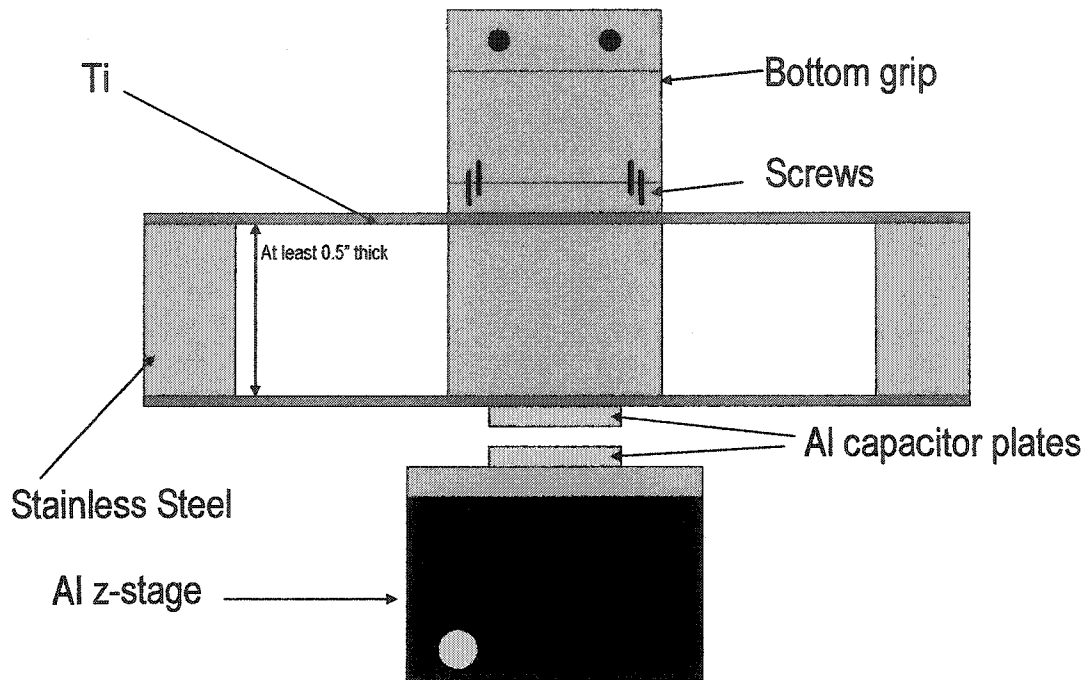


Figure 4-17: A schematic of the double titanium force-sensing beam load cell configuration for microtensile system #2.

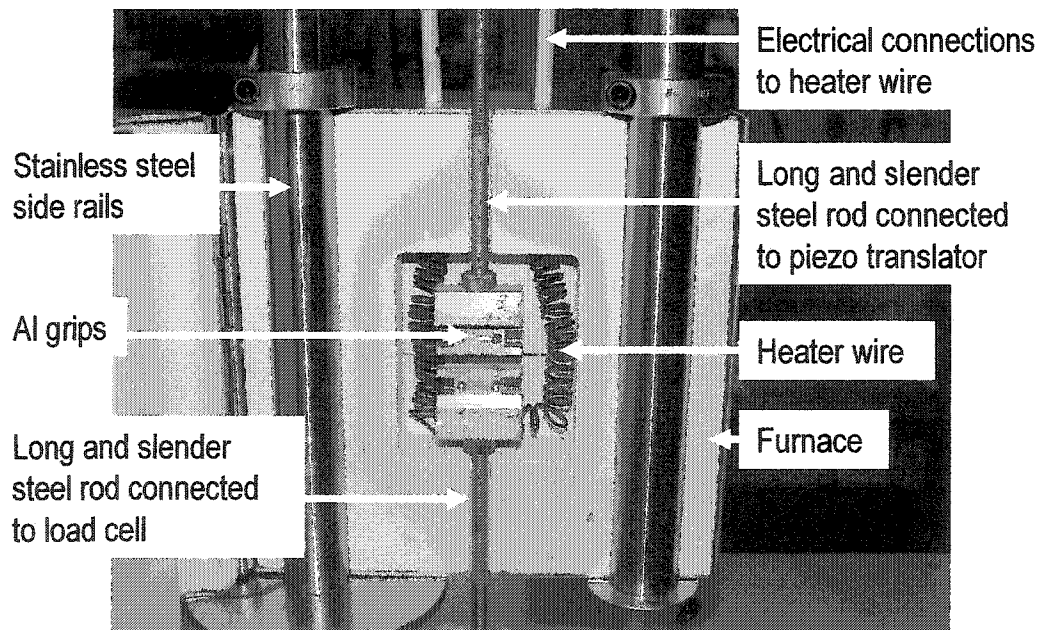


Figure 4-18: A digital image showing the addition of the furnace to microtensile system.

The long load train with the single spring steel force-sensing beam load cell configuration proved to be extremely unstable mechanically at the grips making clamping and cutting of the Si frames nearly impossible (Figure 4-19); it was also unstable with the double titanium force-sensing beam load cell. In order to speed up construction of the system for evaluation purposes, long slender steel rods were used along with Al grips and clamps. These steel rods were eventually replaced by long slender ceramic rods which were eventually replaced by long thick ceramic rods. However, because Al melts at 660°C, the Al grips and clamps were eventually replaced by ceramic grips and clamps; initially ceramic screws were used for securing the clamps to the grips, but because many broke during the tightening process, they were replaced by steel screws. For the many problems associated with the high temperature microtensile system, a decision was eventually made to design a completely dedicated system. With the design of this separate system, the double titanium force-sensing beam load cell configuration was evaluated with a short load train (similar to microtensile system #1). This arrangement proved to be very rigid mechanically at the grips allowing successful clamping and cutting of the Si frames, but it performed poorly when operating under high strain rate or 100 Hz frequency conditions.

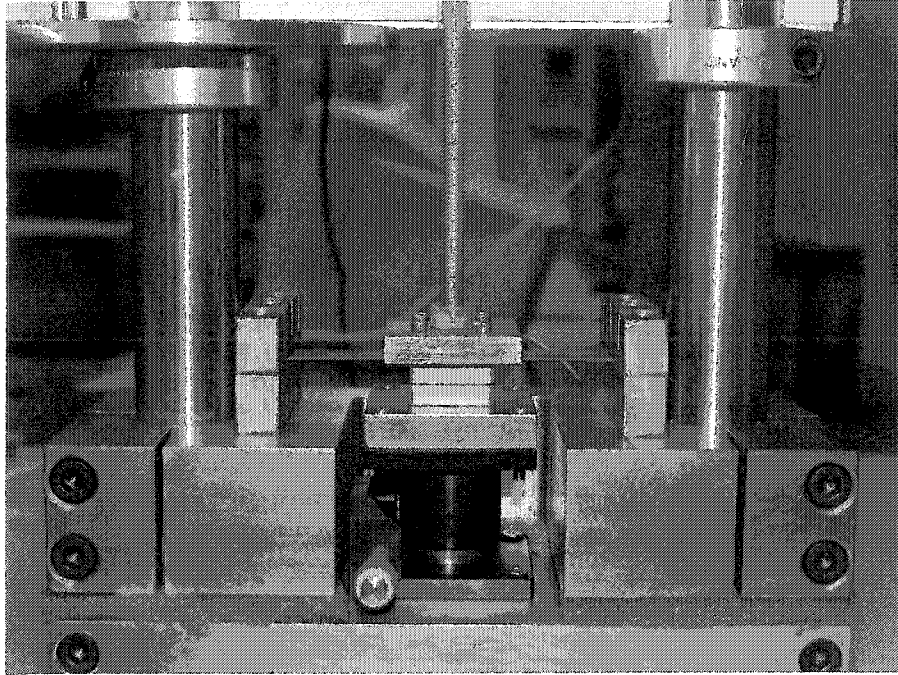


Figure 4-19: A digital image showing the single spring steel force-sensing beam load cell with a long load train (the single spring steel force-sensing beam load cell configuration was used with the long load train before the double titanium force-sensing beam was designed).

4.2.2.1 Microtensile system #2 (long load train) experiments

With the long load train, the double titanium force-sensing beam load cell was calibrated using different thicknesses of plastic shim stock; each thickness was a different weight. The weights (< 7 mN) were chosen to define the calibration curve in the load range that the microtensile beams would be tested. The calibration constant was $39.84 \text{ mN}/\mu\text{m}$ (Figure 4-20). A brief set of experiments were carried out with this system because the dedicated high temperature microtensile system was designed and constructed shortly after this setup.

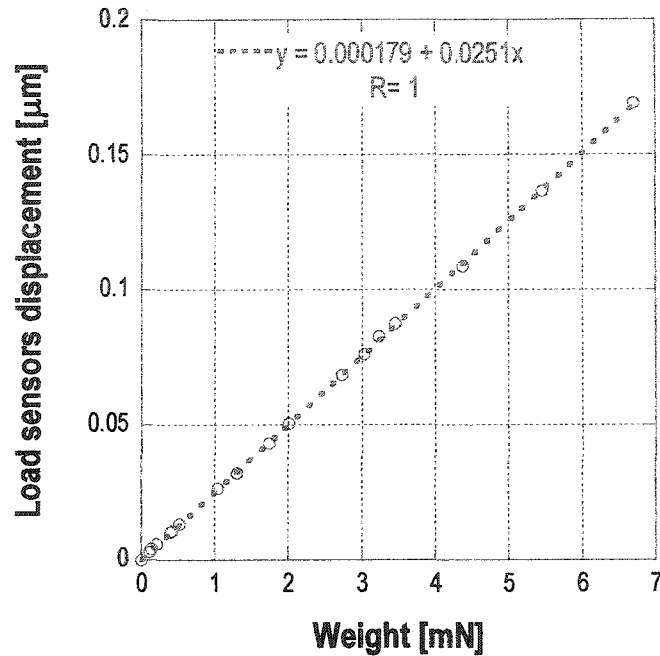


Figure 4-20: A plot of the load sensors displacement vs. weight showing the calibration curve for the double titanium force-sensing beam load cell; the calibration constant = 0.0251 $\mu\text{m}/\text{mN}$.

Some of the experiments included testing 1 μm thick SiO_2 microtensile beams. Most of these SiO_2 microtensile beams had anywhere from 25 to 50 μm of unetched Si remaining on the backside. This extra unetched Si made it possible to clamp and cut the Si frames without having the microtensile beam catastrophically fail. However, the values of stress and strain measured were those of the composite and not the SiO_2 making it difficult to fully understand the mechanical properties of the SiO_2 thin film.

4.2.2.2 Microtensile system #2 (short load train) experiments

It was necessary to characterize each microtensile system after it was built. No microtensile standard was available (and still is not today), so experiments were carried out using 75 μm diameter Al_2O_3 fibers and 25 μm diameter Ni and Cu wires. The ceramic fibers

were no longer glued considering the poor adhesion issues. Instead, it was determined they were large enough to be clamped into the grips without having to glue them to a Si frame. Also, the 25 μm diameter Ni and Cu wires were directly clamped into the grips. A piece of Al foil was wrapped around the grip and another around the clamp; the wire was clamped between the two pieces of foil. This technique allowed the foil to deform around the wire holding it secure when the clamp was tightened. Microtensile system #2 (short load train) was calibrated in the same manner as microtensile system #2 (long load train) using plastic shim stock; the calibration constant was 0.032 $\mu\text{m}/\text{mN}$, or 31.25 $\text{mN}/\mu\text{m}$ (Figure 4-21).

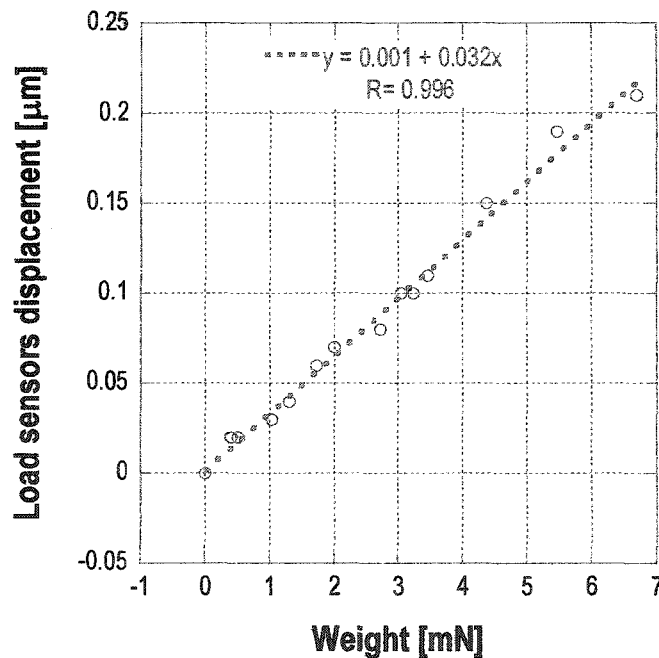


Figure 4-21: A plot of the load sensors displacement vs. weight showing the calibration curve for the double titanium force-sensing beam load cell; the calibration constant = 0.032 $\mu\text{m}/\text{mN}$.

4.2.2.2.1 75 μm diameter Al_2O_3 fiber

This fiber was directly clamped into the grips. The elastic modulus reported by the manufacturer was 434 GPa. Experiments were done using a strain rate of $7.5 \times 10^{-7} \text{ s}^{-1}$ and a strain amplitude of 0.026%. The cross-sectional area was calculated using the area of a circle with a diameter of 75 μm . The stress, in $[\text{N/m}^2] = [\text{Pa}]$, was determined by dividing the measured load by the cross-sectional area. The gage length was taken as the distance between the two clamp edges which was 2.8 mm *{the grips for this experiment were not the same design as those outlined in section 3.5; the clamps for the top and bottom grips were not offset from the lower and upper surfaces, respectively. Instead, the clamps were flush with the lower and upper surfaces of the top and bottom grip}*. And the strain was determined by dividing the grip displacement by the gage length. The measured elastic modulus was approximately 434 (Figure 4-22), which was in excellent agreement with the reported elastic modulus. Keep in mind that this was one measurement performed on one fiber, so there are no statistics.

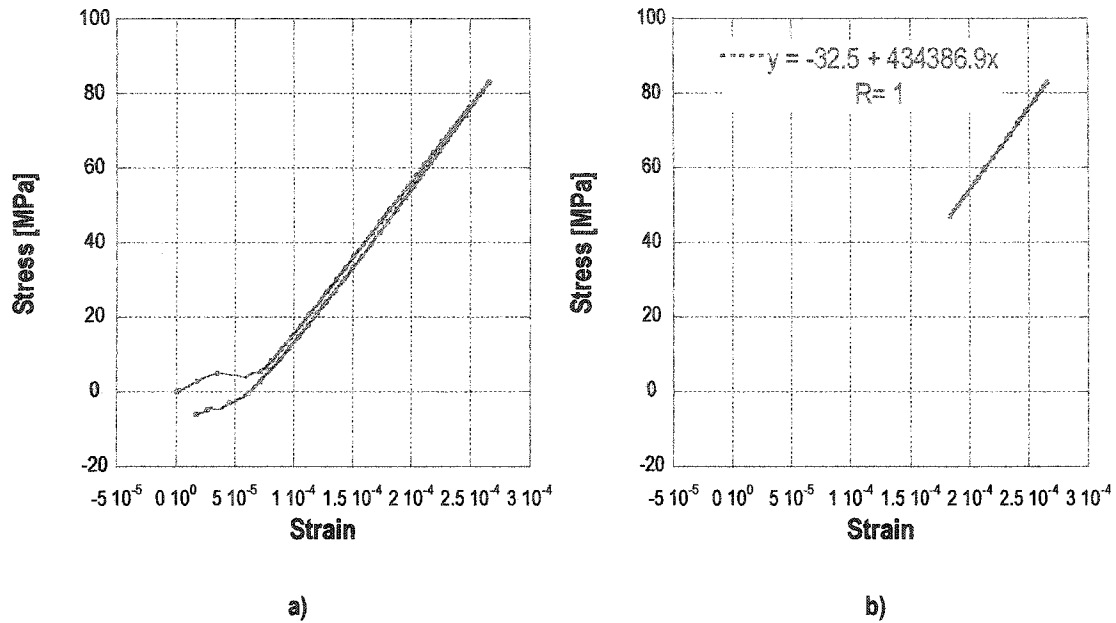


Figure 4-22: The stress-strain plot for an Al_2O_3 ceramic fiber directly clamped into the grips; b) the measured elastic modulus upon unloading was approximately 434 GPa.

4.2.2.2.2 25 μm diameter Cu wire

After testing the ceramic fiber, a 25 μm diameter Cu wire was tested. The goal of this experiment was to obtain the elastic modulus for bulk polycrystalline Cu, 110 GPa.⁶⁹ Furthermore, testing these Cu wires could provide another standard for the microtensile system. The piezo translator was retracted at a constant rate of 0.595 $\mu\text{m/s}$ (or a strain rate of $4.96 \times 10^{-5} \text{ s}^{-1}$) until the load cell reached about 20 mN; the direction of the piezo translator was manually changed, and it was then extended at that same constant rate (Figure 4-23a). Because the piezo translator is mounted upside-down, as it retracts the Cu wire is strained. On the unloading part of the cycle, the piezo translator was stopped when the load reached approximately 0 mN. Because it was manually stopped, the piezo displacement shows different beginning and ending values. Using a gage length of 2.8 mm, the measured elastic modulus upon unloading

was 24 GPa. However, considering the high linearity of the stress-strain plot, this gage length of 2.8 mm appears to be incorrect. Using a gage length of 12 mm, the measured elastic modulus upon unloading was approximately 103 GPa (Figure 4-23b).

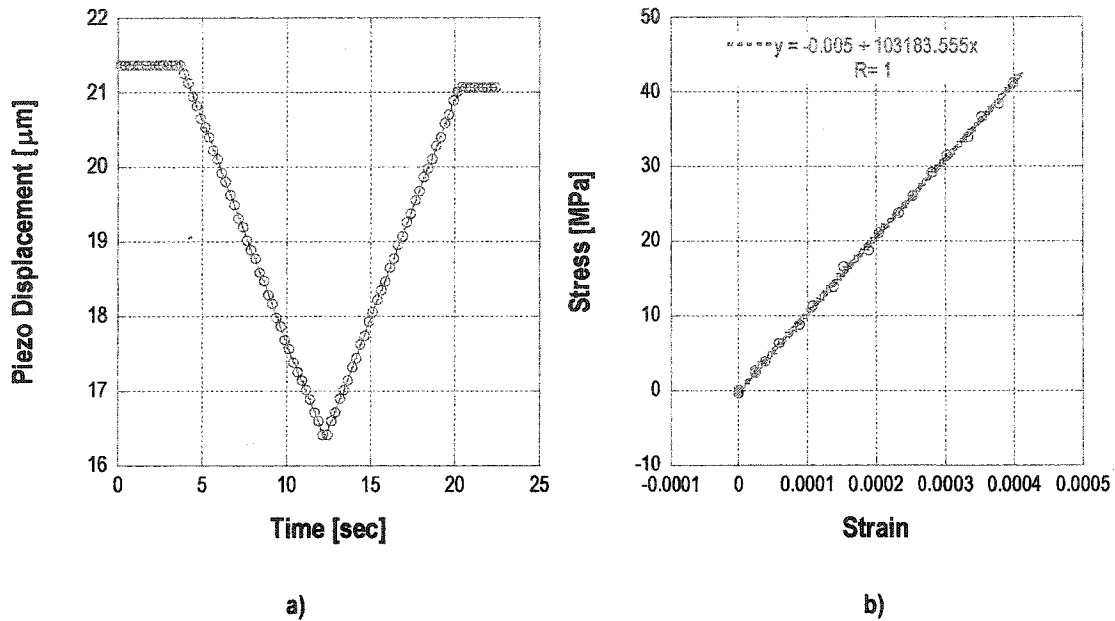


Figure 4-23: a) A plot of piezo displacement vs. time, and b) the stress-strain plot showing an elastic modulus upon unloading of approximately 103 GPa.

A second experiment was performed on the same Cu wire (Figure 4-24). The piezo translator was retracted at a constant strain rate $4.96 \times 10^{-5} \text{ s}^{-1}$, until the load cell reached about 70 mN; the direction of the piezo translator was manually changed and was extended until the load dropped to 20 mN. Once the load reached about 20 mN, the piezo translator direction again was changed. It was retracted until it reached a limit of 0 μm ; the load was 114 mN. Once here, the direction again was changed finally unloading the wire to 80 mN. Upon unloading from 114 mN to 80 mN, the elastic modulus was approximately 110 GPa (Figure 4-25) which is in excellent agreement with the reported elastic modulus for bulk polycrystalline Cu. Note the different levels of stress between the first and second experiments. The first experiment has a

maximum around 40 MPa while the second has a maximum around 240 MPa. The lower elastic modulus for the first experiment may be a result of the smaller stress range. Within 0.05% strain, the measured elastic modulus upon unloading is lower than the modulus of the bulk material because bends, or kinks, in the wire surface are being straightened or worked out.

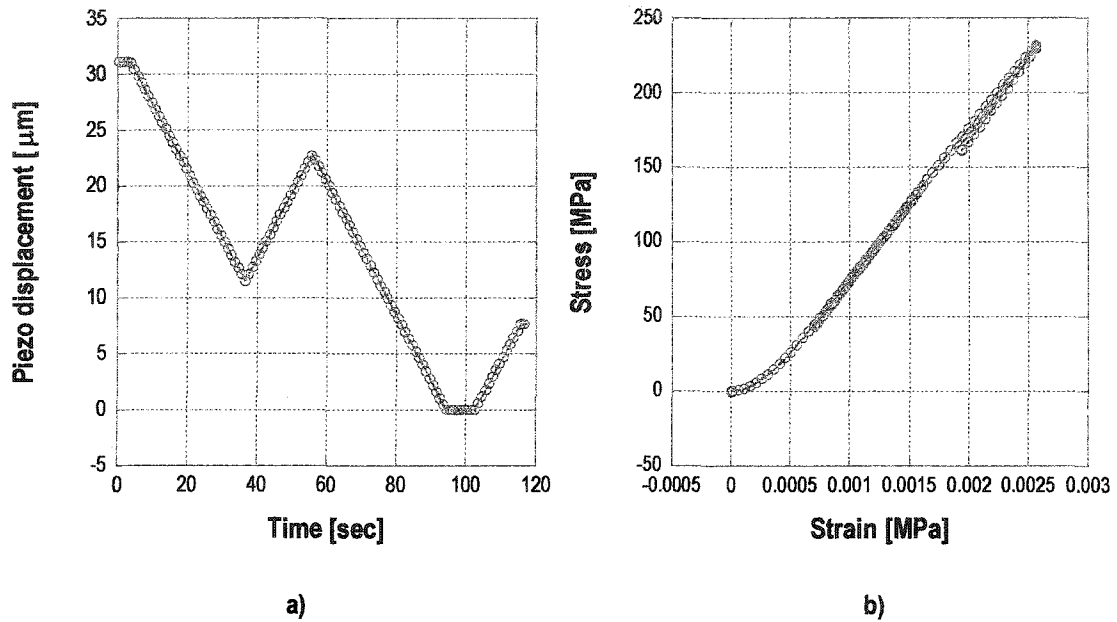


Figure 4-24: a) A plot of piezo displacement vs. time, and b) the stress-strain plot from the second experiment carried out on the 25 μm diameter Cu wire.

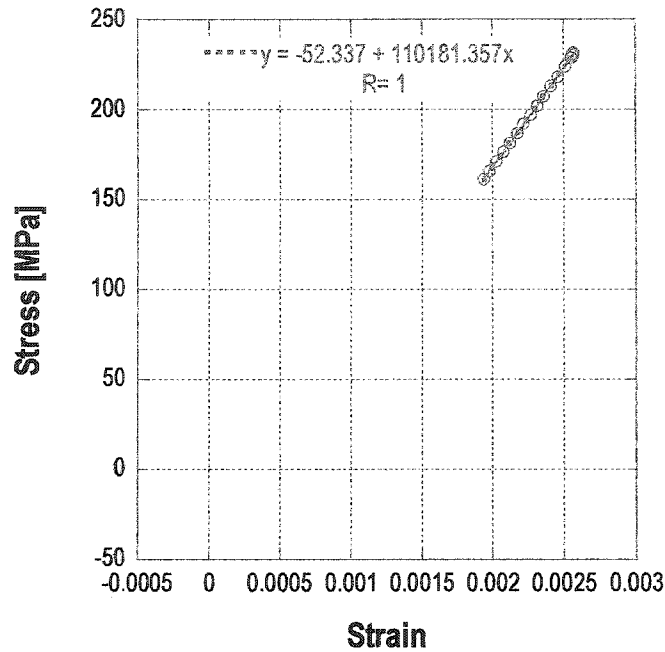


Figure 4-25: The stress-strain plot for the second experiment on the Cu wire showing an elastic modulus upon unloading of approximately 110 GPa.

4.2.2.2.3 25 μm diameter Ni wire

After testing the Cu wire, a Ni wire was then tested to see if the reported elastic modulus (207 GPa) of Ni could be obtained (Figure 4-26). The gage length of the Ni wire was taken to be 2.8 mm which resulted in an elastic modulus of 39 GPa. However, using a gage length of 14 mm greatly increases the value of the elastic modulus from 39 GPa to 194 GPa. With a displacement rate of 0.296 $\mu\text{m/s}$ and a gage length of 14 mm (14,000 μm), the strain rate was $2.11 \times 10^{-5} \text{ s}^{-1}$.

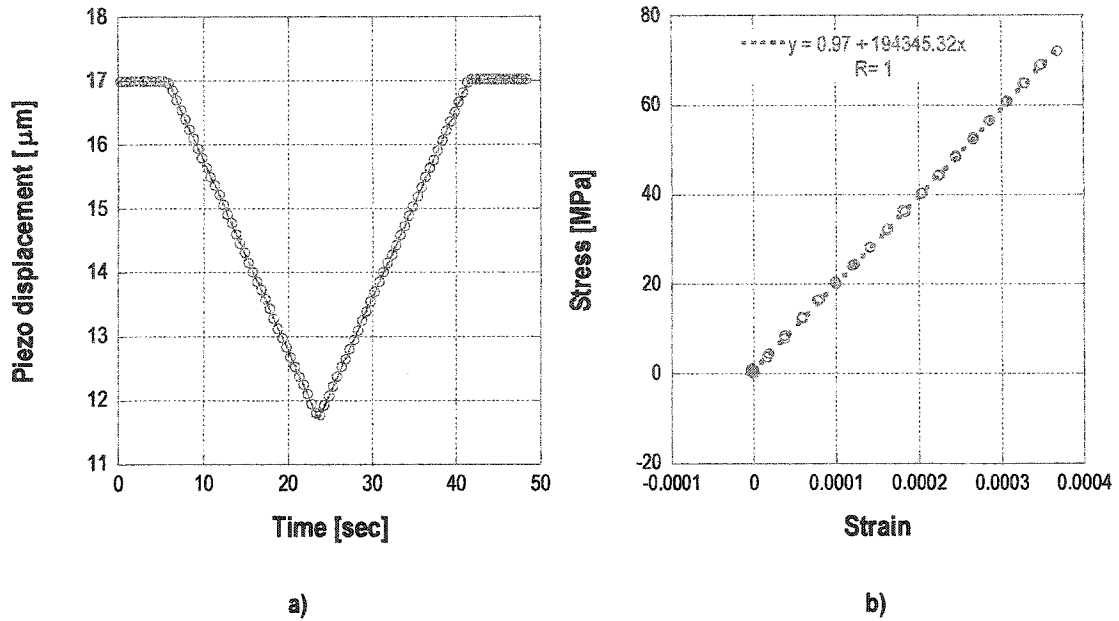


Figure 4-26: a) A plot of piezo displacement vs. time, and b) the stress-strain plot showing an elastic modulus upon unloading of approximately 194 GPa.

A second experiment was performed on the same Ni wire (Figure 4-27). An initial loading and unloading of the wire was followed by three more loading and unloading events until the wire failed. The elastic modulus of 197 GPa was measured from the second unloading curve. The strain rate was $4.20 \times 10^{-5} \text{ s}^{-1}$ for all loading and unloading events. Also, note the excellent agreement among the loading and unloading curves in the stress-strain plot demonstrating the elastic behavior of the Ni wire.

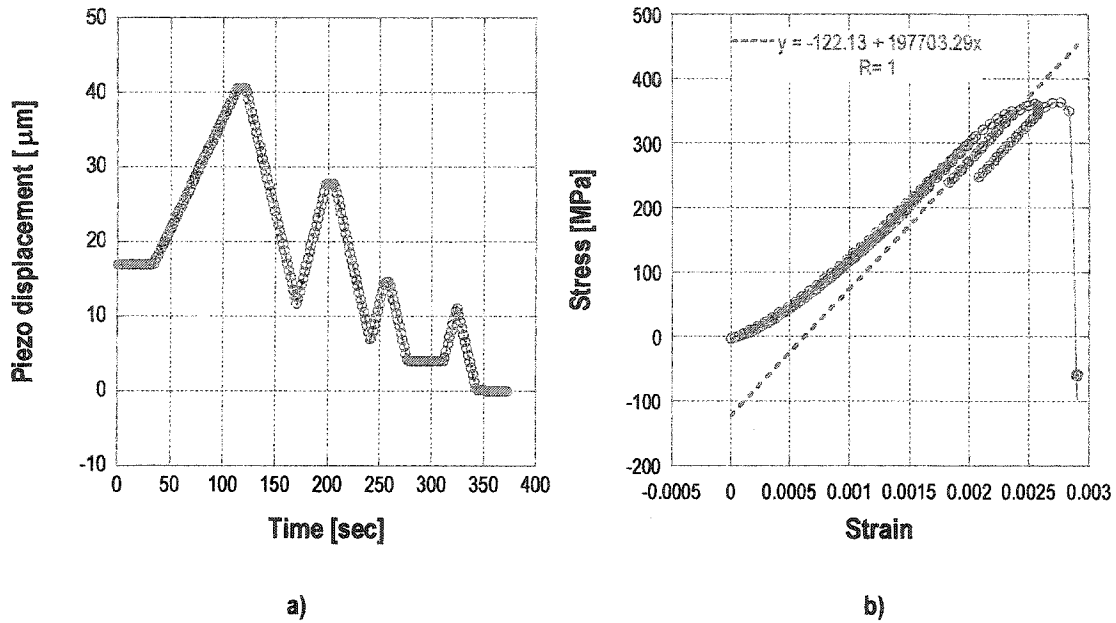


Figure 4-27: a) A plot of piezo displacement vs. time, and b) the stress-strain plot showing an elastic modulus of approximately 197 GPa.

There are unanswered questions as to why the gage length must be different (i.e. 2.8 mm, or 12 mm, or 14 mm) in order to get the reported elastic moduli of the Al_2O_3 fiber, Cu wire, and Ni wire, respectively. The gage length is in question because of the inconsistency of the clamping process (it could range anywhere from 2.8 mm, which is the minimum distance between the clamp edges to 18 mm, or the maximum distance between the clamp edges). However, the stress-strain plots for the fiber experiment and all four wire experiments show a highly linear elastic behavior until failure (which was only observed in the 2nd Ni wire experiment). It was suggested that anelasticity may be a possible reason for the low values of the modulus, but it is unlikely because of the large dimensions of the wires. Excluding the varying gage length issue, the wires proved to be good experiments for validating the effectiveness of microtensile system #2 for testing micro-sized samples (i.e. thin film microtensile beams).

4.2.2.2.4 Thick Si microtensile beam

This experiment is similar to the fiber and wire experiments except this sample has a well-defined gage length. The purpose of this experiment was to compare the measured elastic modulus to the reported value of the elastic modulus for the bulk material. This sample was also tested on microtensile system #3 and microtensile system #4. The thick microtensile Si beam (Figure 4-28) was approximately 125 μm wide, 600 μm long, and 75 μm thick; the strain rate and strain amplitude were $9.88 \times 10^{-4} \text{ s}^{-1}$ and approximately 0.06%, respectively. A measured elastic modulus of 156 GPa (Figure 4-29) compared satisfactorily to the expected elastic modulus of 168 GPa for $\langle 100 \rangle$ Si.⁶⁹ The slope change in the loading curve at 30 MPa is most likely caused by the Si frame sliding in the grips. The next step was to characterize the time response of microtensile system #2 when performing high strain rate (10^{-1} s^{-1}) and 100 Hz frequency (for fatigue) experiments. Al microtensile beams were tested following this thick Si microtensile beam experiment.



Figure 4-28: A stereomicroscope image showing the thick Si microtensile beam during an experiment (Mag. 22x).

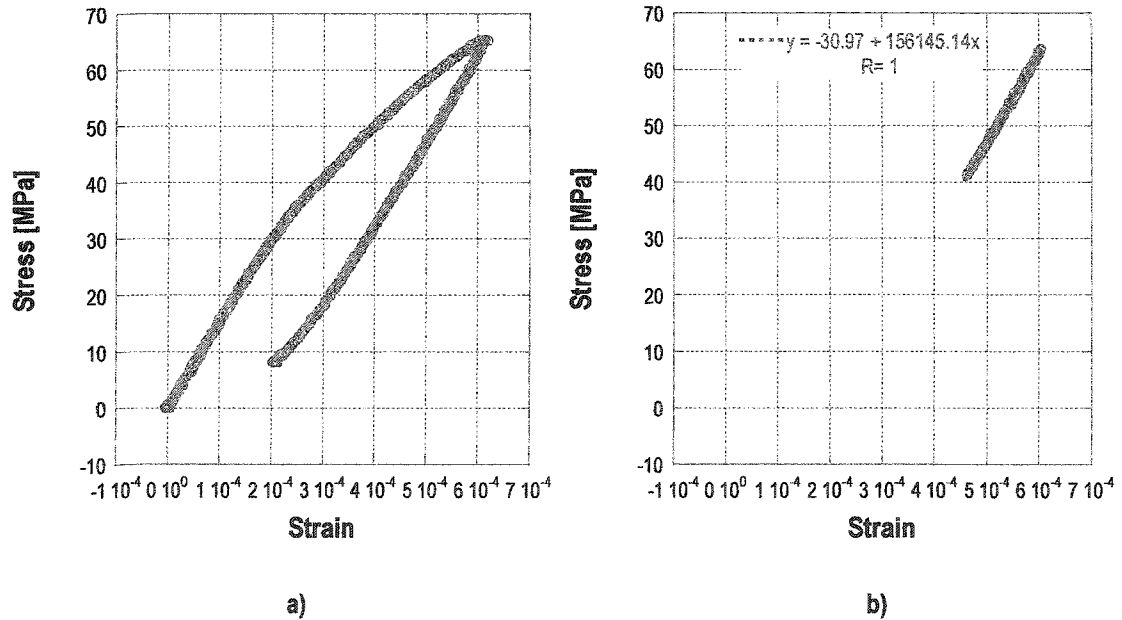


Figure 4-29: a) The stress-strain plot for a thick Si microtensile beam showing b) an elastic modulus upon unloading of approximately 156 GPa.

4.2.2.2.5 High strain rate and frequency experiments

The first 1 μm thick Al microtensile beams fabricated at Lehigh University were tested using the double titanium force-sensing beam load cell. Even though the quality of the microtensile beams may not be great, these experiments provided a starting point for testing thin films on the microtensile system. Furthermore, these samples were not well characterized. Figure 4-30, albeit a little fuzzy, shows the Al microtensile beam (after it was strained to failure) with the side supports of the Si frame missing following the cutting process. The squares above and below the microtensile beam were originally designed to allow the capability for visual strain measurements. Images would be captured during straining and then fed into a program that would calculate the strain based on the position of the squares; this technique is called digital image correlation. A gage length of 500 μm was used.

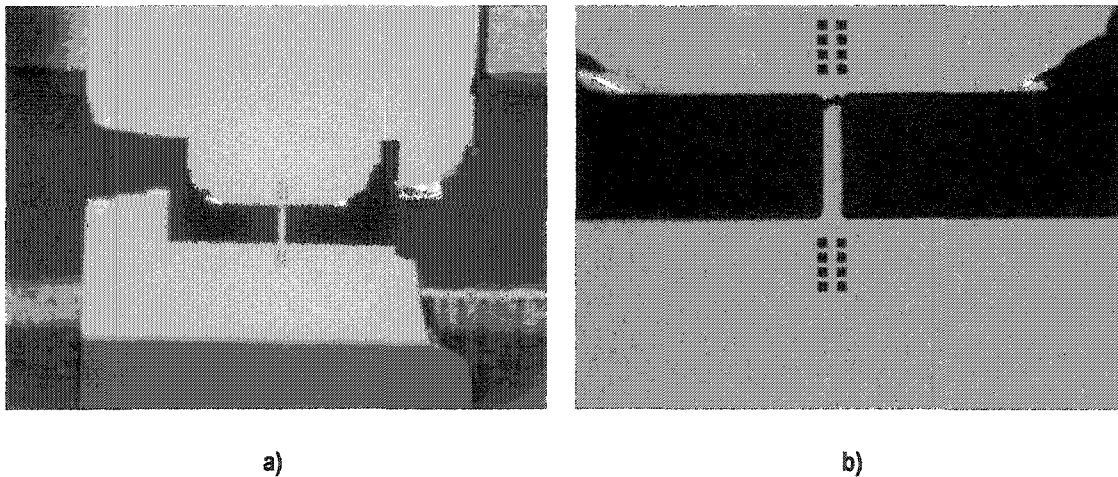


Figure 4-30: a) A stereomicroscope image of an Al microtensile beam clamped into the grips; note the missing side supports (Mag. 8x), and b) a close-up showing the fractured Al microtensile beam (Mag. 22x).

Tightening the clamps created a net tensile force of 60 mN on the Si frame seen as by the sharp jump in load (the red line) in Figure 4-31a. When the side supports of the Si frame were cut (Figure 4-31b), some of this 60 mN tensile force was transferred to the microtensile beam

causing it to be involuntarily strained. After cutting the right side support, the left side support was still intact although a large crack was running through it. The piezo was then retracted 1 μm around 310 seconds (Figure 4-31b). After realizing the two halves of the side support were still touching one other, a second cutting procedure was performed to completely remove the cracked left side support. Once the Si side supports were removed, the piezo translator was then extended to reduce the tensile force applied to the microtensile beam. However, the piezo translator was only extended 2 μm because the load signal began to plateau, hence completely reducing the large tensile force remaining on the microtensile beam. The piezo translator was then retracted and the microtensile beam was pulled to failure at a constant strain rate of $4.00 \times 10^{-5} \text{ s}^{-1}$. Between 30 MPa and 90 MPa, the microtensile beam is elastic (Figure 4-32). Work hardening begins around 90 MPa. At about 1% strain, a crack had initiated and was propagating across the width of the microtensile beam, as revealed by the decreasing stress with increasing strain. After failure, the displacement rate was increased. From the stress-strain plot on this first Al microtensile beam, both the microtensile system and microtensile beams were performing and behaving in a similar manner to the microtensile system and microtensile beams produced at Stanford University.

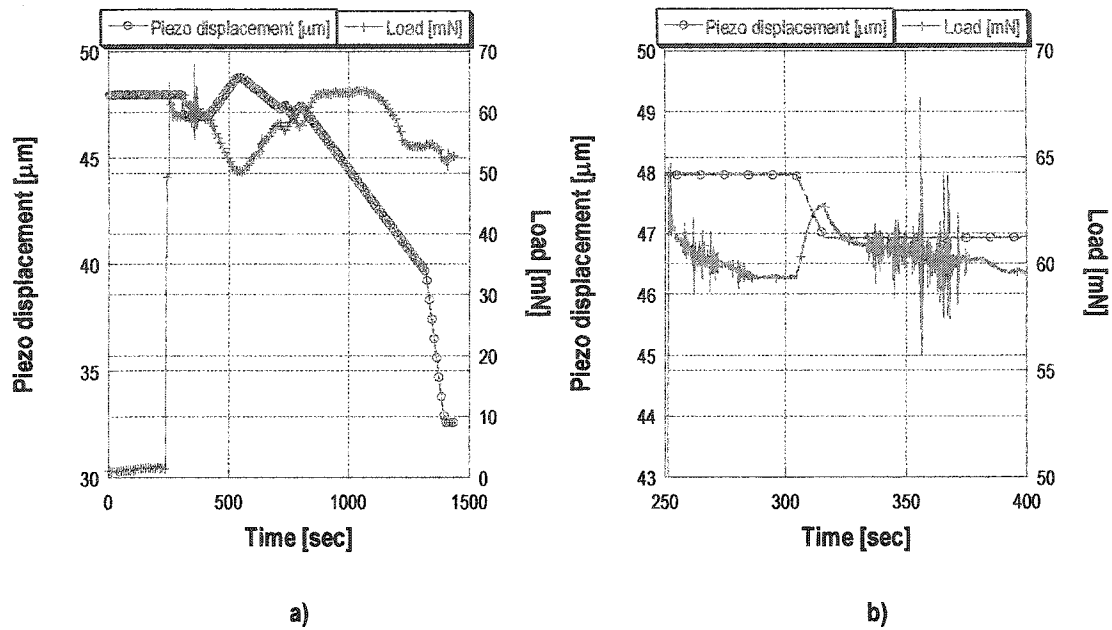


Figure 4-31: a) A plot of piezo displacement and load vs. time, and b) a close-up view showing the amount of vibration caused by cutting the side supports of the Si frame when using the diamond saw blade.

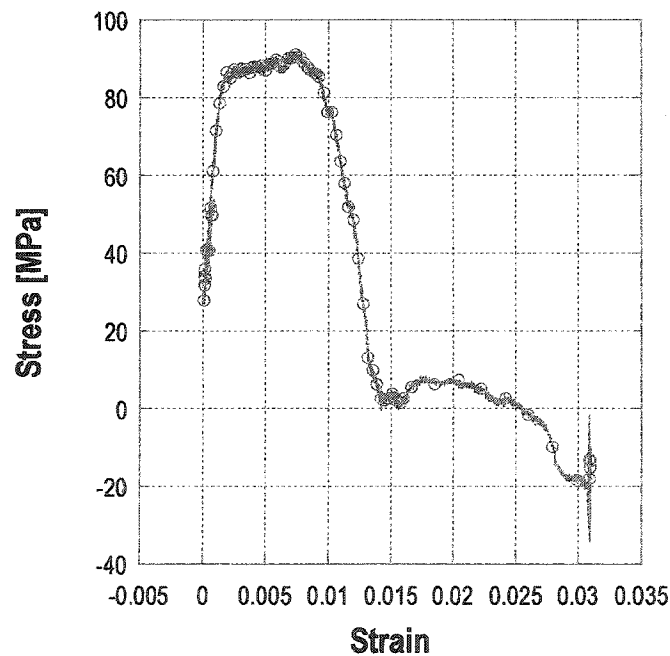


Figure 4-32: The stress-strain plot for the 1st Al microtensile beam fabricated at Lehigh University tested using microtensile system #2.

A 2nd Al microtensile beam was tested to evaluate the system and sample for a stress relaxation response. The strain rate was $1.0 \times 10^{-2} \text{ s}^{-1}$ and the sample was pulled to a maximum strain of approximately 5%. The load offset of + 30 mN is a result of the position of the capacitor plate sensors (Figure 4-33a); at 30 mN, the stress on the microtensile beam is 0 MPa (Figure 4-34). At a strain amplitude of 3%, the piezo displacement was held constant for approximately 10 seconds. The sample was observed to undergo a stress relaxation of around 50% (from 300 MPa to 150 MPa) within 10 seconds (Figure 4-33b). The stress relaxation is a function of both the stored elastic strain and the plastic strain energy. Also, the sample had not relaxed to a steady-state after 10 seconds, but it was unloaded nonetheless. After an elapsed time of around 20 seconds, the microtensile beam was reloaded and unloaded without pausing between the two events. The elastic modulus upon the 1st unloading was approximately 34.6 GPa while the 2nd unloading was approximately 36.5 GPa. The sample was then removed from the system.

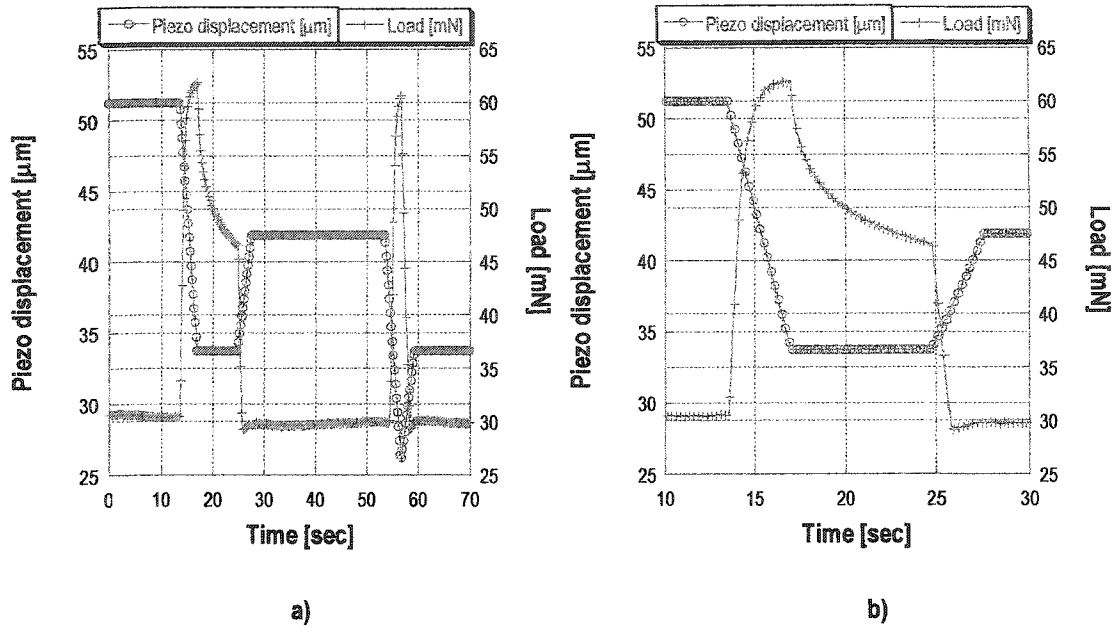


Figure 4-33: a) A plot of piezo displacement and load vs. time showing stress relaxation occurring in the Al microtensile beam followed by a second loading and unloading event, and b) a magnified view of the stress relaxation behavior.

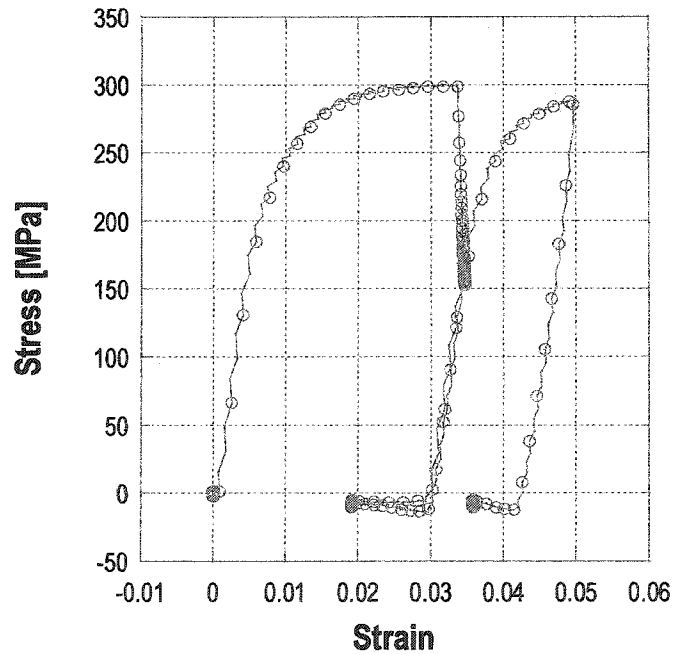


Figure 4-34: The stress-strain plot for a 2nd Al microtensile beam showing the stress relaxation taking place at 3% strain followed by additional straining from 3% to 5%. Note how well the 2nd reloading curve follows the 1st unloading curve.

The next experiment was to investigate the strain rate sensitivity of the Al microtensile beams; as the strain rate increases the measured elastic modulus was expected to increase. This experiment would provide the foundation for understanding issues associated with high strain rate testing for the double titanium beam load cell design. A 3rd Al microtensile beam was tested using a triangular waveform at the strain rates and strain amplitudes outlined in Table 4-1. The triangle waveform was input to the piezo translator using the manual “trigger” feature on the arbitrary waveform generator. Except for the highest strain rate, one triangular cycle for the remaining strain rates was performed on the same microtensile beam in a sequential order from lowest to highest; eight cycles were performed at the highest strain rate.

Table 4-1: Displacement rates, strain rates, and strain amplitudes for the strain rate sensitivity experiment performed on the 3rd Al microtensile beam.

Displacement rate [$\mu\text{m/s}$]	Strain rate [s^{-1}]	Strain amplitude [%]
0.005	1.0×10^{-5}	≈ 0.35
0.050	1.0×10^{-4}	≈ 0.35
0.500	1.0×10^{-3}	≈ 0.35
5.000	1.0×10^{-2}	≈ 0.35
50.000	1.0×10^{-1}	≈ 0.35

After cutting the side supports of the Si frame, the microtensile beam was buckled; the microtensile beam cannot support compressive stresses thus it buckles. The first triangular cycle on the microtensile beam shows that the beam began loading at around 0.5% strain. This means that the piezo translator was retracted about 3 μm before the microtensile beam was unbuckled and pulled taut. Taking the difference between the maximum strain and where the

microtensile beam starts to take on stress is the total strain in the sample; the strain amplitude was approximately 0.35% for all strain rates. However, the piezo displacement was set the same for all strain rates (i.e. the piezo translator moved the same distance of approximately 5 μm for each strain rate); in spite of this, the strain on the sample is different for the first cycle which means that some effect (i.e. removing kinks in the film) is taking place only during the initial straining and not thereafter. For the first cycle, the microtensile beam started to take on load at around 0.5% and in the remaining cycles, this happens around 0.65%. Looking at the stress-strain plot for the first cycle in Figure 4-35b, this difference of 0.15% between the loading and unloading is clearly illustrated. Also, the loading event begins at 0 MPa and the unloading event ends at negative 20 MPa. Negative stress values do not mean that the microtensile beam is in compression; instead, there was temperature drift of the stainless steel side rails causing the plates of the capacitance sensors to move closer together. At negative 20 MPa, the microtensile beam is actually at zero stress. This drift is clearly illustrated by observing the base line load signal in Figure 4-35a. At this lowest strain rate, the temperature drift appears to be the most severe because of the long time during loading and unloading. In Figure 4-35a, Figure 4-36a, Figure 4-37a, Figure 4-38a, and Figure 4-39a, the piezo displacement and load vs. time are shown. For strain rates of 1.0×10^{-5} and 1.0×10^{-4} , both the load and piezo displacement reach their maximums at approximately the same time. For strain rates of 1.0×10^{-3} , 1.0×10^{-2} , and 1.0×10^{-1} , the load cell begins to respond after the piezo translator begins to move. This indicates a time delay between the piezo translator and the load cell. Also, the hysteresis loop between the loading and unloading is decreasing with increasing strain rate up to a strain rate of 1.0×10^{-3} and then begins to increase again with the highest strain rates. For strain rates of 1.0×10^{-5} and 1.0×10^{-4} the loading curve is located above the unloading curve. However, for

strain rates of 1.0×10^{-3} , 1.0×10^{-2} , and 1.0×10^{-1} the loading and unloading curves have switched positions with the unloading curve now located above the loading curve. This is grossly exaggerated for the highest strain rate.

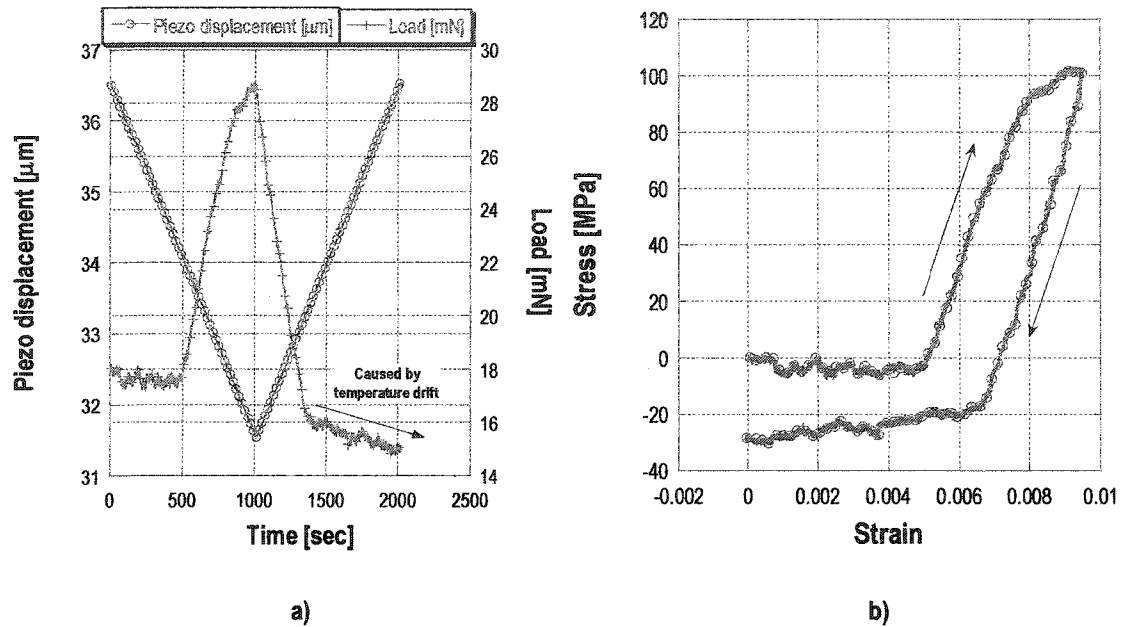


Figure 4-35: a) A plot of piezo displacement and load vs. time, and b) the stress-strain plot for the 3rd Al microtensile beam tested at a strain rate of $1.0 \times 10^{-5} \text{ s}^{-1}$.

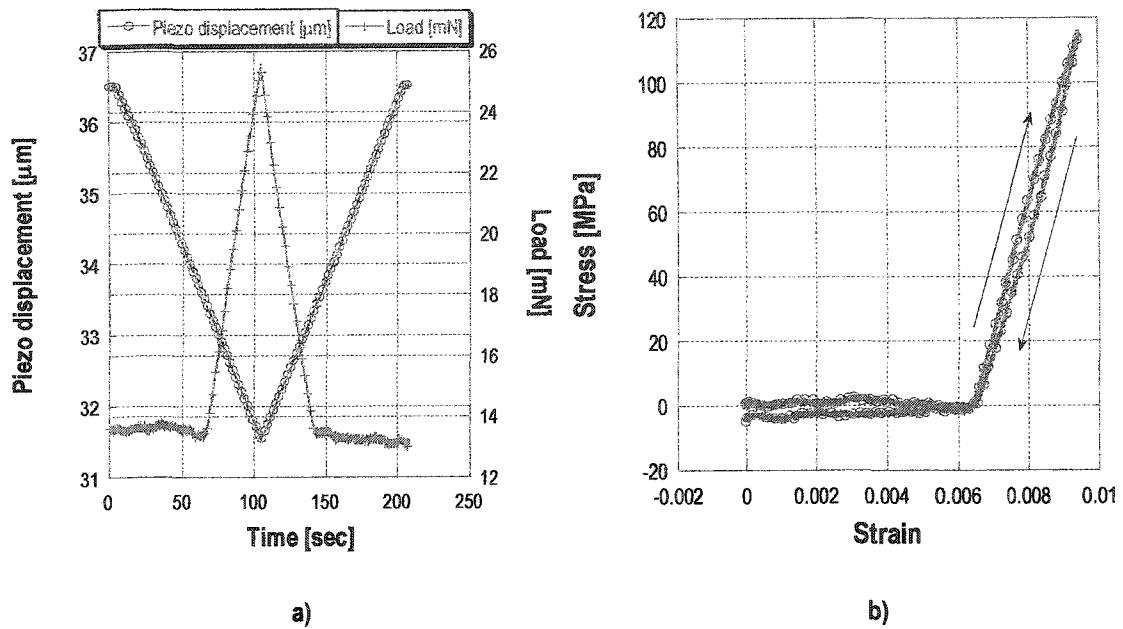


Figure 4-36: a) A plot of piezo displacement and load vs. time, and b) the stress-strain plot for the 3rd Al microtensile beam tested at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$.

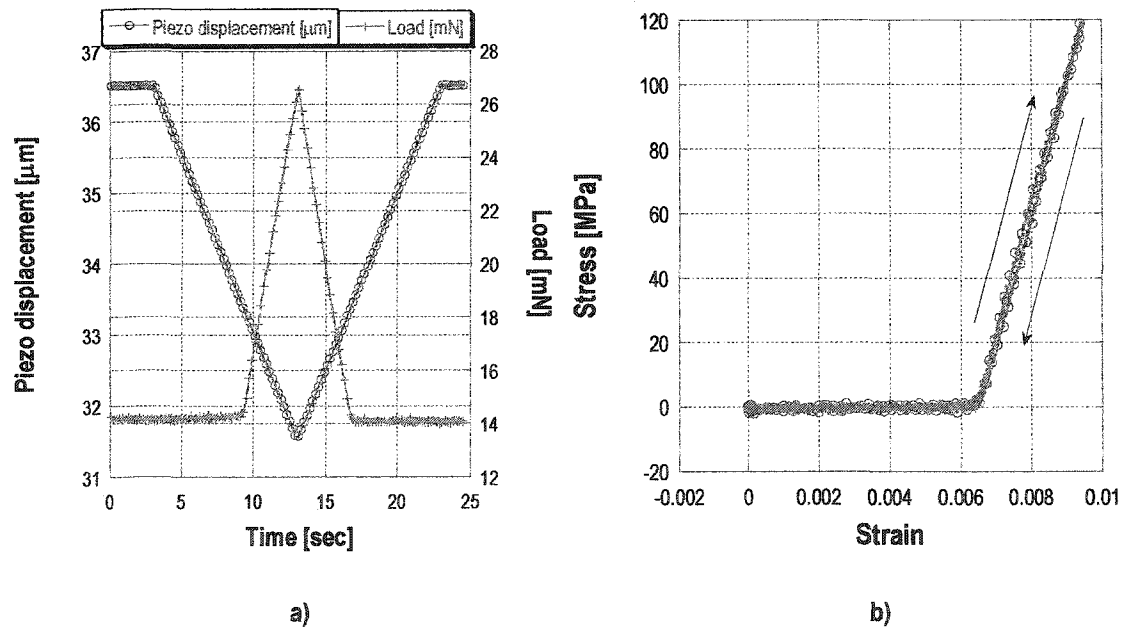


Figure 4-37: a) A plot of piezo displacement and load vs. time, and b) the stress-strain plot for the 3rd Al microtensile beam tested at a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$.

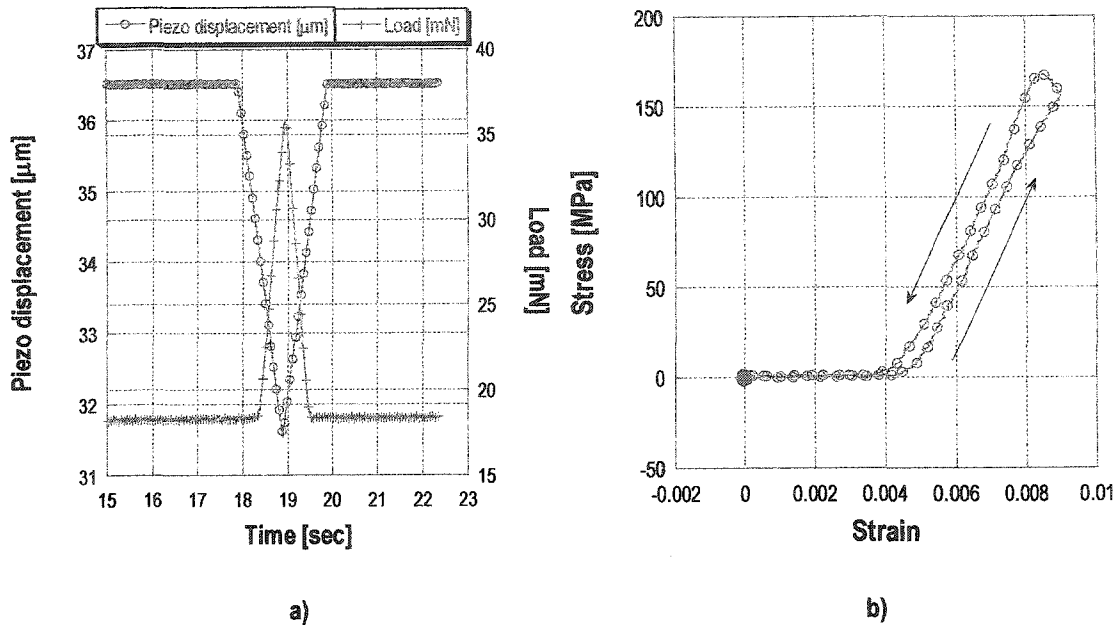


Figure 4-38: a) A plot of piezo displacement and load vs. time, and b) the stress-strain plot for the 3rd Al microtensile beam tested at a strain rate of $1.0 \times 10^{-2} \text{ s}^{-1}$.

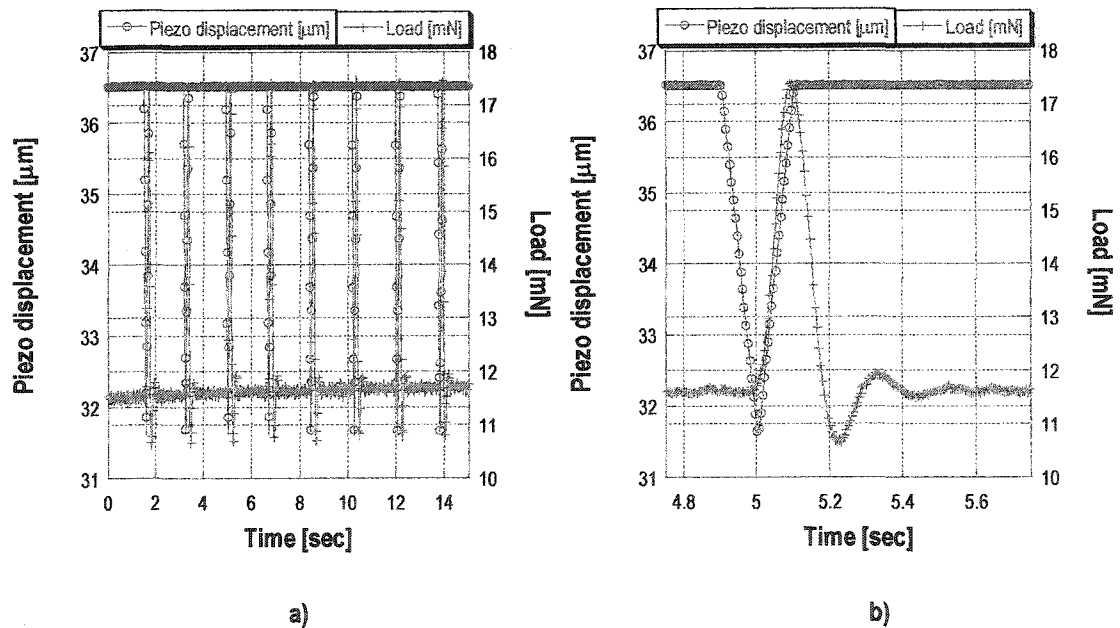


Figure 4-39: a) A plot of piezo displacement and load vs. time for 8 cycles at a strain rate of $1.0 \times 10^{-1} \text{ s}^{-1}$, and b) a magnified view of the 3rd cycle showing the time delay between the piezo displacement and the load cell.

For the highest strain rate, there is a severe breakdown in the microtensile system resulting in the plot shown in Figure 4-40. This plot illustrates that the load cell is not responding to the straining of the microtensile beam until the piezo translator is on its return trip to its starting position. Furthermore, take note of both the maximum stress attained for different rates and the measured modulus. The maximum stress increases by 15 MPa between the two slowest strain rates (1.0×10^{-5} and 1.0×10^{-4}). Between the next two (1.0×10^{-4} and 1.0×10^{-3}) the maximum stress increases by 5 MPa, and by 40 to 50 MPa for the next two rates (1.0×10^{-3} and 1.0×10^{-2}). This variation in maximum stress was not at all expected based on the constant piezo displacement for all strain rates.

The elastic modulus (Table 4-2) for strain rates of 1.0×10^{-5} to 1.0×10^{-4} increases with increasing strain rate as expected; also, the unloading modulus increases slightly. The modulus upon loading was measured using a least squares fit between 20 and 60 MPa; the unloading modulus was measured using a least squares fit through the top 40 MPa of the curve. However, for the strain rate of 1.0×10^{-3} , the unloading modulus drops dramatically. At the peak stress and strain for this strain rate, the loading and unloading curves are beginning to change positions as indicated by a loop in the stress-strain plot shown in Figure 4-41. If the microtensile beams were anelastic, as the strain rate increased the modulus was expected to increase; however, the microtensile beams were not behaving as expected. Because the stress-strain plots for the Al microtensile beams tested at low strain rates were similar to those previously tested at Stanford University, it was thought that the present non-ideal results were caused by the microtensile system and not the samples.

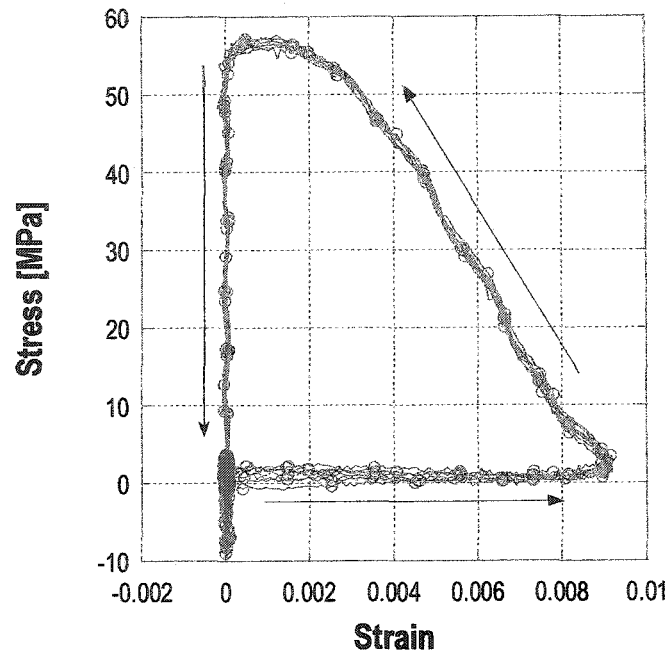


Figure 4-40: The stress-strain plot for the 3rd Al microtensile beam tested at a strain rate of $1.0 \times 10^{-1} \text{ s}^{-1}$ (all 8 cycles are shown). Note the excellent consistency among cycles.

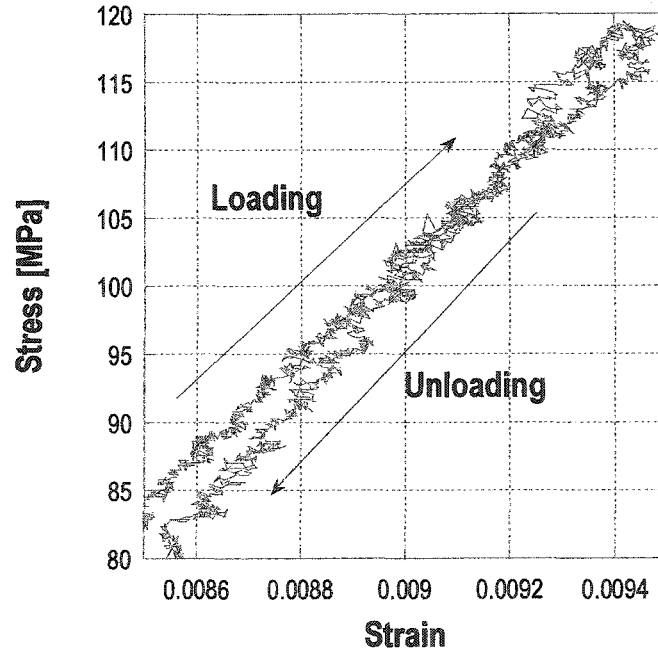


Figure 4-41: A loop in the stress-strain plot for a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ indicating the onset of a breakdown in the load cell response; this is a magnified view of Figure 4-37b.

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Table 4-2: The strain rates and measured elastic moduli upon loading and unloading for the strain rate sensitivity experiment on the 3rd Al microtensile beam tested.

Strain rate [s ⁻¹]	Measured elastic modulus [GPa]	
	<i>Loading</i>	<i>Unloading</i>
1.0 x10 ⁻⁵	38.5	45.6
1.0 x10 ⁻⁴	41.3	47.3
1.0 x10 ⁻³	39.5	40.5
1.0 x10 ⁻²	35.5	45.2
1.0 x10 ⁻¹	N/A	N/A

4.2.2.3 Damping of the double titanium force-sensing beam load cell

This time delay presented a major problem for testing microtensile beams at high strain rates (or under fatigue conditions) using this double titanium force-sensing beam load cell. Experiments were performed to investigate and better characterize the rate effects of this load cell. This load cell design suffered severely from damping issues. In order to illustrate the damping taking place, a stiff piece of Si was clamped into the grips. This was a rigid sample securely tying the driving (piezo translator) and responding (load cell) halves of the system together. When running a sinusoidal frequency sweep from 0.01 Hz to 10 Hz (Figure 4-42a) and then from 0.01 Hz to 100 Hz (Figure 4-42b) on the waveform generator, the load cell signal quickly dies off as the piezo displacement continues to remain at approximately the same amplitude. (Even though the experiments on the Al microtensile beams are performed using a triangular waveform, a sinusoidal frequency sweep was used for ease of calculations). For up to 10 Hz, the piezo displacement remains at the same amplitude. It then begins to drop off also

with increasing frequency. This turned out to be a result of the piezo translator not being correctly “tuned” (see Appendix for piezo translator tuning procedures). It was then re-tuned after these experiments and was able to keep a constant amplitude up to 100 Hz. Nevertheless, even with the poorly tuned piezo translator the damping event is still observed in the load cell.

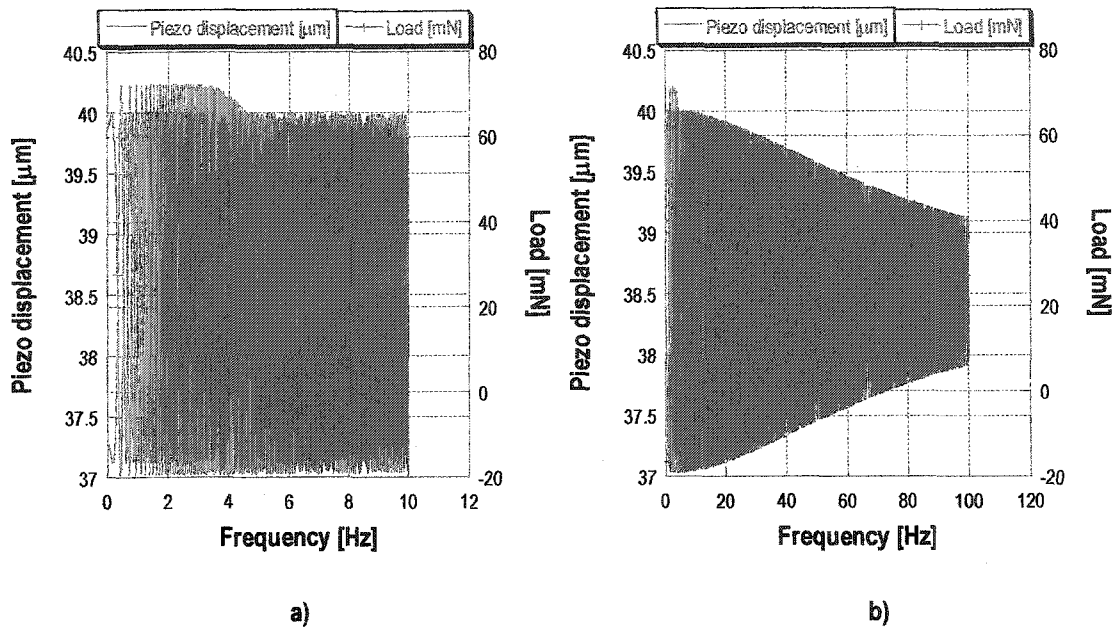


Figure 4-42: a) A plot of piezo displacement and load vs. frequency for a frequency sweep from 0.01 Hz to 10 Hz and b) from 0.01 Hz to 100 Hz; a stiff piece of Si is clamped in the grips.

This damping was modeled using Newton's 2nd law applied to a driven oscillator.⁷⁰ The external driving force is:

$$F(t) = F_o \cos(w_e t)$$

Equation 4-2: The external driving force.

Newton's second law applied to a driven oscillator gives:

$$m \frac{d^2 x}{dt^2} + \gamma \frac{dx}{dt} + kx = F_o \cos(w_e t);$$

Equation 4-3: Newton's 2nd law applied to a driven oscillator.

F is an external driving force and w_e is the angular frequency for this external driving force.

The steady-state solution (i.e. "when the energy dissipated by the damping is exactly balanced by the driving input") for oscillation is:

$$x = A \cos(w_e t + \delta);$$

Equation 4-4: The steady-state solution for oscillation.

A is the amplitude and δ is the phase angle between the displacement, x , and the external driving force, F . By substituting the steady-state solution into Newton's 2nd law for a driven oscillator, the result is:

$$A(w_e) = \frac{F_o/m}{\sqrt{(w_o^2 - w_e^2)^2 + (\gamma w_e/m)^2}};$$

Equation 4-5: Substituting the steady-state solution into Newton's 2nd law for a driven oscillator.

w_o is the natural angular frequency of the system; m is the mass of the bottom grip, which is 0.213 [kg]. Solving for the natural angular frequency of the load cell and bottom grip structure gives:

$$w_o = \sqrt{k/m} = 378[Hz];$$

Equation 4-6: The natural angular frequency of the load cell and bottom grip structure.

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k is the stiffness of the load cell, or 30.51 [mN/ μ m]; the stiffness can also be written as 30,510 [kg/s²]. γ is the damping coefficient. The value it has for critical damping, γ_c , is a function of the natural angular frequency of the mass and the spring constant of the load cell which gives:

$$\gamma_c = \sqrt{2} \cdot m \cdot \omega_o = 113.8 \left[\frac{kg}{sec} \right].$$

Equation 4-7: The critical damping coefficient.

Using this analysis for a damped oscillator, the damping behavior taking place in the double titanium force-sensing beam load cell during frequency sweeps to 100 Hz can be modeled.

The response of the load cell is calculated for a variety of damping coefficients. For a large damping coefficient of 500 kg/s, the system is over-damped. However, for a small damping coefficient of 75 kg/s, the under-damping of the system is significant with a notable resonance peak at 45 Hz. The damping behavior of the double titanium force-sensing beam is best described using the large damping coefficient of 500 kg/s.

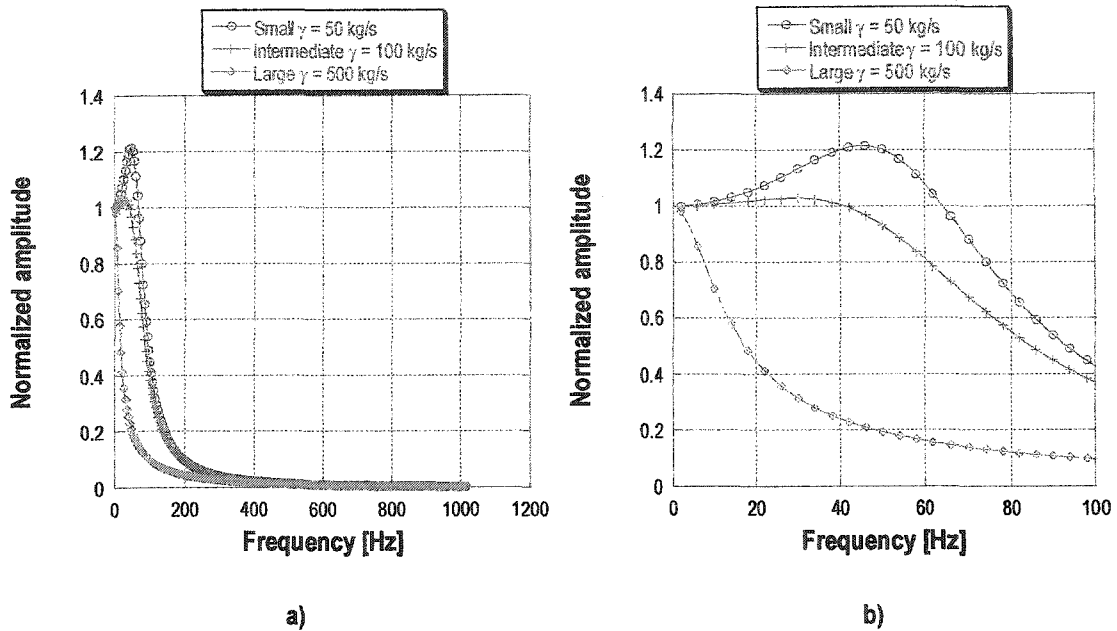


Figure 4-43: A plot of the normalized amplitude vs. frequency showing the analysis for three different damping coefficients of an externally driven damped oscillator.

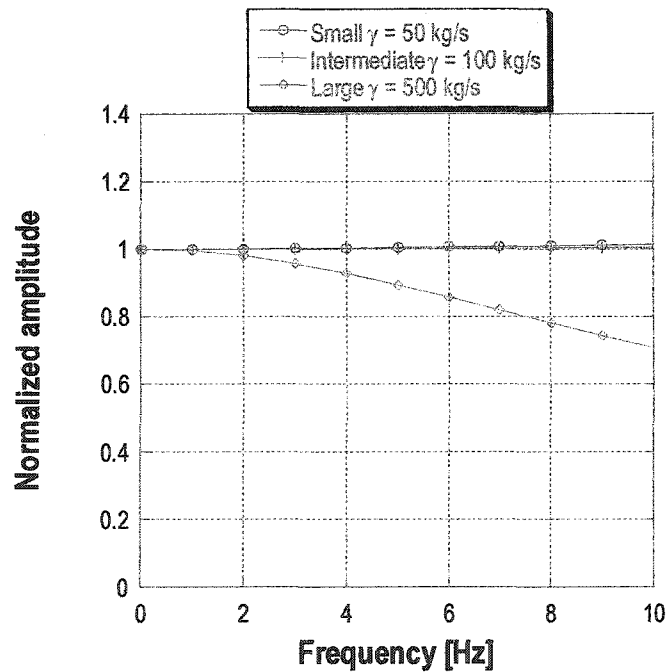


Figure 4-44: A plot of the normalized amplitude vs. frequency illustrating the response from the three damping coefficients within a frequency range of 10 Hz.

In order to see the comparison between the model and the real data, compare the large γ line in the model (Figure 4-44) to the upper line of the envelope produced in the real load data (Figure 4-42a). The real load data is slightly less damped than what the large γ shows in that the real load data begins to drop off at around 4 Hz. Because the goal was to perform fatigue experiments at a frequency of 100 Hz as well as test at high strain rates, it was concluded the double titanium force-sensing beam load cell was too slow. It was then decided to use a commercial semiconductor load cell and see if it performed as expected.³⁷

4.2.3 Microtensile system #3: commercial semiconductor load cell

The load cell configuration for System #3 was a commercial Honeywell Sensotec model 31 tension/compression miniature semiconductor load cell (Figure 4-45). The total load range for this load cell is ± 500 grams (or $\pm 4,903$ mN). When the bottom grip was attached, the load cell registers a compressive load of around 1,300 mN due to the weight of the bottom grip.

{Throughout this entire section, all measured loads are compressive on an absolute scale; however these loads are converted to relative tensile loads by subtracting the 1300 mN weight of the bottom grip}. A stiffness of 386 mN/ μ m was reported, but a stiffness of 550 mN/ μ m was measured. This load cell was rigid in that the bottom grip did not move a large amount and the microtensile beams were successfully clamped and tested. However, no information was available on its response behavior. Discussions with Honeywell Sensotec engineers confirmed that this load cell would be suitable for frequency experiments and would respond reliably under high strain rate conditions. It turned out that the response characteristics of this load cell under high strain rate conditions were very poor. An Al microtensile beam was tested for strain rate sensitivity. Again, the results from this microtensile beam were neither ideal nor expected; and

similar to the thought process for microtensile system #2, either something was wrong with the microtensile system (load cell included) or the microtensile beams. Since the microtensile beams behaved at slow strain rates, the source of the problems was most likely the load cell. Therefore, the microtensile system with the semiconductor load cell was characterized using a 20 mm (long) x 5 mm (wide) stiff piece of Si clamped in the grips.

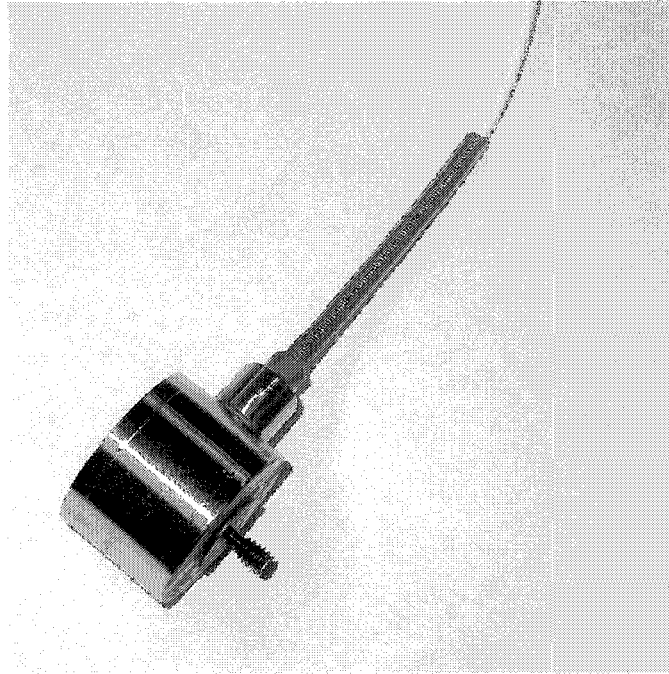


Figure 4-45: A digital image of the Honeywell Sensotec model 31 tension/compression miniature semiconductor load cell.

4.2.3.1 Microtensile system #3 experiments

4.2.3.1.1 Al microtensile beam experiment

Before characterizing the load cell, a strain rate sensitivity experiment (similar to the strain rate sensitivity experiment outlined in section 4.2.2.2.5) was performed on an Al microtensile

beam using a triangular waveform. Table 4-3 shows the displacement rates, the strain rates, and the strain amplitude measured at each strain rate. A gage length of 500 μm was used.

Table 4-3: Displacement rates, strain rates and strain amplitudes for strain rate sensitivity experiment performed on an Al microtensile beam using microtensile system #3.

Displacement rate [$\mu\text{m/s}$]	Strain rate [s^{-1}]	Strain amplitude [%]
0.005	1.0×10^{-5}	≈ 0.60
0.050	1.0×10^{-4}	≈ 0.40
0.100	2.0×10^{-4}	≈ 0.40
0.500	1.0×10^{-3}	≈ 0.40
1.000	2.0×10^{-3}	≈ 0.40
5.000	1.0×10^{-2}	N/A
10.000	2.0×10^{-2}	N/A

After cutting the side supports of the Si frame, the force on the microtensile beam was zeroed by moving the piezo translator and monitoring the load cell signal until a plateau was reached. Once the load no longer changed when extending the piezo translator, the triangle waveform was then input to the piezo translator using the manual "trigger" feature on the waveform generator. The piezo displacement of 5 μm was the same for all strain rates, and it always started at 27 μm , retracted to 22 μm , and then extended back to 27 μm for each experiment (Figure 4-46a). At a piezo displacement of approximately 3 μm (or when the absolute position was 25 μm), the microtensile beam started to register a load. All strain rates were performed on the same microtensile beam in a sequential order from lowest to highest; more than one cycle was performed with some strain rates. For the lowest strain rate of $1.0 \times 10^{-5} \text{ s}^{-1}$, only one cycle was carried out and the measured elastic modulus upon unloading was 46.3 GPa (Figure 4-47). Also, the piezo displacement and load cell track each other precisely

with respect to time (i.e. when the piezo displacement is at a maximum, the load is at a maximum as shown in Figure 4-46a).

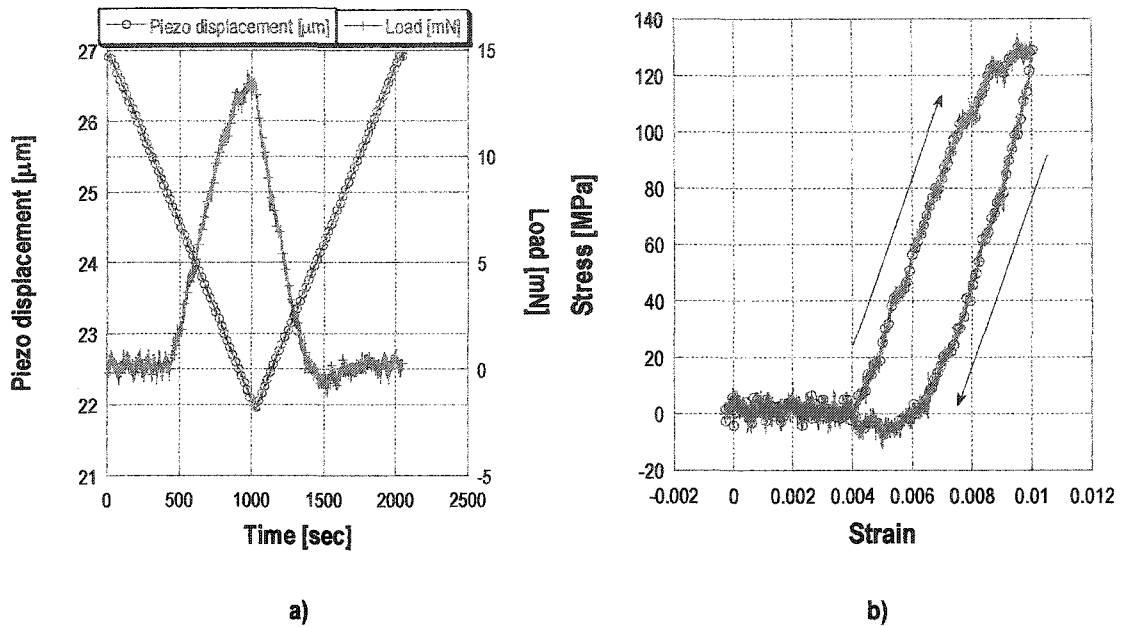


Figure 4-46: a) A plot of piezo displacement and load vs. time for one triangular cycle, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $1.0 \times 10^{-5} \text{ s}^{-1}$; scan rate = 10 scans/sec.

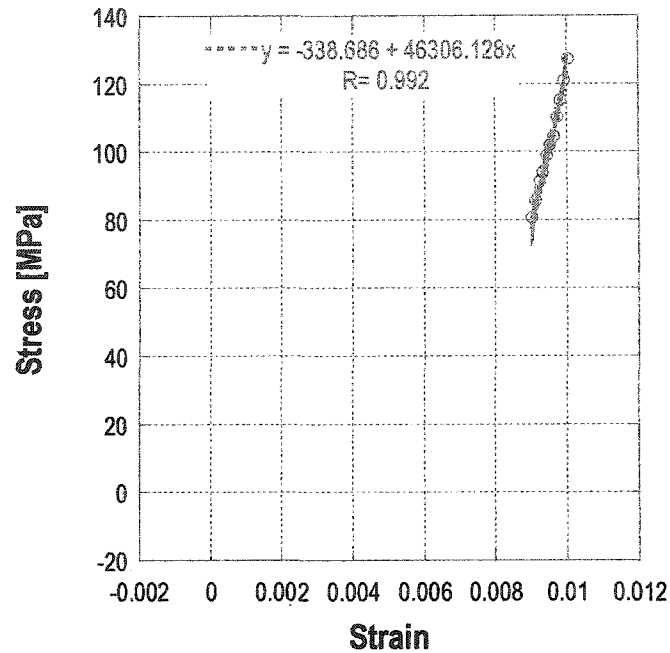


Figure 4-47: The stress-strain plot for a strain rate of $1.0 \times 10^{-5} \text{ s}^{-1}$ showing the measured elastic modulus upon unloading to be approximately 46.3 GPa.

Two cycles (with a short pause between them) were performed at the next strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$ (Figure 4-48a); the data acquisition rate was 20 scans/sec. For the initial cycle at the lowest strain rate of $1.0 \times 10^{-5} \text{ s}^{-1}$ the microtensile beam starts taking on load at around 0.4%; however, for the two cycles performed at the strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$, the microtensile beam does not take on load until 0.2% strain later, or around 0.6% strain. Most likely during the first cycle at the lowest strain rate the microtensile beam is being straightened and kinks/bends are being worked out. Also, note the maximum stress values in Figure 4-46b and Figure 4-48b; the maximum stress value reduced by about 10 MPa as the strain rate increases one order of magnitude. This may be a result of the difference in the relative strain amplitude between the two experiments, a smaller relative strain amplitude of 0.4% ($1\% - 0.6\% = 0.4\%$) for the higher strain rate results in a lower stress value of 120 MPa while the larger relative strain value of

0.6% ($1\% - 0.4\% = 0.6\%$) for the lower strain rate results in a higher maximum stress of 130 MPa. The elastic modulus upon unloading is approximately 38.9 GPa. This drop in modulus value with increasing strain rate was not expected. Moreover, this result is similar to that of testing the Al microtensile beams on the double titanium force-sensing beam load cell. The stress range for both moduli values is approximately 50 MPa. Also, temperature fluctuations can be observed when looking at the baseline of the load cell signal. When the microtensile beam is buckled, the system is drifting as a result of small variations in room temperature. This drift is not associated with the load cell itself because of its built-in temperature compensation function. These temperature fluctuations are not critical during quick experiments (< 20 minutes) but become extremely critical when performing long term (> 1 hour and up to 120 hours) experiments.

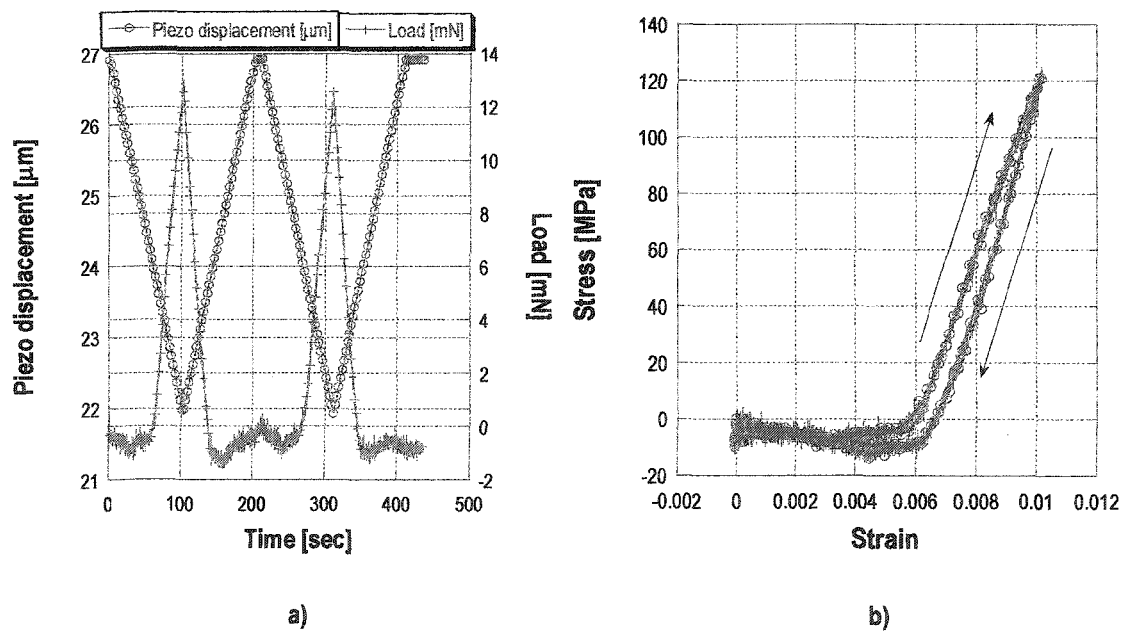


Figure 4-48: a) A plot of piezo displacement and load vs. time for two triangular cycles, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$; scan rate = 20 scans/sec.

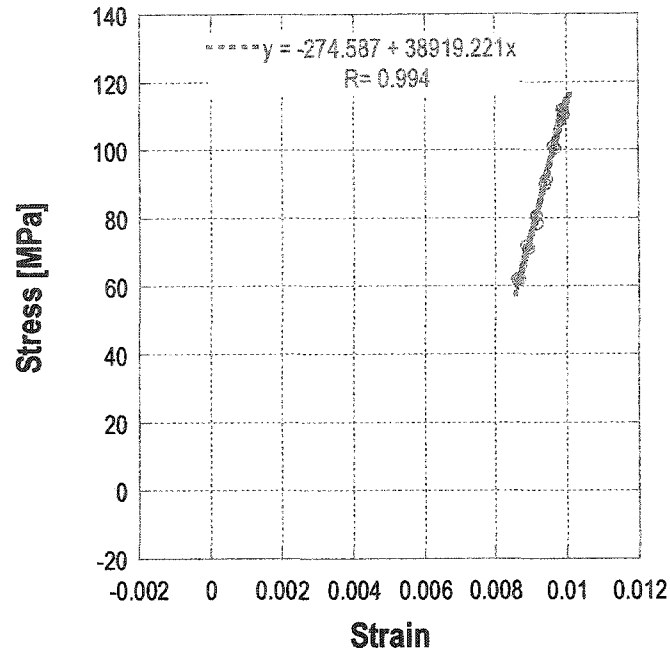


Figure 4-49: The stress-strain plot for a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$ showing the measured elastic modulus upon unloading to be approximately 38.9 GPa.

The piezo displacement and load vs. time and stress-strain plots for the remaining strain rates are shown in Figure 4-50, Figure 4-51, Figure 4-52, Figure 4-53, Figure 4-54, Figure 4-55, Figure 4-56, and Figure 4-57; the associated elastic modulus measured upon unloading for each strain rate is shown in Table 4-4. With increasing strain rate, the elastic modulus decreases; also, the opening between the loading and unloading curve becomes smaller with a crossover occurring at a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ (Figure 4-58). This crossover indicates a breakdown in the load cell response with respect to the piezo translator. Taking a closer look at the 4th loading/unloading cycle for the strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$, a loop exists between the loading and unloading cycles (this loop is present in all five cycles at this strain rate).

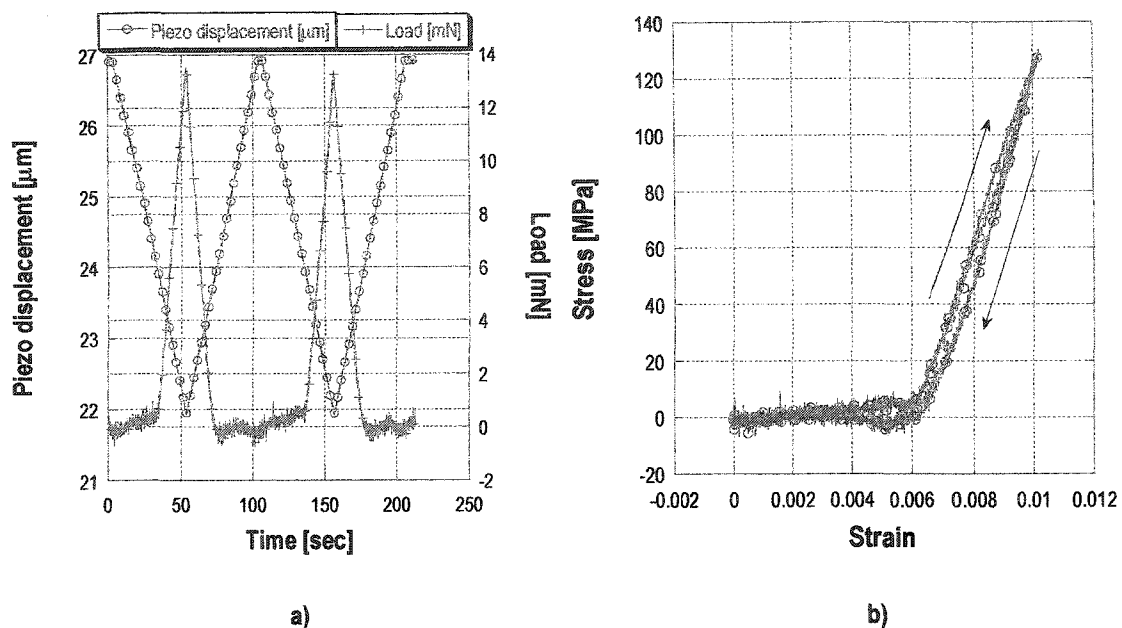


Figure 4-50: a) A plot of piezo displacement and load vs. time for two triangular cycles, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $2.0 \times 10^{-4} \text{ s}^{-1}$; scan rate = 20 scans/sec.

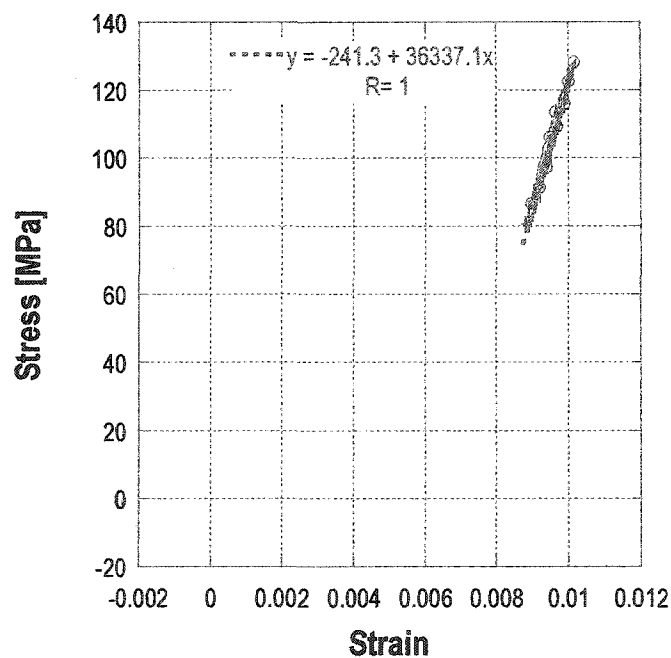


Figure 4-51: The stress-strain plot for a strain rate of $2.0 \times 10^{-4} \text{ s}^{-1}$ showing the measured elastic modulus upon unloading to be approximately 36.3 GPa

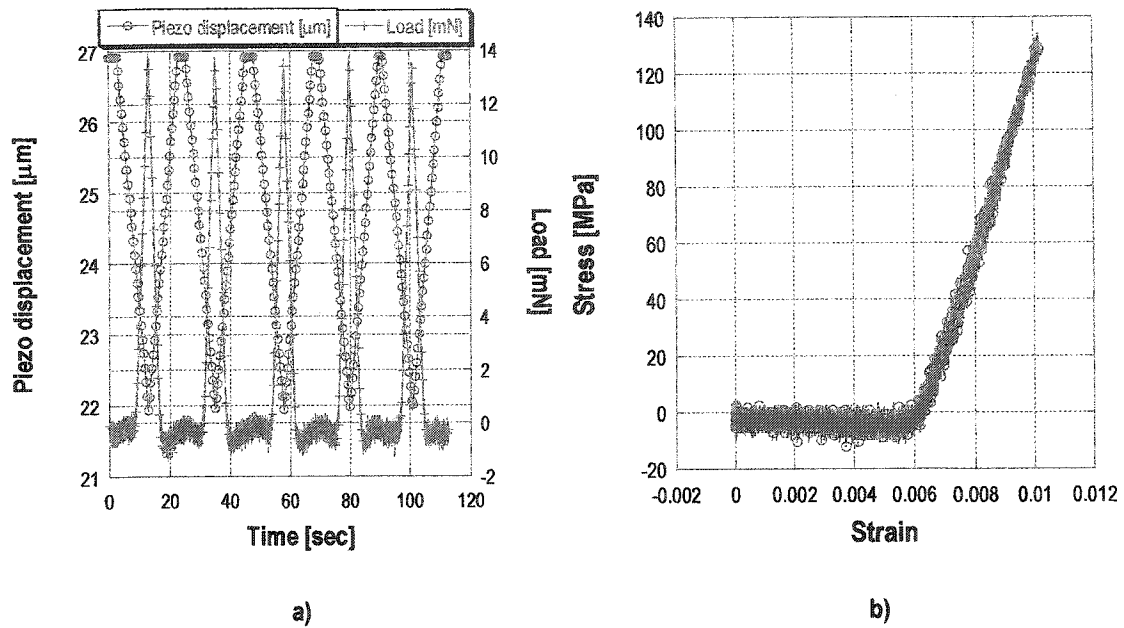


Figure 4-52: a) A plot of piezo displacement and load vs. time for five triangular cycles, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$; scan rate = 50 scans/sec.

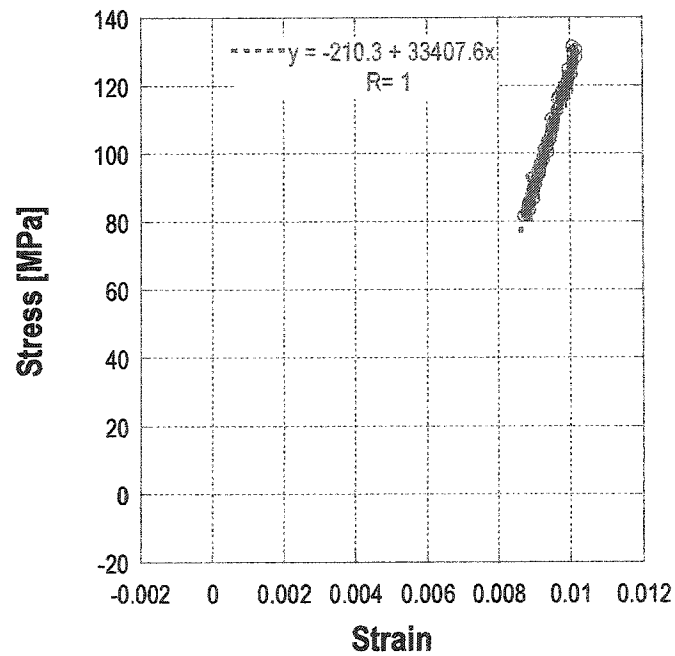


Figure 4-53: The stress-strain plot for a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ showing the measured elastic modulus upon unloading to be approximately 33.4 GPa.

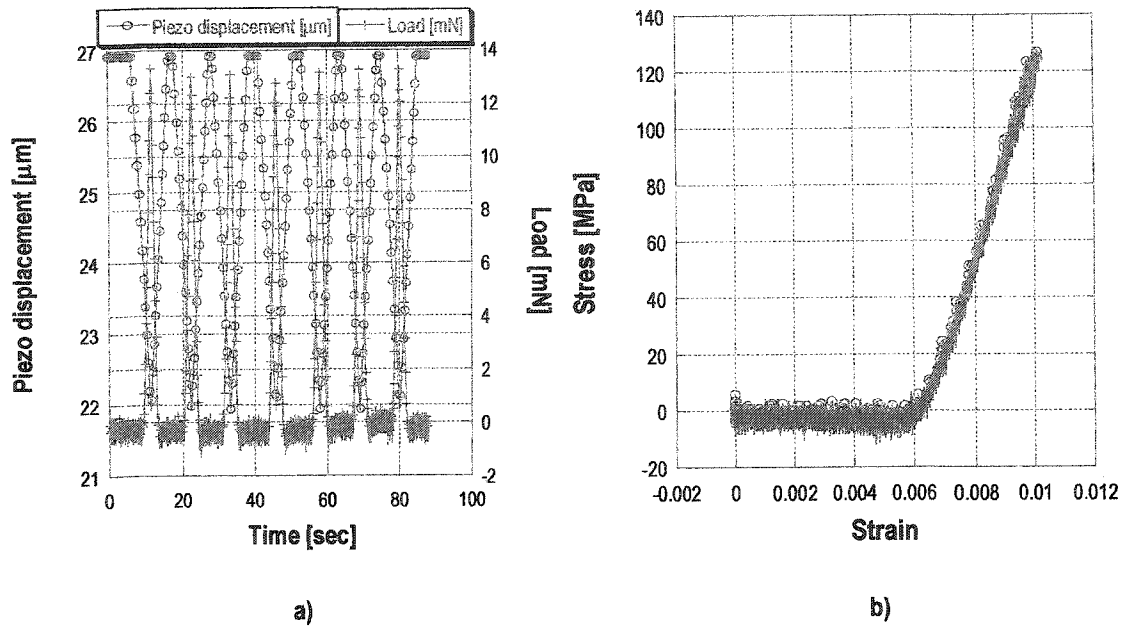


Figure 4-54: a) A plot of piezo displacement and load vs. time for seven triangular cycles, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $2.0 \times 10^{-3} \text{ s}^{-1}$; scan rate = 50 scans/sec.

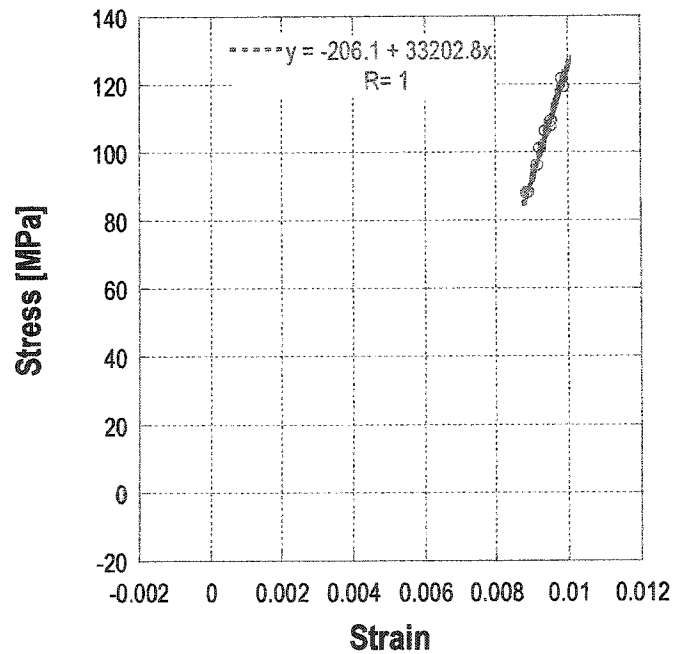


Figure 4-55: The stress-strain plot for a strain rate of $2.0 \times 10^{-3} \text{ s}^{-1}$ showing the measured elastic modulus upon unloading to be approximately 33.2 GPa

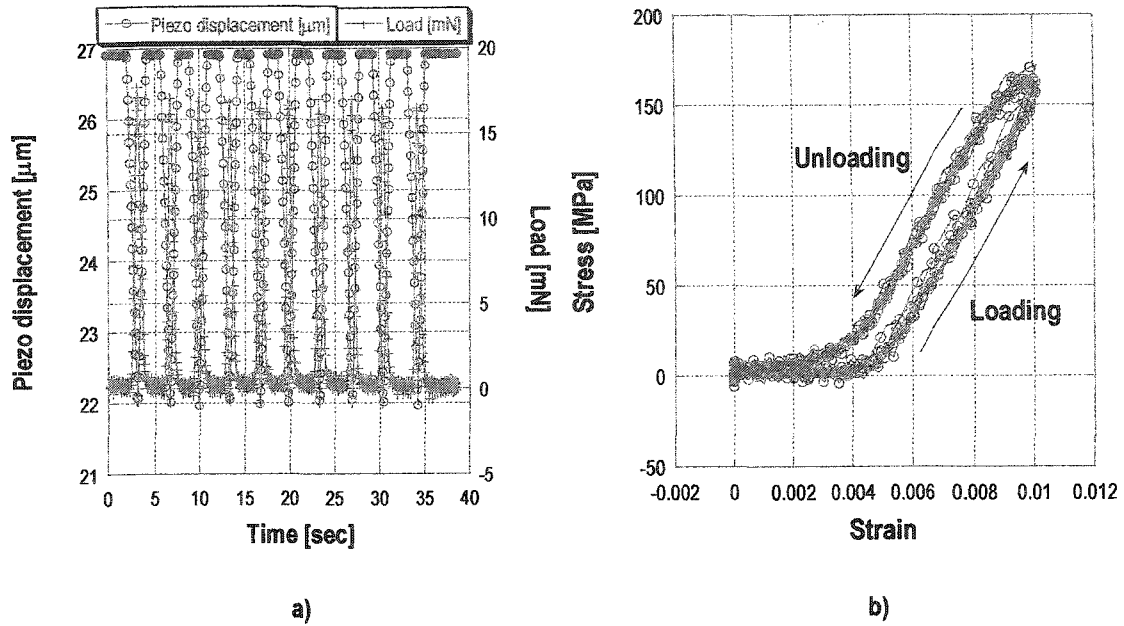


Figure 4-56: a) A plot of piezo displacement and load vs. time for ten triangular cycles, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $1.0 \times 10^{-2} \text{ s}^{-1}$; scan rate = 50 scans/sec.

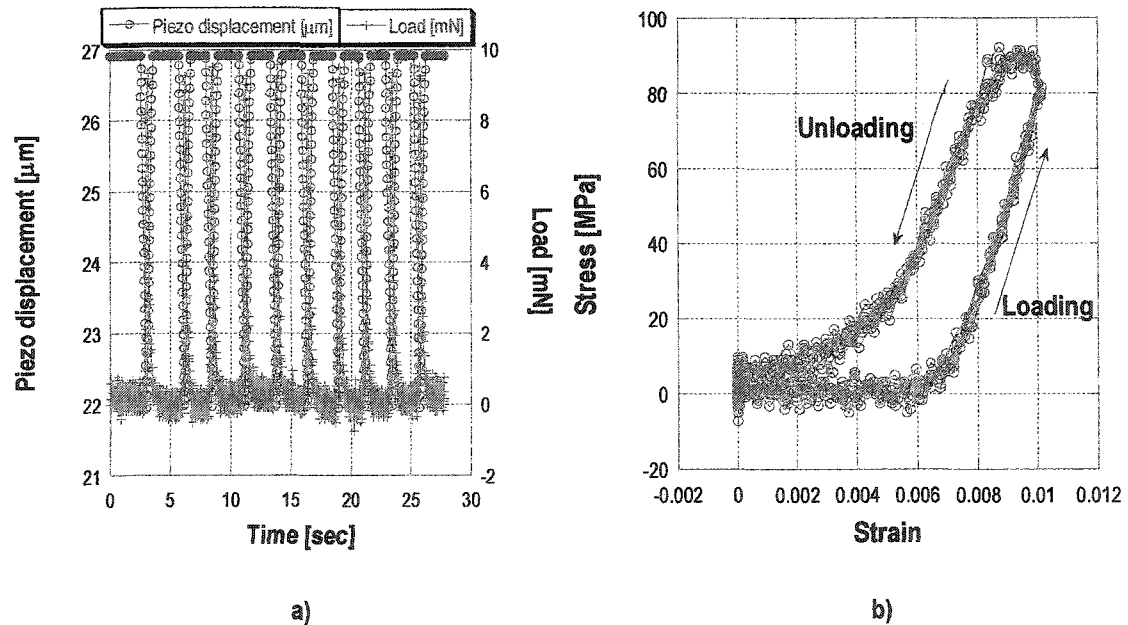


Figure 4-57: a) A plot of piezo displacement and load vs. time for ten triangular cycles, and b) the stress-strain plot for the Al microtensile beam tested at a strain rate of $2.0 \times 10^{-2} \text{ s}^{-1}$; scan rate = 50 scans/sec.

Table 4-4: Strain rate and elastic modulus measured upon unloading for the strain rate sensitivity experiment on the Al microtensile beam tested using microtensile system #3.

Strain rate [s ⁻¹]	Elastic modulus measured upon unloading [GPa]
1.0 x10 ⁻⁵	46.3
1.0 x10 ⁻⁴	38.9
2.0 x10 ⁻⁴	36.3
1.0 x10 ⁻³	33.4
2.0 x10 ⁻³	33.2
1.0 x10 ⁻²	N/A
2.0 x10 ⁻²	N/A

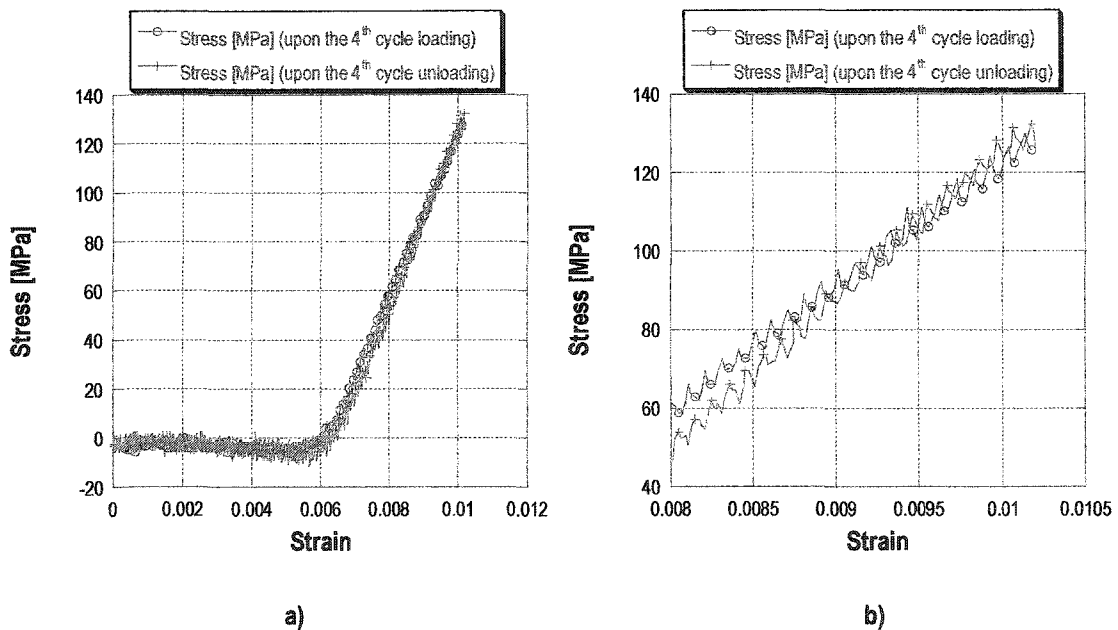


Figure 4-58: a) The 4th cycle in the stress-strain plot for a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ showing a loop between the loading and unloading curves indicating a time delay between the load cell response and the piezo translator; b) a magnified view of the crossover.

Based on the experience from the double titanium force-sensing beam load cell, this indicates a time delay between the piezo translator and the load cell response. At the highest strain rate, a time delay of 200 milliseconds exists between the piezo translator and the load cell

signals (Figure 4-59). Because of this 100 millisecond delay, it was determined that during a high strain rate cycle (or throughout many cycles in a fatigue experiment) the maximum attainable load that can be reached under slow strain rate conditions would never be reached under these high strain rate conditions; as the load cell was responding to the initial movement of the piezo translator, the piezo translator was already changing its direction. Thus, the load cell would end up changing direction prior to ever reaching its peak load. To reach the peak load, the piezo translator would have to move and be held constant at its maximum displacement for a minimum of 100 milliseconds while the load cell responded. Therefore, this load cell was regarded incapable of testing under high strain rate and fatigue conditions.

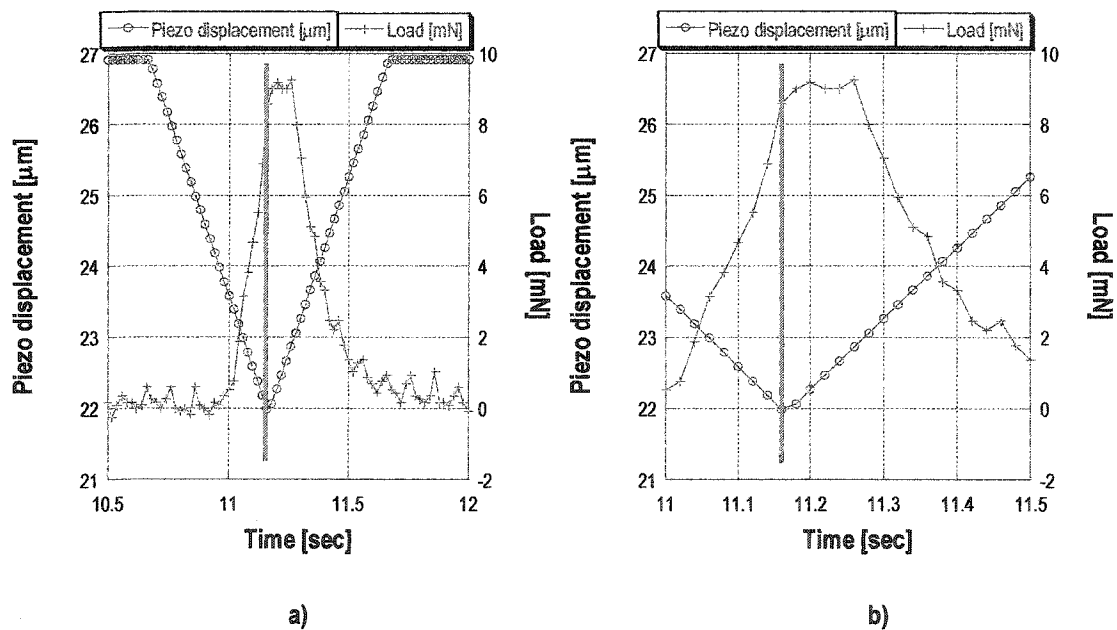


Figure 4-59: a) A plot of piezo displacement and load vs. time for the 4th cycle at a strain rate $2.0 \times 10^{-2} \text{ s}^{-1}$; the green line indicates the time at which the piezo displacement is at a maximum; note the time delay of 100 milliseconds between the maximum piezo displacement and maximum load; and b) a close-up view.

4.2.3.1.2 High strain rate and fatigue experiment

To completely characterize microtensile system #3, a stiff Si frame (with the side supports still intact, but no microtensile beam) was clamped between the grips and load-unload experiments using piezo displacement rates of 0.050 $\mu\text{m/s}$, 0.500 $\mu\text{m/s}$, 5.000 $\mu\text{m/s}$, and 50.000 $\mu\text{m/s}$ were performed (Figure 4-60, Figure 4-61, Figure 4-62, and Figure 4-63). For these experiments, load is measured in volts and not millinewtons.

The maximum piezo displacement in Figure 4-60b and Figure 4-61b is not the same. Both plots have the same range for piezo displacement and load but a different time scale. The reason for this is because of the scan rate. Because the scan rate is not fast enough for the faster piezo displacement rate, data points are being missed by the LabVIEW[®] program (i.e. under-sampling of the data is occurring). However, the piezo translator is arriving at the same final position for both displacement rates. The load cell is also reading a lower voltage for the faster rate showing that the final position of the load cell is influenced by how fast the sample is moving. This result does not bode well when trying to investigate the strain rate sensitivity of the Al microtensile beams.

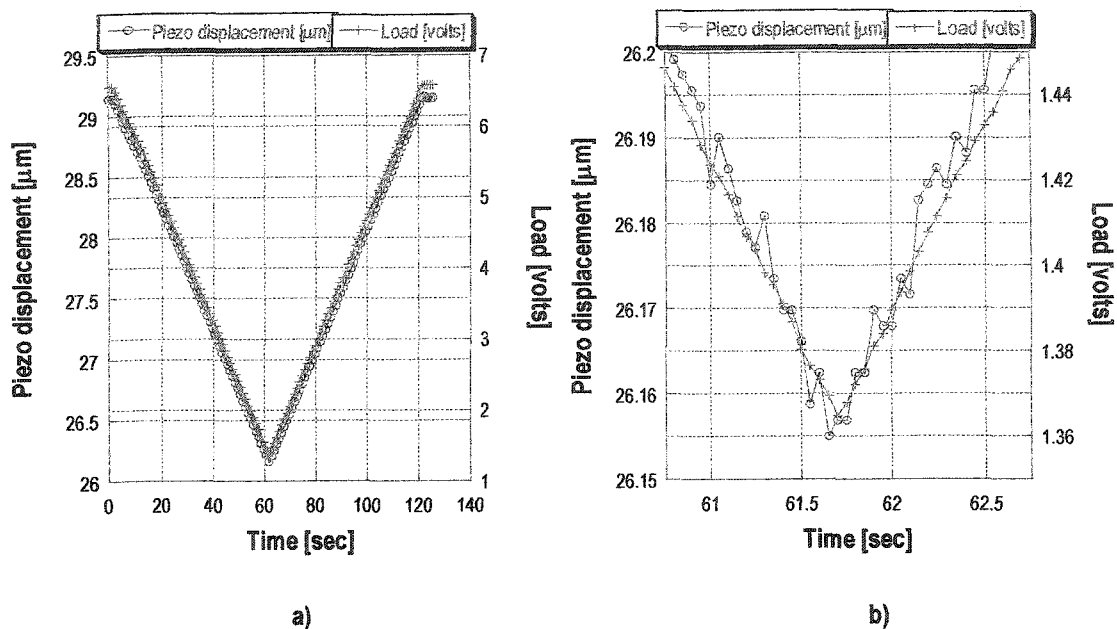


Figure 4-60: a) A plot of piezo displacement and load vs. time for a piezo displacement rate of $0.050 \mu\text{m/s}$, and b) a magnified view illustrating no noticeable time delay between signals; scan rate = 20 scans/sec.

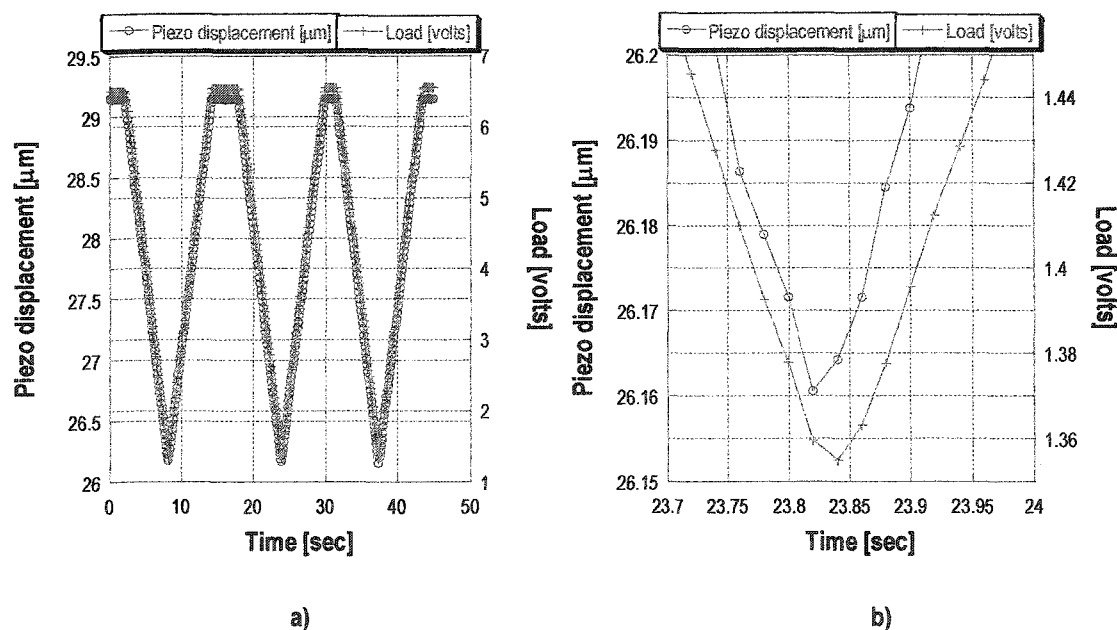


Figure 4-61: a) A plot of piezo displacement and load vs. time for a piezo displacement rate of $0.500 \mu\text{m/s}$, and b) a magnified view illustrating a slight time delay of at least 0.02 seconds between signals; scan rate = 50 scans/sec.

In Figure 4-62b, the scales of both the piezo displacement and load are increased from 0.05 μm to 0.15 μm and from 0.10 volts to 0.30 volts, respectively. The load cell has a higher voltage for the faster piezo displacement rate of 5.000 $\mu\text{m/s}$. This result contradicts the two previous experiments where the load cell range increases slightly with increasing piezo displacement rate. The final position of the load cell does not depend on the piezo displacement rate in any predictable manner. The 0.02 second time delay is not noticeable because of the under-sampling with the slow scan rate. Furthermore, an arc is beginning to take shape on the return track in the load cell signal. This arc is more prominent in Figure 4-63 when the time delay between the two signals is obvious; under-sampling is still present for this displacement rate of 50.000 $\mu\text{m/s}$. Again, note the range of the scales on the x- and y-axes.

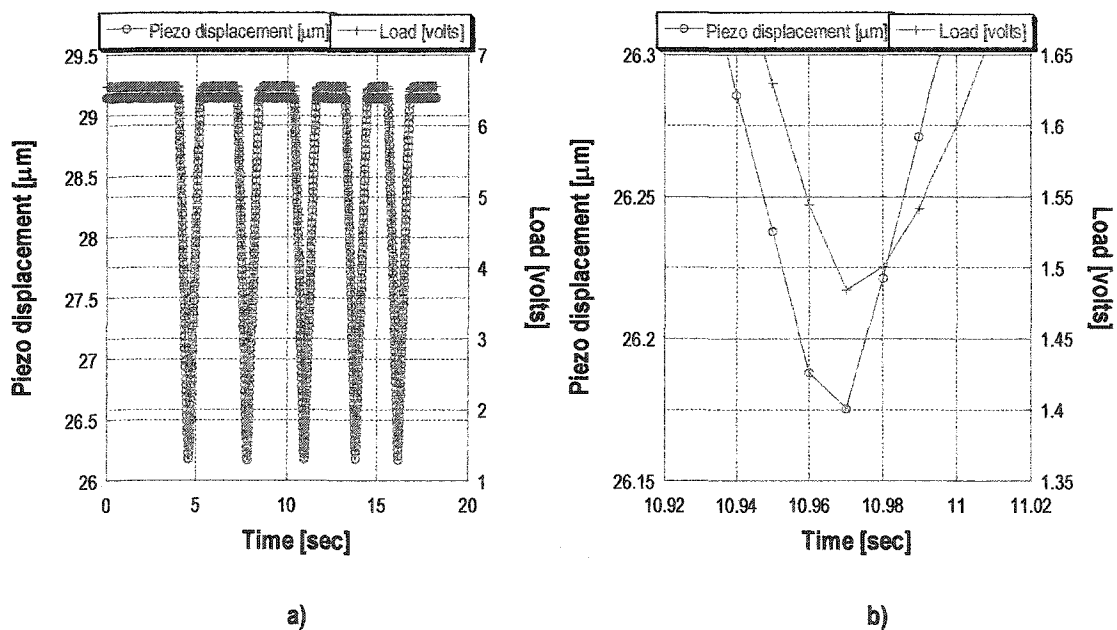


Figure 4-62: a) A plot of piezo displacement and load vs. time for a piezo displacement rate of 5.000 $\mu\text{m/s}$, and b) a magnified view illustrating the start of an arc on the return track in the load cell signal; scan rate = 100 scans/sec.

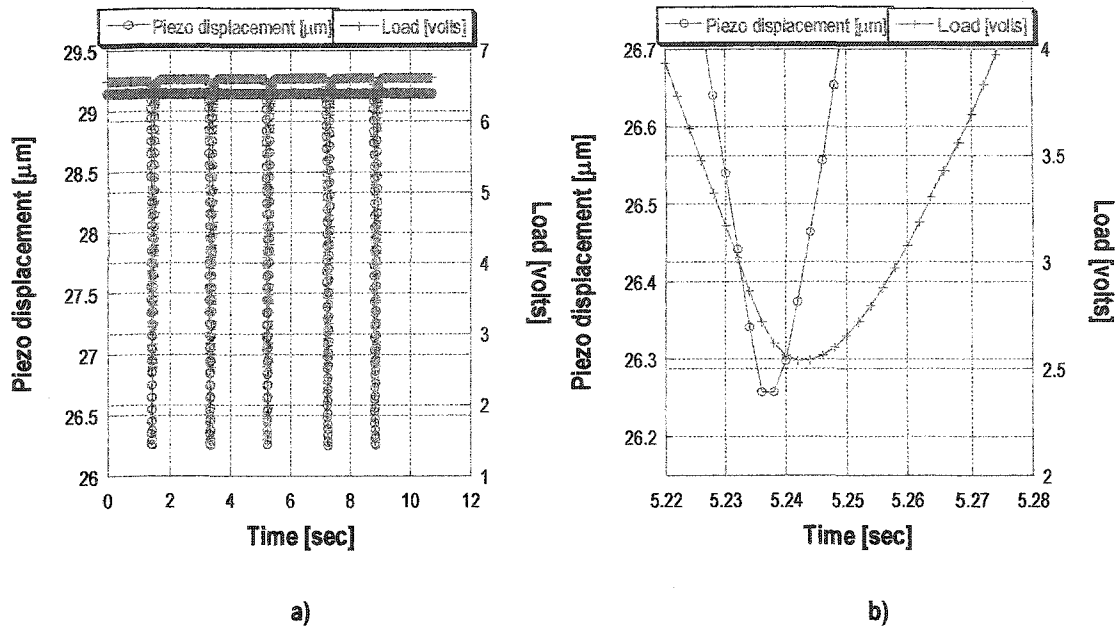


Figure 4-63: a) A plot of piezo displacement and load vs. time for a piezo displacement rate of 50.000 $\mu\text{m/s}$, and b) a magnified view illustrating a prominent arc in the load cell signal and an obvious time delay between signals; scan rate = 500 scans/sec.

Based on this performance at displacement rates of 0.050, 0.500, 5.000 and 50.000 $\mu\text{m/s}$, this load cell was deemed inadequate for high strain rate conditions. To characterize the load cell under fatigue conditions, this same sample (a Si frame) was clamped between the grips while a frequency sweep to 10, 20, and 50 Hz was performed. These experiments clearly demonstrate this load cell is also inadequate under fatigue conditions. Figure 4-64 shows the frequency sweep from 0.01 Hz to 10 Hz; note the drop in the amplitude of the load cell signal between 0.8 Hz and 1 Hz. The green line is placed on the maximum load cell signal at 0.7 Hz.

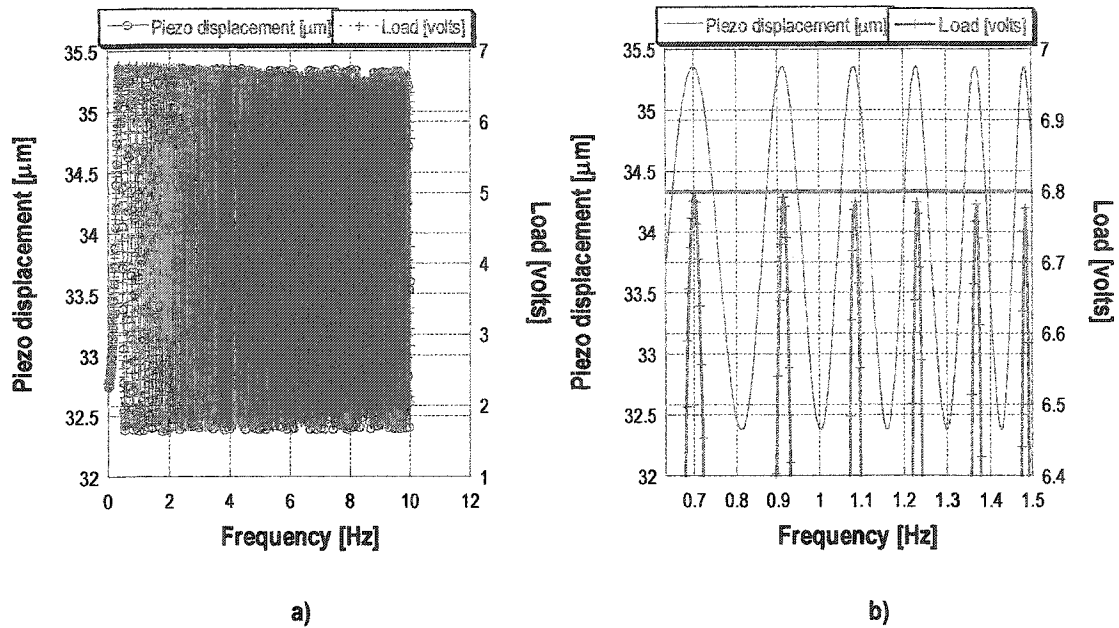


Figure 4-64: a) A plot of piezo displacement and load vs. frequency for a frequency sweep from 0.01 Hz to 10 Hz, and b) the load signal starts to drop off between 0.8 and 1 Hz; scan rate = 50 scans/sec.

Also, with the Si frame still clamped into the grips, the piezo translator was jumped by 1.15 μm at a displacement rate of approximately 280 $\mu\text{m/s}$ (Figure 4-65). This jump was executed by using the manual control on the waveform generator; the voltage changed by 100 mV. The first result from this experiment shows that the load cell has a settling time; this was expected based on the time delay observed in the previous experiments. The second result of this experiment shows an unexpected phenomenon; either the load cell or bottom grip rings after the piezo translator jump is executed; the ringing is a result of the Si frame geometry, which has a opening in the center. However, this ringing effect is not present in the load cell voltage signal. It was observed only in the capacitance grip sensor signal (Figure 4-66). The capacitance grip sensor proved to be an extremely valuable tool for characterizing this load cell. The purpose of the “ringing” experiment was to show that measuring only the piezo translator and load cell signals was not sufficient for really understanding the intricacies of the microtensile system.

Figure 4-67 more clearly illustrates the settling time of the load cell to be on the order of > 1 second. This experiment was performed with a solid piece of Si, and not the Si frame; no ringing is present in the capacitance grip sensor signal. Note the absolute load scale in Figure 4-67a.

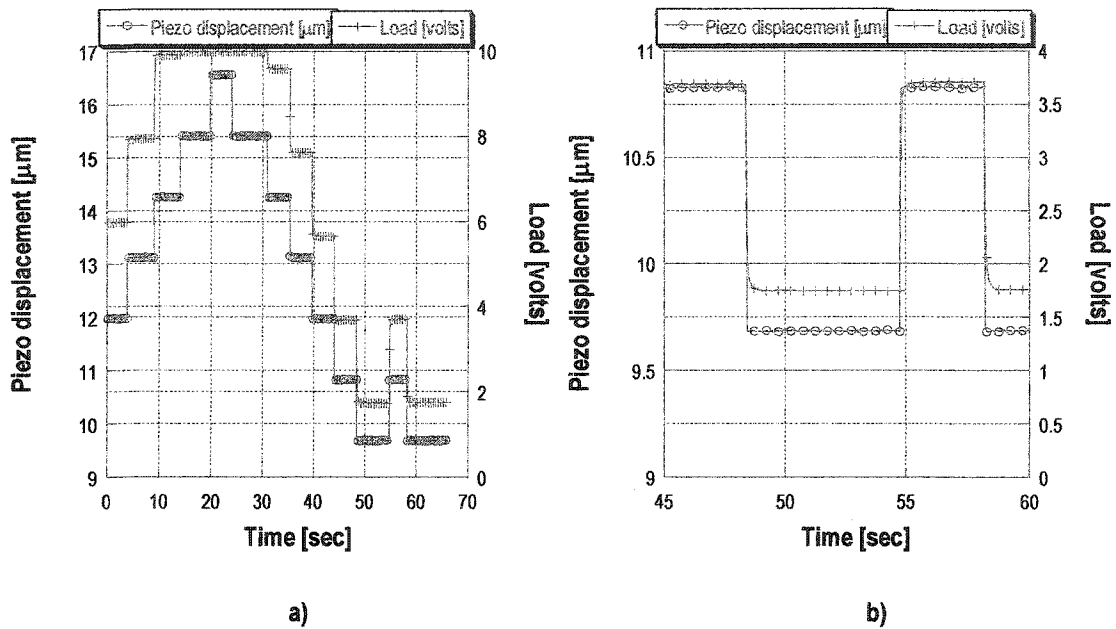


Figure 4-65: a) A plot of piezo displacement and load vs. time showing multiple jumps made with the piezo translator, and b) a magnified view of two jumps in both directions; note the transient in the load cell signal following a jump.

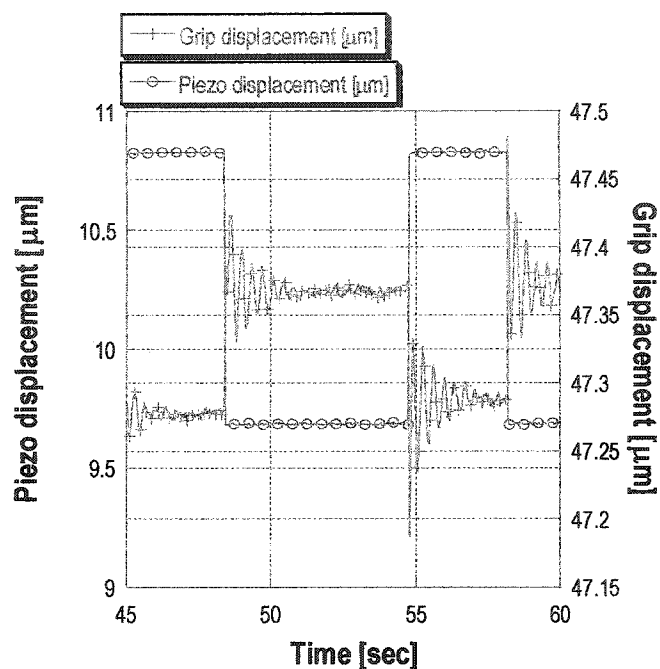


Figure 4-66: A plot of piezo displacement and grip displacement vs. time showing a ringing effect in either the load cell or bottom grip; it is not visible in the voltage signal from the load cell.

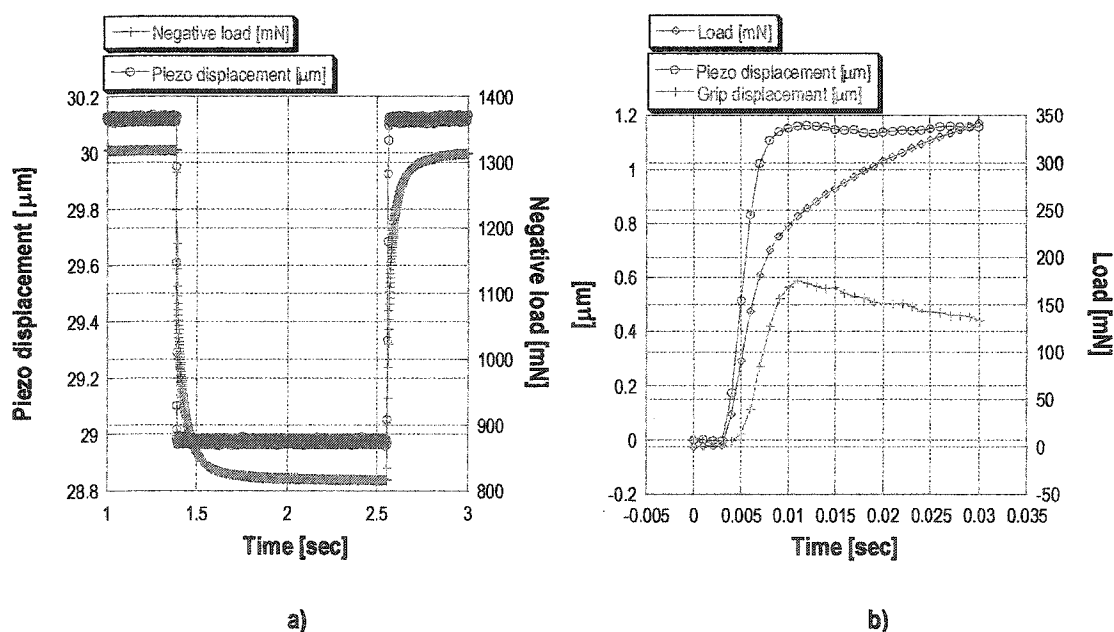


Figure 4-67: a) A plot of piezo displacement and negative load vs. time showing the settling time for the load cell to be > 1 second, and b) a plot of piezo displacement, grip displacement, and load vs. time showing that after 0.03 seconds the load cell obviously has not reached its equilibrium position.

These experiments confirm that the Honeywell Sensotec model 31 tension/compression miniature semiconductor load cell is not useful for monitoring rapid reactions in the applied load. However, it can be used for low strain rate experiments performed under short time intervals; Al microtensile beams were tested under slow strain rates (10^{-5} and 10^{-6} s $^{-1}$) using this load cell. An important message from these experiences is to first characterize the system and not test real samples until the characterization is thoroughly completed and well understood.

4.2.4 Solid load cell experiment

Because the Honeywell Sensotec load cell was inadequate for high strain rate and fatigue conditions, it was removed and replaced with a solid load cell (i.e. Figure 4-68 shows a solid piece of Al that was machined so as to have a similar stiffness (550 mN/ μ m) to the Honeywell Sensotec load cell). This allowed the remaining portions of the load train to be examined and characterized without any settling or ringing effects from the load cell or bottom grip.

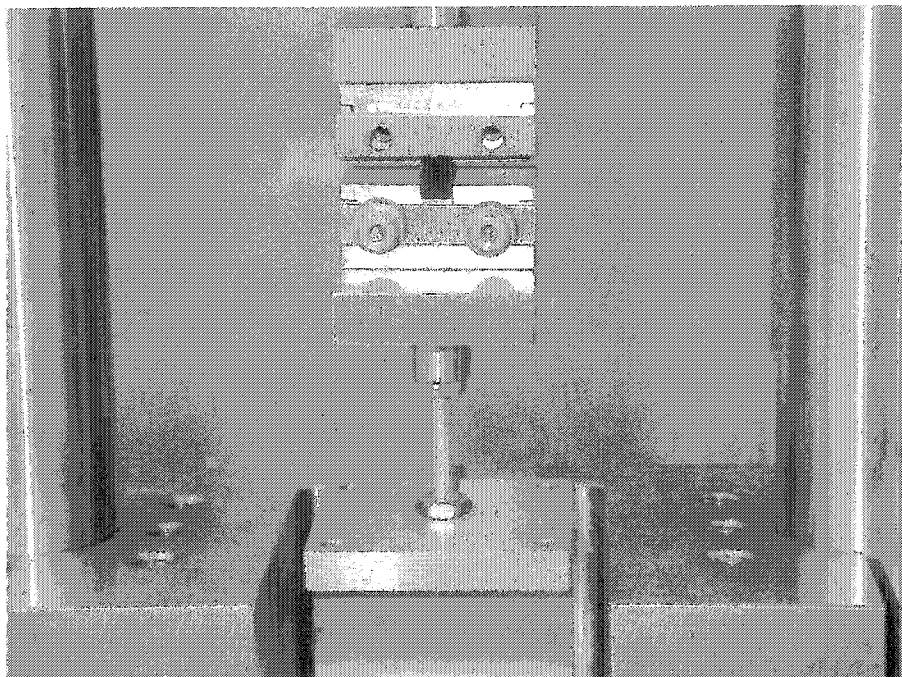


Figure 4-68: Solid Al load cell; there is a stiff Si piece (not Si frame) clamped between the grips.

With a stiff piece of Si clamped in the grips, the piezo translator was jumped using the manual control on the waveform generator; the voltage changed by 100 mV. The data acquisition rate was approximately 1000 scans/sec. Only the piezo displacement and grip displacement are recorded; no load signal is possible with this solid load cell. Figure 4-69b has two unique features: 1) the capacitance grip sensor does not move as much as the piezo translator; this is a result of the bottom grip moving upward when the stiff Si piece is pulled upward. In fact, looking at the 2nd data point on the linear part of the curve shows this effect. If no sample was held between the grips, the piezo displacement and grip displacement would be the same value; instead the grip displacement is lower than the piezo translator displacement. And 2) the capacitance grip sensor signal is trailing behind the piezo translator signal by 0.002 seconds.

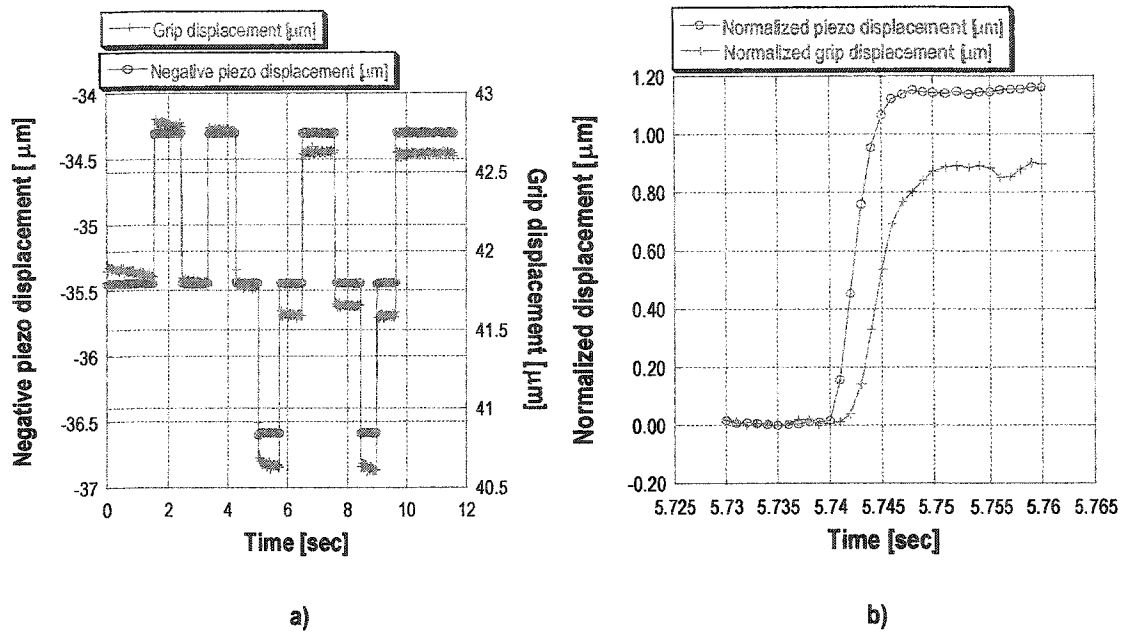


Figure 4-69: a) A plot of negative piezo displacement and grip displacement vs. time and b) a plot of both the piezo and grip displacements normalized vs. time showing the grip displacement signal trailing the piezo displacement signal by 0.002 seconds.

From this experiment, it was determined that nothing else in the load train is causing this 2 millisecond delay in the capacitance grip sensor signal (refer to section 4.1). This solid load cell experiment also pointed the way to investigating the piezoelectric load cell employed in microtensile system #4. Only one company was selling piezoelectric load cells that were capable of measuring small loads – Kistler. Before moving on with the discussion of microtensile system #4, more experiments were performed at low strain rates on the microtensile system with the Honeywell Sensotec model 31 load cell installed.

4.3 Al microtensile beam experiments (microtensile system #3)

Additional experiments were performed on 1.00 μm thick Al microtensile beams at low strain rates using the Honeywell Sensotec model 31 load cell. From the preliminary experiments on microtensile system #3, the Honeywell Sensotec load cell appeared to behave at low strain

rates. However, experiments over only a small range of low strain rates were performed. Furthermore, the fabrication procedure for the microtensile beams was refined, thereby improving the quality of the microtensile beams. The objective was to test these improved microtensile beams with the Honeywell Sensotec load cell at low strain rates. The motivation driving these experiments was (1) to observe and characterize how these new microtensile beams behaved upon a few loading and unloading cycles at slightly different low strain rates, (2) to observe a straining-to-failure experiment, and (3) to observe the stress-strain behavior without the high noise level present when using the piezoelectric load cell.

The first microtensile beam experiment was performed on sample 1A-1L-3-9. A relative strain amplitude of approximately 0.19% was used with strain rates of 8.36×10^{-5} , 2.51×10^{-4} , and $8.36 \times 10^{-4} \text{ s}^{-1}$. The gage length used in this experiment was 500 μm . The piezo translator was set to run a triangular waveform with a displacement amplitude of approximately 1.15 μm ;

$$\frac{1.15 \mu\text{m}}{500 \mu\text{m}} = 0.0023 = 0.23\% .$$

However, after cutting the Si side supports the microtensile

beam was buckled. The relative strain amplitude was derived from the grip displacement. Therefore, the relative strain amplitude was approximately 0.115% because 0.075% was needed to pull the microtensile beam taut ($0.19\% - 0.075\% = 0.115\%$). One load/unload cycle was performed for each strain rate, and the strain rates were performed in a consecutive sequence from low-to-high. A straining-to-failure experiment was carried out following the last loading/unloading event; the strain rate was $8.36 \times 10^{-5} \text{ s}^{-1}$. For all experiments carried out on 1A-1L-3-9, the data acquisition rate was 50 scans/sec. The elastic modulus measured for the lowest strain rate was approximately 63 GPa (Figure 4-70); at the two higher strain rates, the load cell starts to show the time delay effect previously discussed (Figure 4-71).

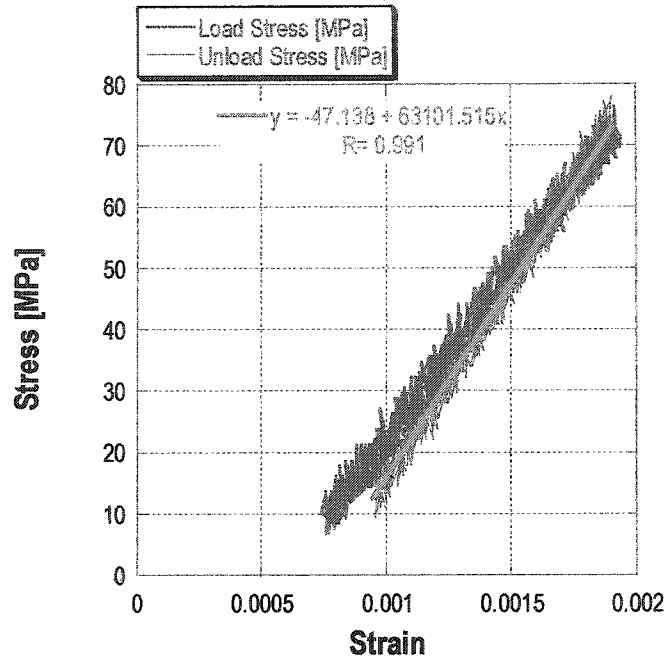


Figure 4-70: The stress-strain plot for the lowest strain rate of $8.36 \times 10^{-5} \text{ s}^{-1}$; the elastic modulus upon unloading was measured to be approximately 63 GPa.

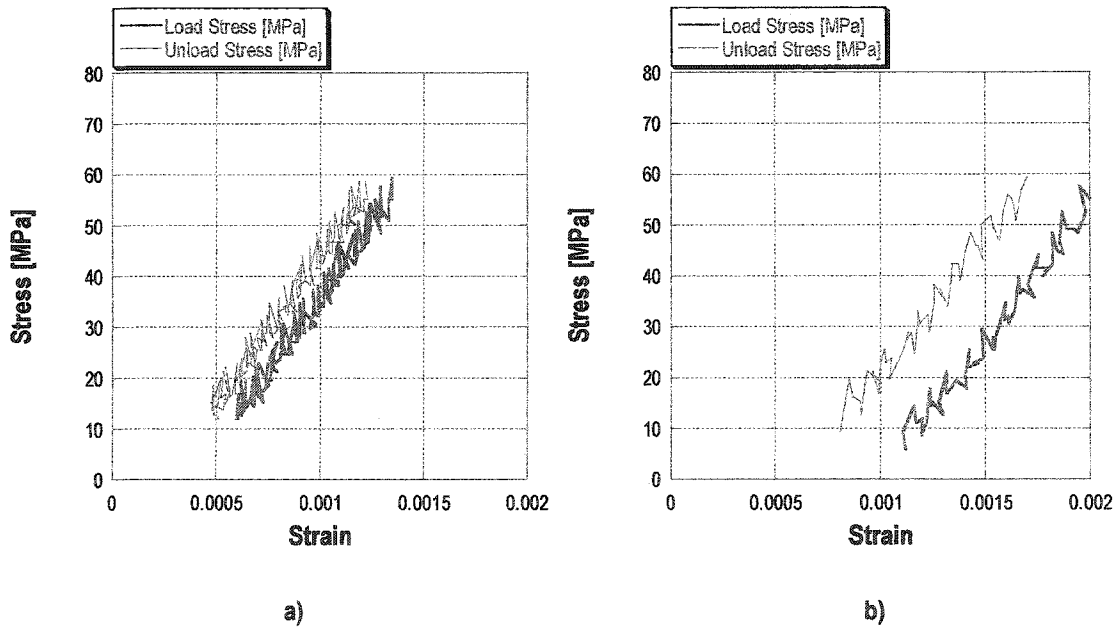


Figure 4-71: a) The stress-strain plots for the higher strain rates of $2.51 \times 10^{-4} \text{ s}^{-1}$ and b) $8.36 \times 10^{-4} \text{ s}^{-1}$; the complete stress-strain curve is not shown.

After these six loading/unloading cycles were performed, the microtensile beam was strained to failure. The stress-strain plot (Figure 4-72) shows non-linear behavior from $< 0.2\%$ strain up until failure, which occurs catastrophically at around 0.8% strain. Using a 0.2% strain offset and a modulus of 63 GPa , the yield stress is approximately 252 MPa . This non-linear behavior observed may be elastic, plastic, or a combination of both. However, it is unknown because the sample was never unloaded. It may also be that the microtensile beam is entirely anelastic up until failure; if it was unloaded prior to failure and if it enough time was given before reloading, then when it was reloaded the strain would start from zero and not some positive offset plastic strain. This anelastic behavior indicates that the microtensile beam may completely recover any deformation incurred upon loading.

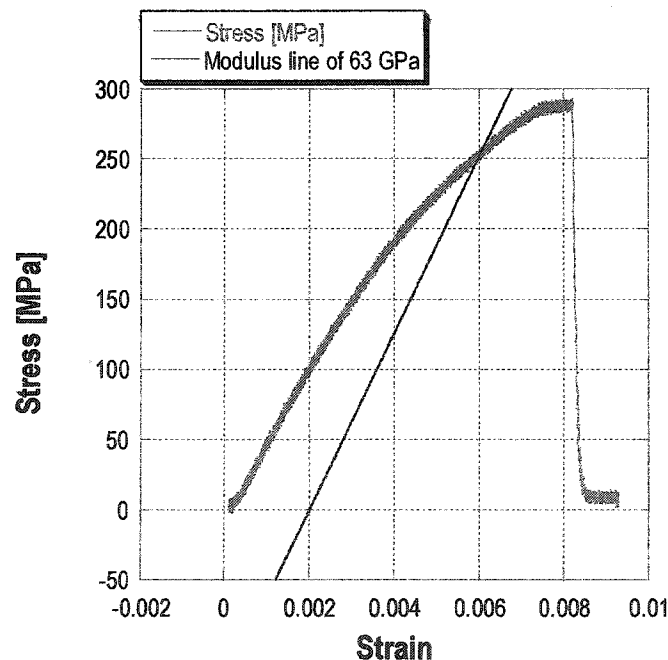


Figure 4-72: The stress-strain plot for the strain-to-failure experiment showing a yield stress of approximately 252 MPa measured using a modulus line of 63 GPa at a 0.2% strain offset.

A second 1 μm thick Al microtensile beam was tested with the Honeywell Sensotec load cell and again using three low strain rates. The highest strain rate for this experiment was the lowest strain rate on the previous experiment; the strain rates were 1.74×10^{-5} , 4.02×10^{-5} , and $8.15 \times 10^{-5} \text{ s}^{-1}$. Each experiment was run using a triangular waveform where the loading and unloading strain rates were the same; only one loading/unloading cycle was performed for each strain rate, and the strain amplitude was set at approximately 0.2%. The elastic modulus was measured for both the loading and unloading curves for each strain rate (Figure 4-73). The relative strain amplitude was different between the first and second microtensile beam experiments because of the zeroing procedure carried out immediately following cutting of the Si side supports. If the zeroed loads are slightly different, then the strain at which the beam takes on stress will be slightly different (i.e. the microtensile beam is buckled more in one case).

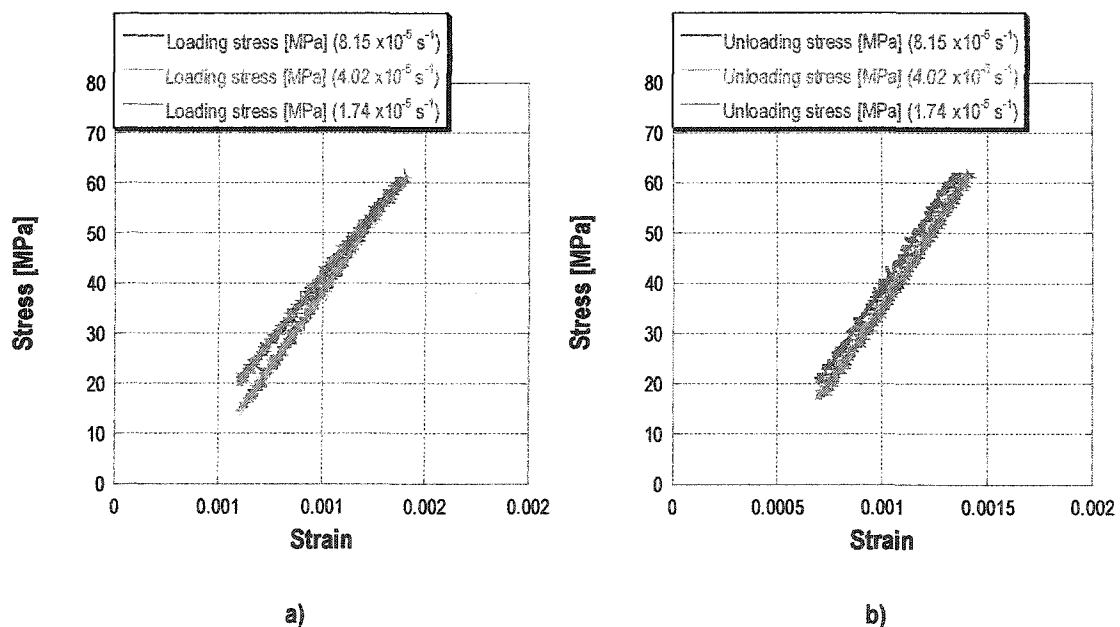


Figure 4-73: a) The stress-strain plots illustrating a slight strain rate sensitivity in the loading curves for all three strain rates; b) there is a lesser strain rate sensitivity upon unloading.

None of these lower strain rates performed on the second microtensile beam showed a crossover of the loading and unloading curves as was present for the higher strain rates on the previous experiment. It turns out that $8.3 \times 10^{-5} \text{ s}^{-1}$ is the highest strain rate for successfully testing Al microtensile beams using the Honeywell Sensotec load cell. For both microtensile beams tested with the Honeywell Sensotec load cell, the measured elastic modulus upon unloading was 63 GPa and it was not strain rate dependent. Although, upon loading, the second Al microtensile beam displays a stronger strain rate sensitivity (Figure 4-74). This strain rate sensitivity upon loading is most likely a result of 1) dislocations being forced into motion by the applied stress, or 2) a build-up of strain energy along the grain boundary caused by the applied stress, or 3) a combination of both dislocations and grain boundary energy. The loading rate (*not strain rate*) of the microtensile beam is not limited by the activity of these mechanisms. Instead, more dislocations are put into motion or more strain energy is stored along the grain boundaries. Upon unloading, however, the response from the stored strain energy (either from the dislocations, or along the grain boundary, or both) is limited; this stored strain energy limits the unloading rate of the microtensile beam, and is independent of strain rate; this can explain why the elastic modulus upon unloading is independent of strain rate.

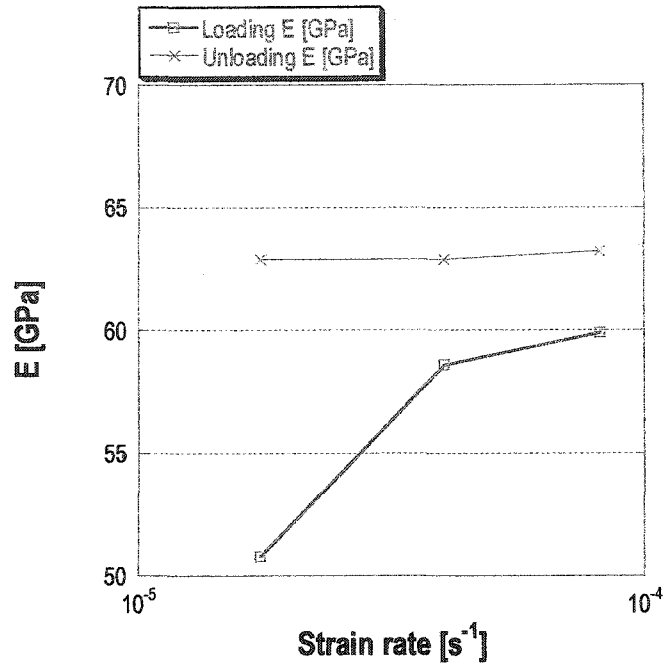


Figure 4-74: A plot of E vs. $\dot{\epsilon}$ showing the strain rate sensitivity for the above loading and unloading events.

After running one loading/unloading event at each strain rate for all three strain rates, a stress relaxation experiment was run at each strain rate (Figure 4-75). The loading curves are not shown. For the intermediate strain rate, two stress relaxation experiments at different starting stress levels were performed. This was done to observe the stress relaxation behavior of the Al microtensile beams and demonstrate how strain rate and stress level affect the amount of stress relaxation over a specific time period.

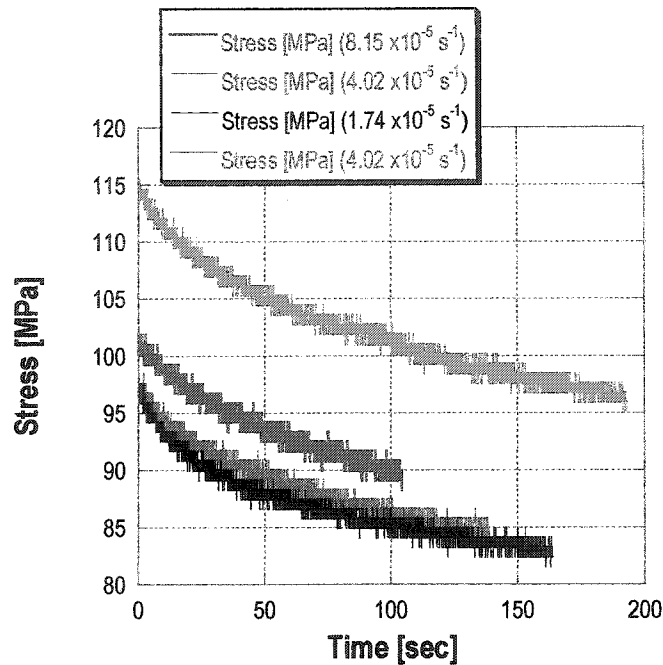


Figure 4-75: A plot of stress vs. time showing stress relaxation occurring for all strain rates; the medium rate was performed at two different starting stress levels, approximately 100 MPa and 115 MPa.

However, this system was not temperature controlled which makes results from these experiments suspicious. There was definitely some expanding and contracting of the entire system (i.e. stainless steel side rails, etc.) as indicated by monitoring grip displacement (Figure 4-76 and Figure 4-77). For these experiments, the strain was measured from the grip displacement. In a stress relaxation experiment, the piezo translator is retracted and then held constant. The load cell then relaxes as a result of the microtensile beam relaxing; if a stiff sample is exchanged for the microtensile beam, the load cell does not relax. While the piezo translator is held constant, the capacitance grip sensor can measure the displacement between the two grips; ideally, when the load decreases, this gap increases and vice versa. The amount of increase or decrease of the grip displacement is dependent on the stiffness of the load cell.

For an extremely stiff load cell (i.e. 40 N/ μm), the grip displacement is negligible.

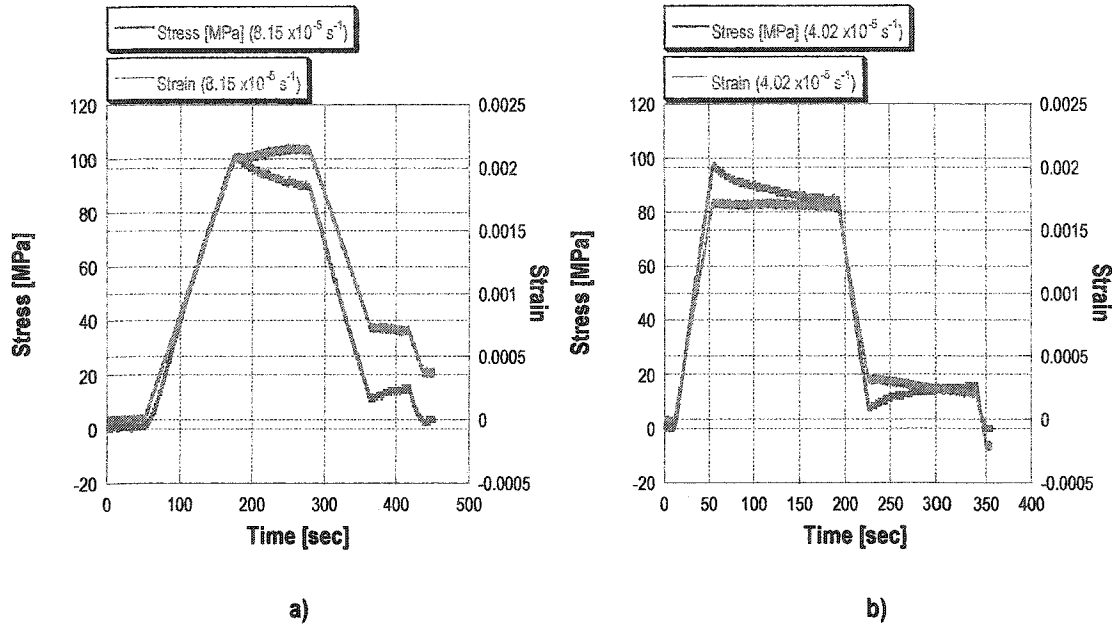


Figure 4-76: a) A plot of stress and strain vs. time showing the stress relaxation for the low strain rate of $8.15 \times 10^{-5} \text{ s}^{-1}$, and b) stress and strain vs. time for the intermediate strain rate of $4.02 \times 10^{-5} \text{ s}^{-1}$; note the inconsistency in the strain values during stress relaxation and anelastic recovery.

In Figure 4-76 and Figure 4-77, not only is the stress relaxation evident, but there is also anelastic recovery occurring after the microtensile beam is unloaded. This indicates that the Al microtensile beams exhibit a clear time dependent stress recovery and indeed behave as anelastic systems. Once more, with this data it was difficult to distinguish between behavior of the microtensile beam and that influenced by temperature. Although temperature may affect the shape of the relaxation and recovery curves, it is not a dominating variable on which the observed relaxation and recovery can be overlooked.

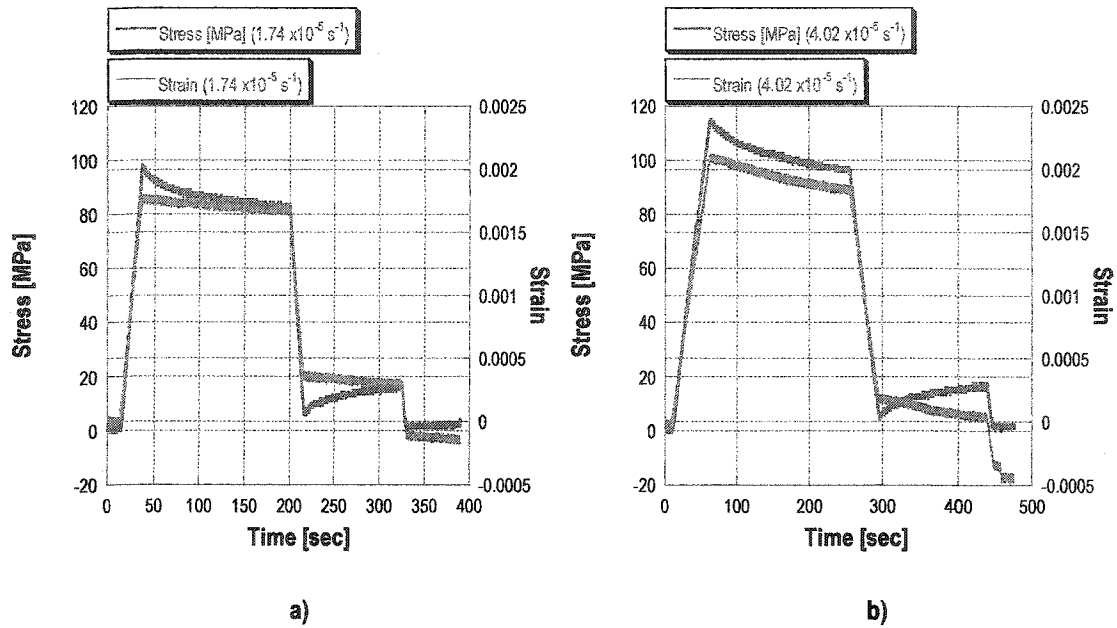


Figure 4-77: a) A plot of stress and strain vs. time for the high strain rate of $1.74 \times 10^{-5} \text{ s}^{-1}$, and b) stress and strain vs. time for the intermediate strain rate of $4.02 \times 10^{-5} \text{ s}^{-1}$ at the higher stress level.

Consider the stress relaxation for the low strain rate. Figure 4-78 illustrates how the elastic modulus varies with time. By using a least squares fit function (LINEST function in Microsoft Excel[®]) over a range of 500 data points along the stress-strain plot, a “moving” modulus was produced; with this, the maximum and minimum elastic moduli values in the stress-strain plot become clear. Furthermore, as the number of points included in the least squares fit changes (i.e. instead of 500, use 100 or 1000), then the modulus value slightly changes. Because of this variability, it was determined to be inadequate for precisely measuring the elastic modulus values.

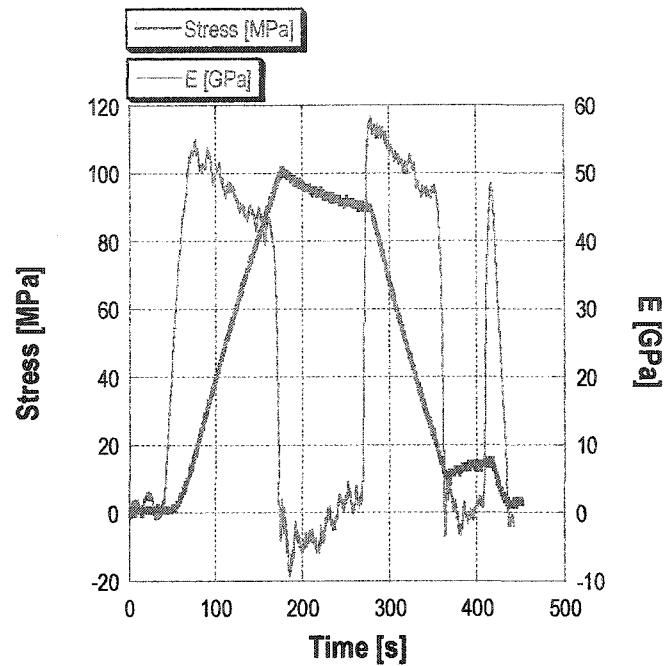


Figure 4-78: A plot of stress and E vs. time for the stress relaxation experiment at the low strain rate of $8.15 \times 10^{-5} \text{ s}^{-1}$; note how the elastic modulus varies throughout the experiment.

For all stress relaxation experiments performed, note the time scale. Initially, this time scale was necessary to make a preliminary observation of stress relaxation behavior in the microtensile beams. However, a central motivation for this research was to understand a much faster relaxation (i.e. time intervals of < 1 second); this is the reason behind investigating the piezoelectric load cell. After the stress relaxation experiments, this microtensile beam was strained to failure (Figure 4-79). This failure curve looks very similar to the first microtensile beam showing a consistency between the two Al microtensile beams and the microtensile system. Before moving on with the discussion of microtensile system #4, additional characterization was performed on the microtensile system with the Honeywell Sensotec model 31 load cell installed; this is included in the next two sections for completeness.

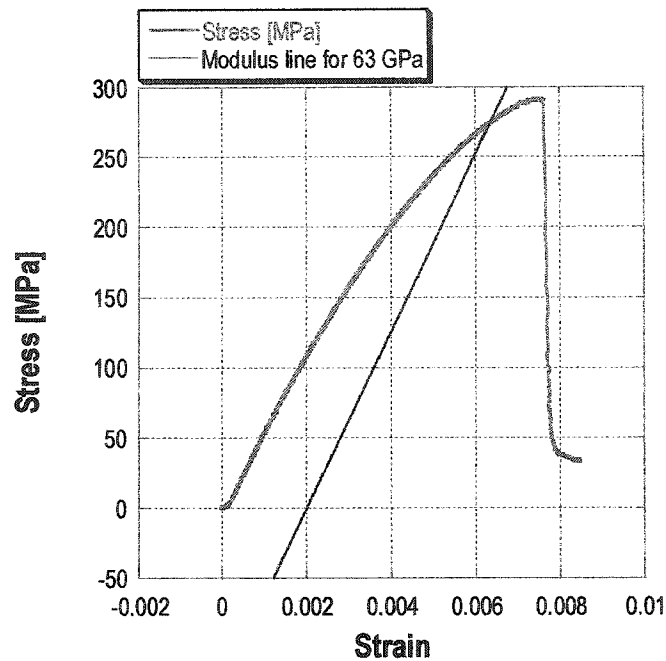


Figure 4-79: The stress-strain plot for the strain-to-failure experiment showing a yield stress of approximately 275 MPa measured using a modulus line of 63 GPa offset 0.2%.

4.3.1 Effect of humidity on the Honeywell Sensotec model 31 load cell

During long term (> 1 hour) experiments with the Honeywell Sensotec model 31 load cell, a noticeable drift was present in the signal; this load cell was affected by variation in humidity. No sample was clamped into the grips. Figure 4-80 shows the grip displacement signal drifting as the grips moved away from one another; the only way this could happen was if the stainless steel side rails of the microtensile system were expanding. Also, note how the load cell drifted into compression over the 5 hour period. Again, because nothing was clamped into the grips and the load cell was temperature compensated, another possible variable that could cause the observed drift was humidity. This was further proven when flowing dried air into the enclosed chamber containing the entire microtensile system; the drift in the load cell signal over the same

time period was dramatically reduced from 50 mN to < 1 mN. The model 31 load cell is also available in a welded format for underwater applications; this welded load cell should also be used for situations where variations in humidity levels may be present.

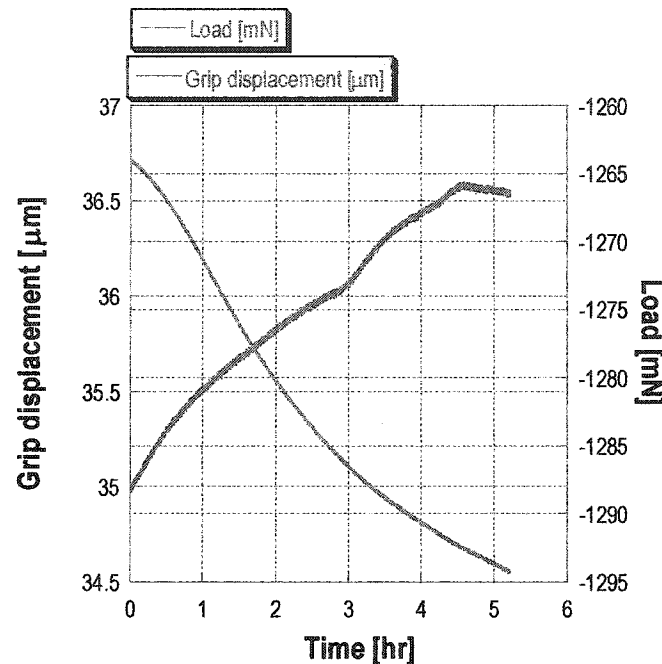


Figure 4-80: A plot of grip displacement and load vs. time showing drift in the load cell caused by humidity, and drift of the grip displacement caused by the microtensile system expanding and contracting.

4.3.2 Temperature drift of the microtensile system

The Honeywell Sensotec model 31 load cell itself was temperature compensated. Therefore, without a sample clamped into the grips and monitoring the drift in the grip displacement (Figure 4-81a), it was discerned that the stainless steel side rails of the microtensile system were in fact expanding and contracting with changes in temperature. Additionally, the piezo translator was affected by temperature as displayed by the piezo displacement in Figure 4-81b. This was a result of either the piezo crystals expanding and

contracting or something in the Polytec PI electronics that fluctuated with temperature. This experiment motivated the construction of a temperature-controlled thermal insulating box. Without precisely controlling temperature ($< 0.1^{\circ}\text{C}$), the microtensile system would have provided complicated results for long term (< 1 hour) experiments.

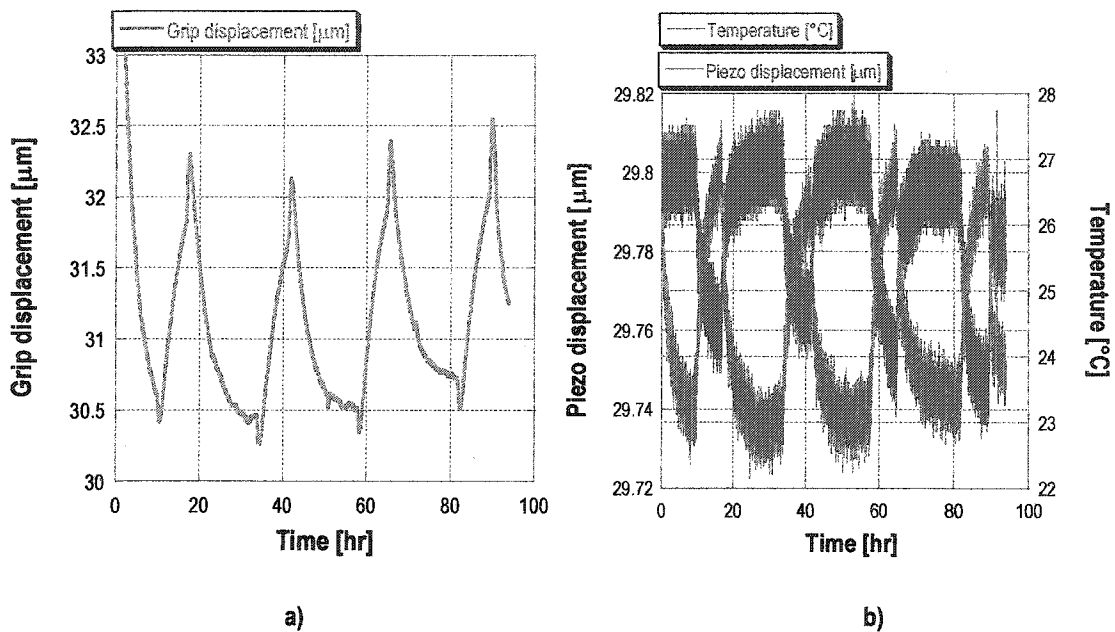


Figure 4-81: a) A plot of grip displacement vs. time showing how the stainless steel side rails of the microtensile system expand and contract, and b) a plot of piezo displacement and temperature vs. time.

4.4 Microtensile system #4: commercial piezoelectric load cell

A Kistler model 9203 piezoelectric load cell was purchased and installed into the microtensile system. Prior to testing any microtensile beams, microtensile system #4 was characterized using a stiff piece of Si clamped between the grips. This system provided the best response characteristics for high strain rate and fatigue conditions. In addition, the high stiffness of the load cell offered the freedom of having to only use the piezo displacement as a measure of sample strain without correcting for the compliance of the load cell. Also, the load cell itself

provided a very rigid connection for the bottom grip (i.e. clamping the Si frame and cutting the side supports to free the microtensile beam was the most successful with this system).

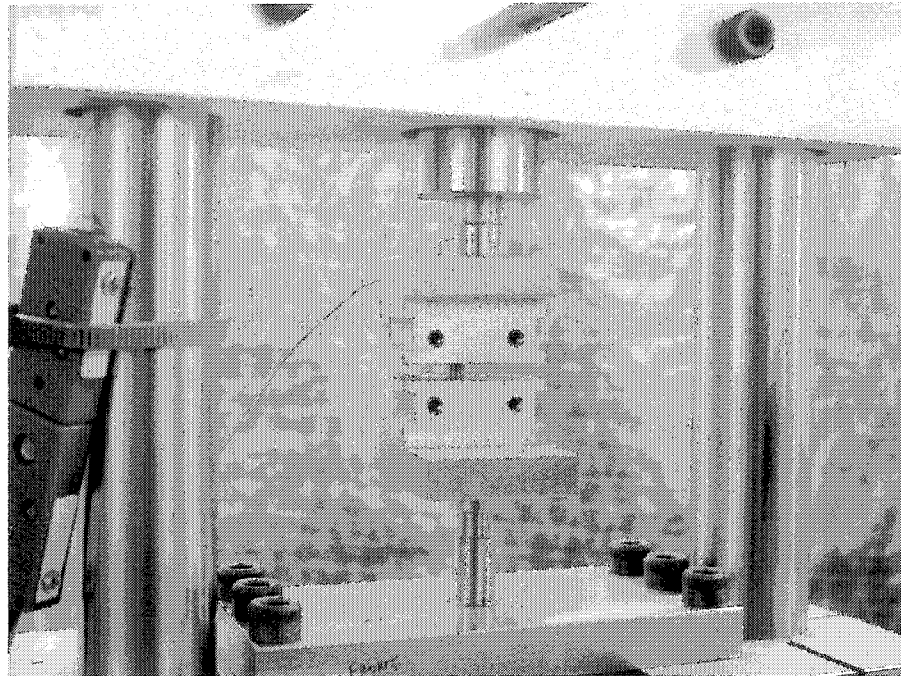


Figure 4-82: A digital image of microtensile system #4 with the piezoelectric load cell and the top and bottom grips; the capacitance grip sensor is visible between the two grips and a fine-wire thermocouple is taped to the back of the top grip.

4.4.1 Microtensile system #4 experiments

4.4.1.1 Time response of the piezoelectric load cell

The piezoelectric load cell was characterized by clamping a Si piece into the grips and then extending and retracting the piezo translator and observing the response of the load cell. Because the Si sample has a high stiffness, the piezo translator was extended a short distance; approximately 30 to 40 nm. However, the load cell registered 175 mN immediately upon the piezo translator moving. There is no detectable time delay present between the response of the

piezoelectric load cell and the driving piezo translator. Note the 2 millisecond delay is still present between the piezo translator and the capacitance grip sensor signal. The scan rate for this experiment was 1000 scans/sec.

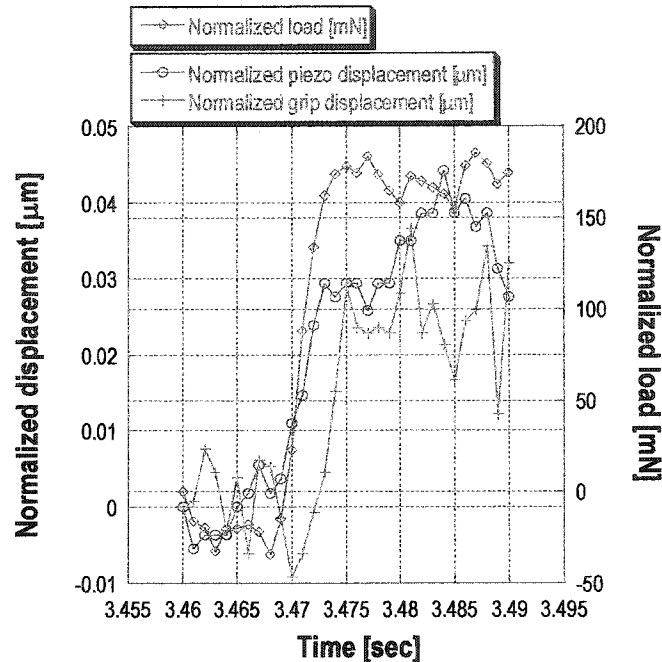


Figure 4-83: A plot of normalized piezo and grip displacement, and load vs. time showing NO time delay between the piezo translator signal and piezoelectric load cell signal.

4.4.1.2 Al microtensile beam experiments

Two 1.00 μm thick Al microtensile beams were tested to examine the strain rate sensitivity and to observe the failure curves. These results are noisy, which was the reason why ten (instead of one) cycles were performed at the two higher strain rates; only three strain rates were used (8.33×10^{-4} , 8.33×10^{-3} , and $8.33 \times 10^{-2} \text{ s}^{-1}$). The floor noise level for this load cell is approximately 10 times higher than that of the Honeywell Sensotec load cell. The primary cause of this noise is the vibrating piezo translator. The piezo translator was designed to have a

feedback loop from its internal strain gage sensor to the amplifier in order to maintain linearity between voltage and displacement. This feedback loop works with a 60 Hz frequency, therefore the position of the piezo translator is constantly being updated from the strain gage; in other words very small vibrations (i.e. corrections in displacement) are being produced. These vibrations resonate throughout the entire microtensile system (i.e. the stainless steel side rails, etc.) where the load cell picks them up. Because the load cell is extremely stiff, it does not take a lot of vibration to cause the signal to jump around and have a high noise level. When the servo controller (i.e. feedback loop) on the piezo translator is turned off, the noise in the load cell signal drops by a factor of 10. To reduce the floor noise level, an additional vibration-isolation table, on which the entire microtensile system sits, was purchased. This table does not remove vibrations that occur throughout the microtensile system, but does take care of external vibrations (i.e. from someone walking by, or from large cars/trucks driving by on the street, etc.).

A strain rate sensitivity experiment was performed on a 1.00 μm thick Al microtensile beam. Only one triangular cycle was performed (Figure 4-84a) at the low strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$; the scan rate was 50 scans/sec. A measured elastic modulus of 54 GPa was determined from the stress-strain plot (Figure 4-84b) using a least squares fit from 0.2% to 0.5% strain. A gage length of 600 μm was used. The strain for these experiments was measured by dividing the grip displacement by the gage length. The piezo displacement was used in later experiments covered in Chapters 5 and 6.

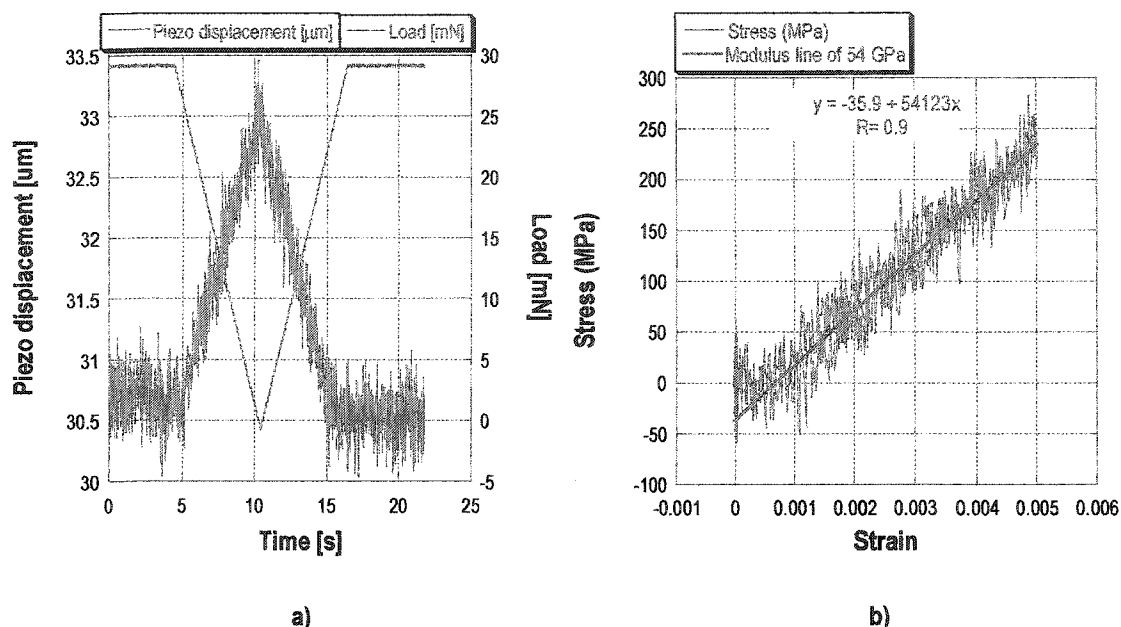


Figure 4-84: a) A plot of piezo displacement and load vs. time for a strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$, and b) the stress-strain plot showing an elastic modulus of 54 GPa measured between 0.2% and 0.5% strain for the 1st microtensile beam.

The next strain rate was $8.33 \times 10^{-3} \text{ s}^{-1}$, and the scan rate was 100 scans/sec. Ten cycles were performed so as to have better statistics for the modulus measurement. Figure 4-85 shows all ten cycles for the complete stress-strain curve. The stress-strain curves were then split into their respective loading and unloading cycles. The slopes (Figure 4-86) from the top 30 points of loading and unloading cycles were then averaged to obtain a mean modulus upon loading and unloading, respectively (Table 4-5). The mean modulus was then plotted against cycle # (Figure 4-87). No noticeable trend between modulus and cycle number was observed. Following the ten cycles at the strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$, another ten cycles were performed at a higher strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ (Figure 4-88).

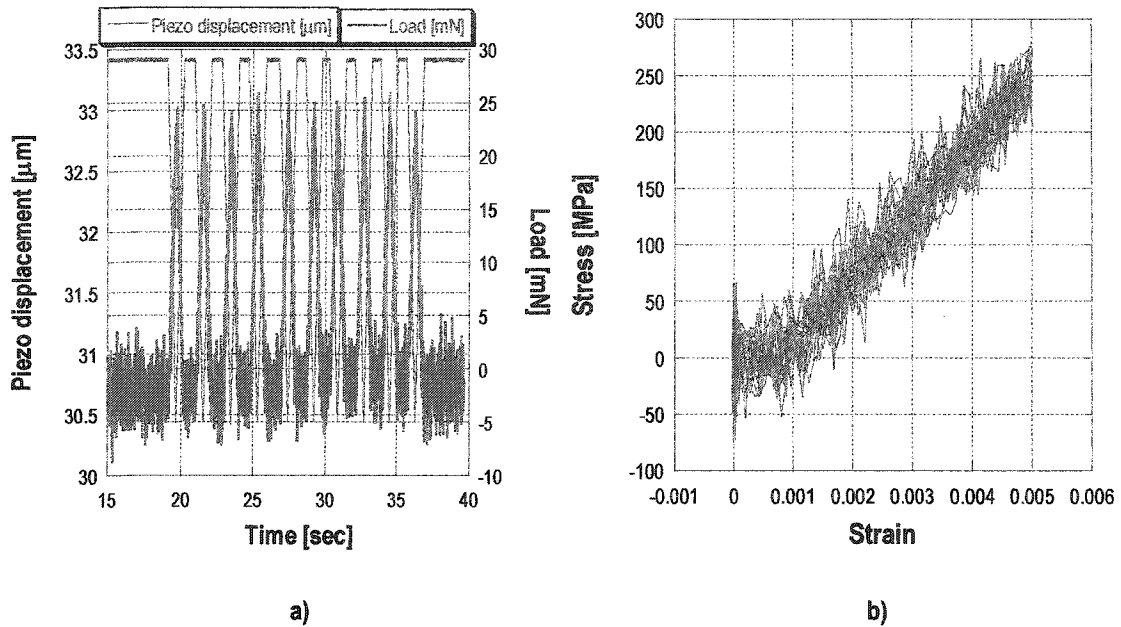


Figure 4-85: a) A plot of piezo displacement and load vs. time showing all ten cycles performed at a strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$, and b) the stress-strain plot showing all ten cycles for the 1st microtensile beam.

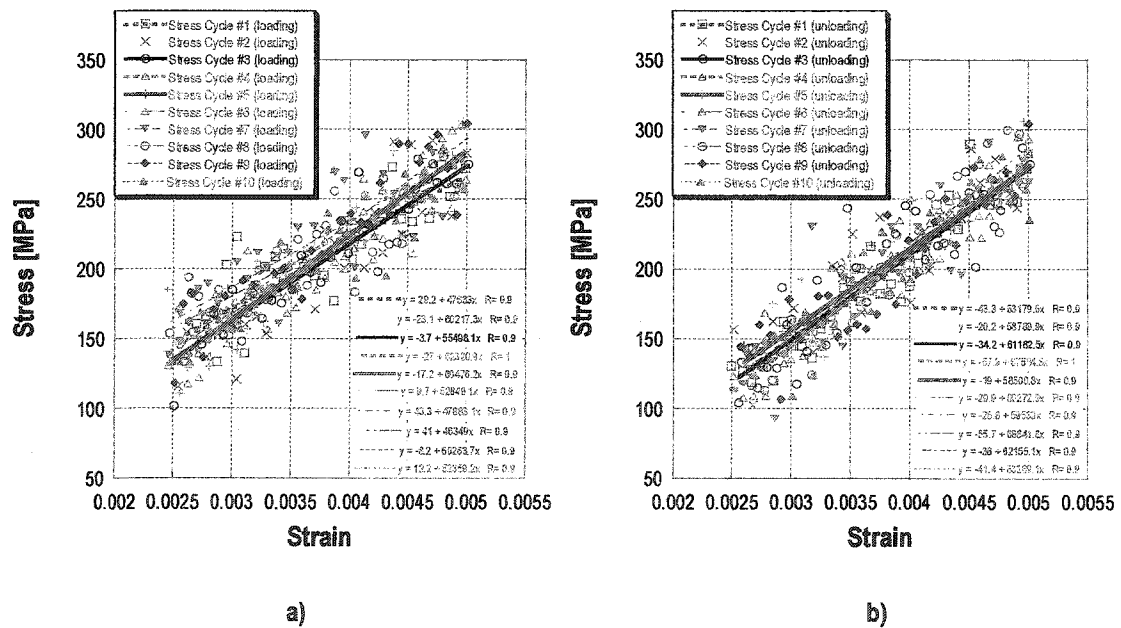


Figure 4-86: a) The stress-strain plot for all ten loading cycles and b) unloading cycles at a strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$ for the 1st microtensile beam

Table 4-5: Lists the cycle # and the modulus measured for both the loading and unloading cycles at a strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$ for the 1st microtensile beam.

Cycle #	Measured modulus [GPa]	
	<i>Loading</i>	<i>Unloading</i>
1	46.6	61.9
2	59.0	57.6
3	54.4	59.9
4	62.0	66.2
5	59.3	57.3
6	51.8	59.1
7	46.9	58.3
8	45.5	67.2
9	59.1	60.9
10	51.4	62.0
Average	53.6	61.1
Standard deviation	± 6.0	± 3.4

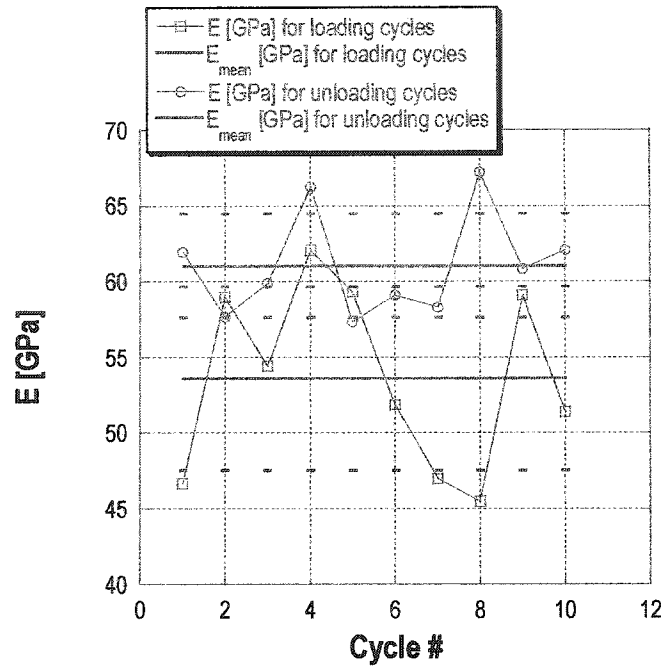


Figure 4-87: A plot of E vs. cycle # for all ten loading and unloading cycles at a strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$ for the 1st microtensile beam.

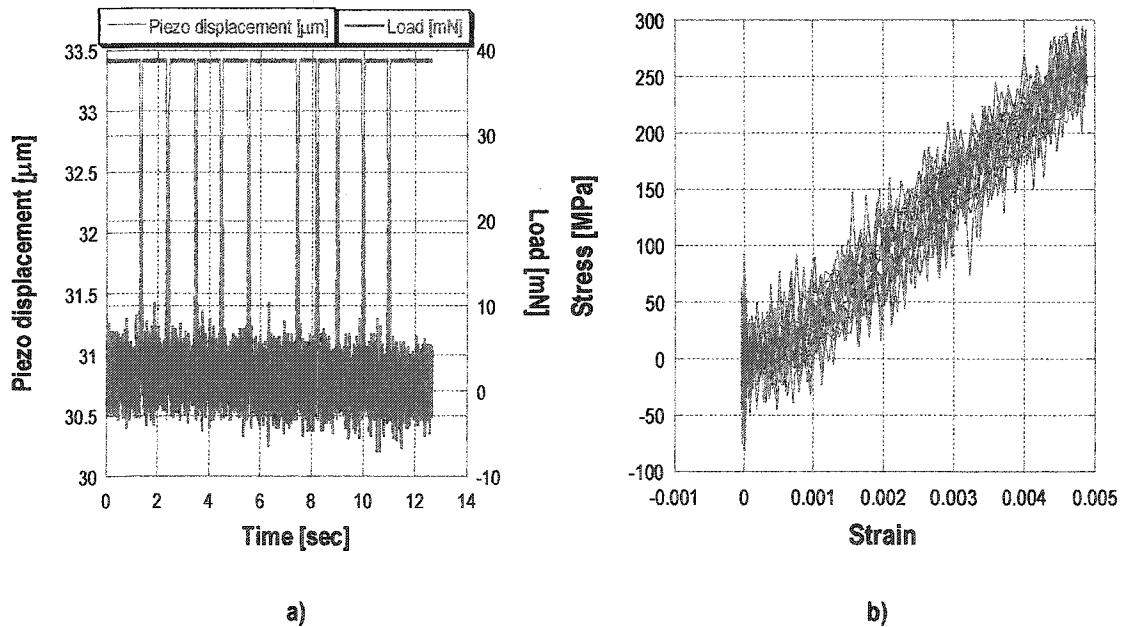


Figure 4-88: a) A plot of piezo displacement and load vs. time showing all ten cycles performed at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$, and b) the stress-strain plot showing all ten cycles for the 1st microtensile beam.

The same analysis as above was carried out for the ten cycles performed at the highest strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$. The scan rate was 1000 scans/sec. Table 4-6 shows the measured modulus for each cycle and Figure 4-89 shows the measured modulus vs. cycle #. There is no noticeable trend of E with cycle #. However, the higher strain rate has a smaller standard deviation.

With increasing strain rate, the mean loading and unloading moduli increase from 53.6 to 55.8 GPa, and from 61.1 to 62.2 GPa, respectively. This indicates very slight strain rate sensitivity for the 1st microtensile beam. Nonetheless, consideration of the error bars makes the strain rate sensitivity unnoticeable. A strain rate sensitivity exponent was not calculated for these experiments.

Table 4-6: Lists the cycle # and the modulus measured for both the loading and unloading cycles at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ for the 1st microtensile beam.

Cycle #	Measured modulus [GPa]	
	<i>Loading</i>	<i>Unloading</i>
1	54.2	63.8
2	57.4	63.9
3	56.8	60.6
4	55.4	66.9
5	56.2	62.7
6	51.9	61.6
7	56.7	61.5
8	56.6	64.1
9	58.0	65.8
10	54.3	60.8
Average	55.8	62.2
Standard deviation	± 1.8	± 2.1

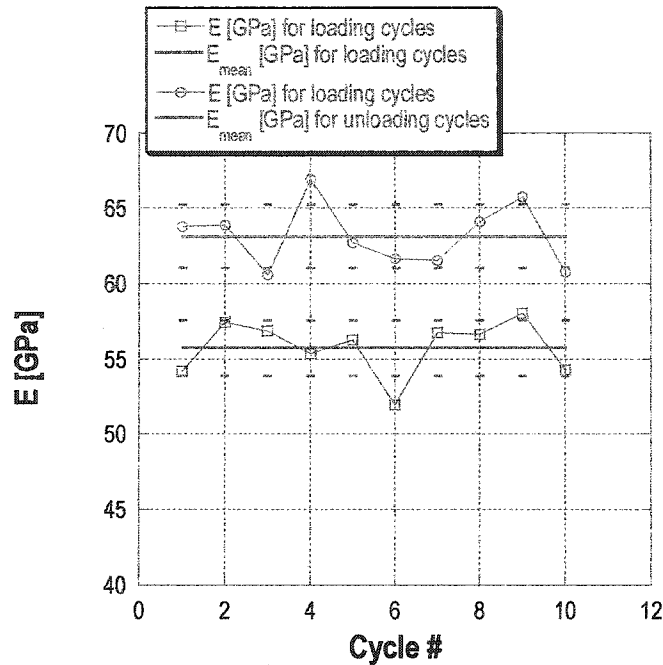


Figure 4-89: A plot of E vs. cycle # for all ten loading and unloading cycles at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ for the 1st microtensile beam.

After performing the final ten cycles, the 1st microtensile beam was strained to failure where it broke at approximately 325 MPa and a strain of approximately 0.65%. This was done by manually moving the piezo translator using the manual control on the waveform generator. From this strain-to-failure experiment, it was concluded that the strain rate sensitivity experiments performed prior to the strain-to-failure experiment used the complete stress-strain curve. It turned out that the microtensile beam broke in a brittle manner at 0.65% even though the strain rate was approximately $1.3 \times 10^{-3} \text{ s}^{-1}$.

Following this experiment, a 2nd microtensile beam was strained to failure without performing any prior cycles (Figure 4-90). The goal of this second experiment was to determine if the 21 cycles performed in the previous strain rate sensitivity experiment caused the microtensile beam to break in a brittle manner. This 2nd microtensile beam also broke in a brittle

manner concluding that the 21 cycles in the previous experiment did not perceptibly affect the microtensile beam.

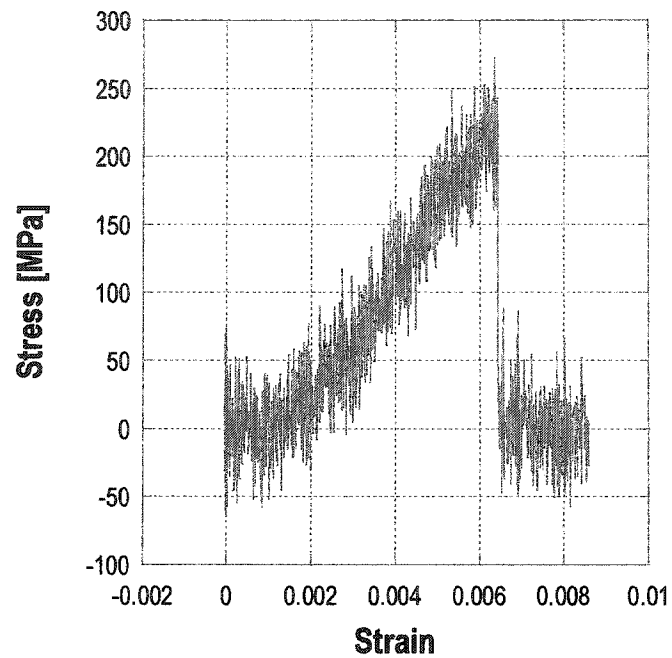


Figure 4-90: The stress-strain plot for a strain rate of $8.60 \times 10^{-4} \text{ s}^{-1}$; the failure stress and strain were approximately 225 MPa and 0.64% for the 2nd microtensile beam.

5 Microtensile beam quality effects

The quality of evaporated Al thin films strongly affects their mechanical properties. Even though the Al films studied were evaporated under similar conditions (i.e. base pressure, evaporation rate, etc.) and the microtensile beams were fabricated under similar conditions, the observed mechanical behavior varies. This was not realized until performing both strain rate sensitivity and strain-to-failure experiments. Characterization techniques (i.e. XPS, XRD, TEM, SEM, and AFM) discussed in Chapter 2 section 2.3 were employed in order to investigate possible avenues for the observed differences in mechanical behavior. However, characterizing the quality of the thin films and understanding the origin of the differences was not the focus of this research. It most likely would turn out to be an extremely useful, but complex endeavor. All the experiments in this chapter were carried out using microtensile system #4. Four main sections will be described in this chapter: strain rate sensitivity experiments, stress relaxation experiments, strain-to-failure experiments, and the characterization of anomalously strong Al microtensile beams.

5.1 *Strain rate sensitivity experiments*

Strain rate sensitivity experiments were performed on freestanding Al microtensile beams 600 μm long, 100 μm wide, and with thicknesses of 0.25 μm , 0.50 μm , 0.75 μm , and 1.00 μm . Displacement controlled experiments employing five strain rates between $8.33 \times 10^{-4} \text{ s}^{-1}$ and $3.33 \times 10^{-1} \text{ s}^{-1}$ were performed. However, effective modulus values for the highest strain rate were not determined after it was found that this strain rate was too high for the system to reliably measure at the time of these experiments. A crosshead displacement (i.e. strain

amplitude) of $1.00\text{ }\mu\text{m}$ (0.17%) was exercised in order to investigate behavior in the elastic region of the stress-strain curve. Three distinct experimental measurements were made: effective modulus (E) upon loading and unloading, and the amount of stress relaxation during the holding period at maximum strain.

A trapezoidal waveform (Figure 5-1) was performed to investigate whether stress relaxation was occurring during a holding period at the maximum strain amplitude. In addition, using the trapezoidal waveform allowed the piezo translator enough time to attain its maximum position, even at the highest strain rate. When using a triangular waveform, the piezo translator was unable to reach its maximum position at the highest strain rate. Two unique characteristics of the trapezoidal waveform are: 1) the loading and unloading rates are identical, and 2) the presence of a hold time at the maximum piezo displacement between the loading and unloading curves. This hold time at the maximum piezo displacement is proportional to the strain rate. Therefore, for the low strain rate of $8.33 \times 10^{-4}\text{ s}^{-1}$, the hold time is 9.12 seconds while the high strain rate of $1.67 \times 10^{-1}\text{ s}^{-1}$ has a 0.045 second hold time.

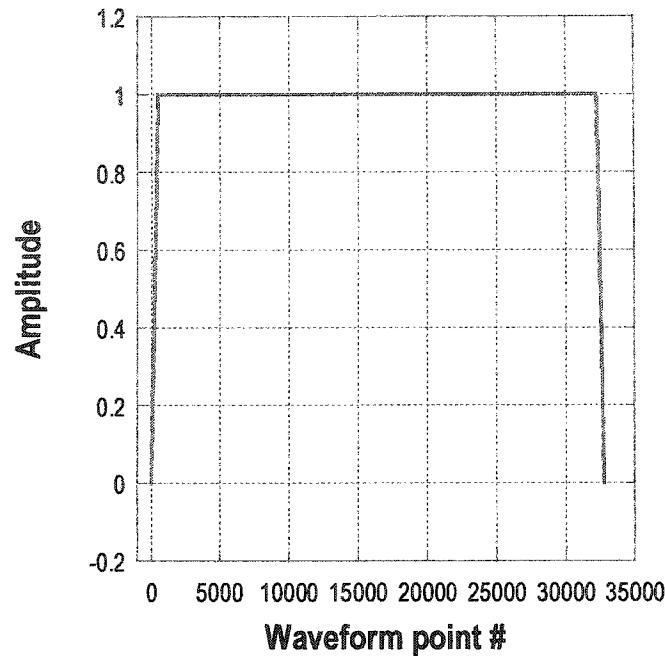


Figure 5-1: A plot of amplitude vs. waveform point # for a trapezoidal waveform (wave_F.csv); this is sent from the arbitrary waveform generator to the control input of the high voltage amplifier driving the piezo.

A total of 10 cycles were performed at each strain rate (Table 5-1) on the same microtensile beam. After the final 10 cycles, another 5 cycles were performed at one of the previous strain rates to investigate a strain-history effect. Therefore, for each microtensile beam tested a total of approximately 55 cycles were performed prior to the strain-to-failure experiment. An average effective modulus for all 10 cycles was determined upon both loading and unloading. The average effective modulus was then plotted against cycle number to investigate the dependence on cycle number; the logarithm of the average effective modulus was plotted against the logarithm of the strain rate to display the strain rate sensitivity. The time spent loading and unloading the microtensile beam as well as the total cycle time (i.e. loading time + hold time + unloading time) is listed in Table 5-2. For all strain rates, the time between

consecutive cycles was approximately 1 to 2 seconds; when adjusting to a new strain rate, the elapsed time was approximately 1 minute.

Table 5-1: Experimental parameters for the strain rate sensitivity experiments performed at a strain amplitude of 0.17%; the file labeled “wave_F.csv” was downloaded to the function generator.

Strain [%]	Piezo displacement rate [$\mu\text{m/s}$]	Strain rate [s^{-1}]	Frequency setting [Hz]	Voltage setting [mV]
0.17	0.05	8.33×10^{-5}	250.37	166
0.17	0.50	8.33×10^{-4}	2503.71	166
0.17	5.00	8.33×10^{-3}	25,037.15	166
0.17	50.00	8.33×10^{-2}	250,371.58	166
0.17	100.00	1.67×10^{-1}	500,743.16	166
0.17	200.00	3.33×10^{-1}	1,001,486.32	166

Table 5-2: Loading, unloading and total cycles times for the strain rate sensitivity experiments performed at a strain amplitude of 0.17%; the file labeled wave_F.csv was downloaded to the function generator.

Strain [%]	Piezo displacement rate [$\mu\text{m/s}$]	Loading and unloading time [s]	Hold time [s]	Total cycle time [s]
0.17	0.05	≈ 20.000	≈ 91.000	≈ 131.000
0.17	0.50	≈ 2.000	≈ 9.100	≈ 13.100
0.17	5.00	≈ 0.200	≈ 0.910	≈ 1.310
0.17	50.00	≈ 0.020	≈ 0.091	≈ 0.131
0.17	100.00	≈ 0.010	≈ 0.045	≈ 0.065
0.17	200.00	≈ 0.005	≈ 0.023	≈ 0.033

A total of nine microtensile beams were tested for a strain rate sensitivity at the strain amplitude of 0.17%. Three $1.00 \mu\text{m}$, two $0.75 \mu\text{m}$, three $0.50 \mu\text{m}$, and one $0.25 \mu\text{m}$ thick microtensile beams were tested. The strain rate sensitivity and strain-history dependence was investigated for each microtensile beam tested. The dependence of effective modulus on cycle

number was also investigated for each microtensile beam, but is only illustrated for one of the 0.50 μm thick microtensile beams (specifically, microtensile beam 0.5A-1L-5-7). This dependence on cycle number was consistent across all nine microtensile beams in that no noticeable dependence on cycle number was observed. For all nine microtensile beams, the strain was determined by dividing the piezo displacement by a 500 μm gage length and applying a correction factor of 1.18; the correction factor is discussed in Chapter 2 section 2.2.

Figure 5-2 shows the stress-strain plot for all 10 loading cycles at the strain rate of $1.67 \times 10^{-1} \text{ s}^{-1}$ for a 0.50 μm thick Al microtensile beam. The strain was offset for visual clarity; otherwise all 10 curves would lie on top of one another. The effective modulus for each cycle was determined by fitting a line to the upper 60 MPa of the stress-strain curve.

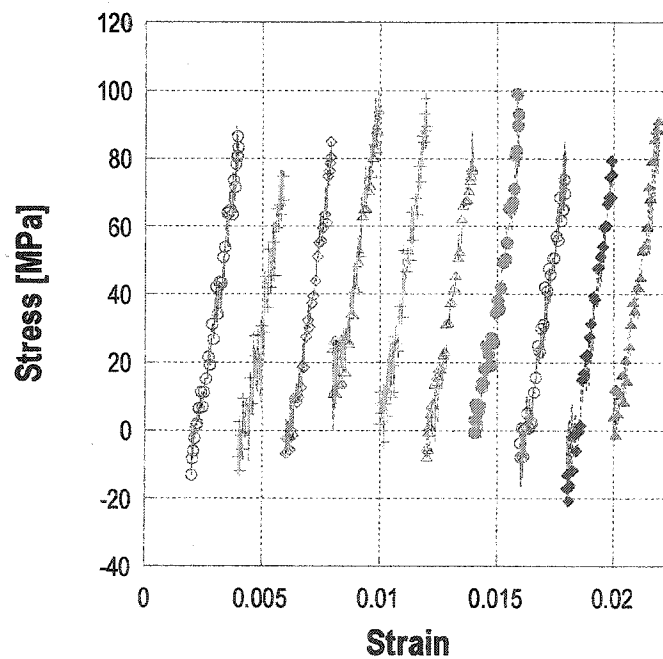


Figure 5-2: The stress-strain plot for all 10 loading cycles at the strain rate of $1.67 \times 10^{-1} \text{ s}^{-1}$ for a 0.50 μm thick Al microtensile beam (specifically microtensile beam 0.5A-1L-5-7).

The effective modulus upon loading and unloading vs. cycle number for a 0.50 μm thick Al microtensile beam is shown in Figure 5-3 and Figure 5-4, respectively. A total of 45 points are shown in these plots. However, 55 cycles (with the highest strain rate excluded) were performed prior to the strain-to-failure experiment. These plots of effective modulus vs. cycle # clearly illustrate for the 0.50 μm thick Al microtensile beam that there is no significant dependence of either the loading or unloading effective modulus on cycle number for a strain amplitude of 0.17%. The other microtensile beams (1.00 μm , 0.75 μm , and 0.25 μm thick) exhibited similar trends in effective modulus vs. cycle number at the strain amplitude of 0.17%. The plots for the other thicknesses are not included.

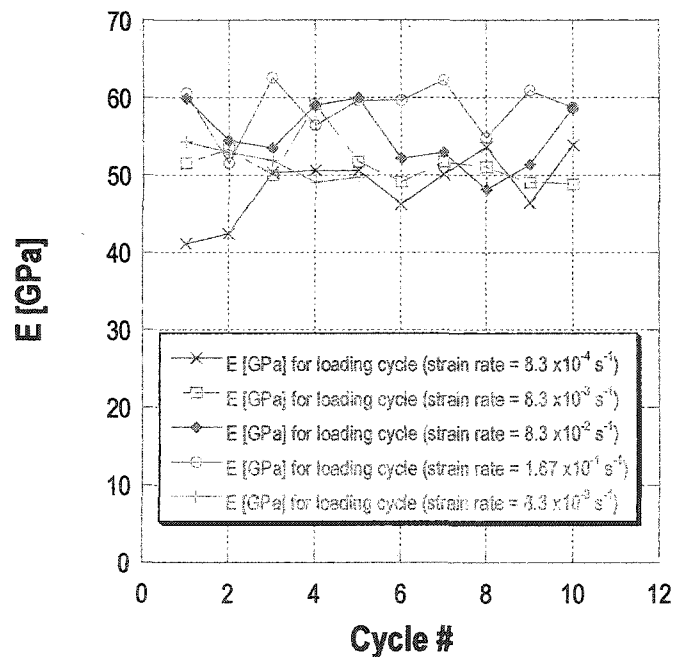


Figure 5-3: A plot of effective modulus vs. cycle # for all loading cycles at each strain rate for a 0.50 μm thick Al microtensile beam (specifically microtensile beam 0.5A-1L-5-7).

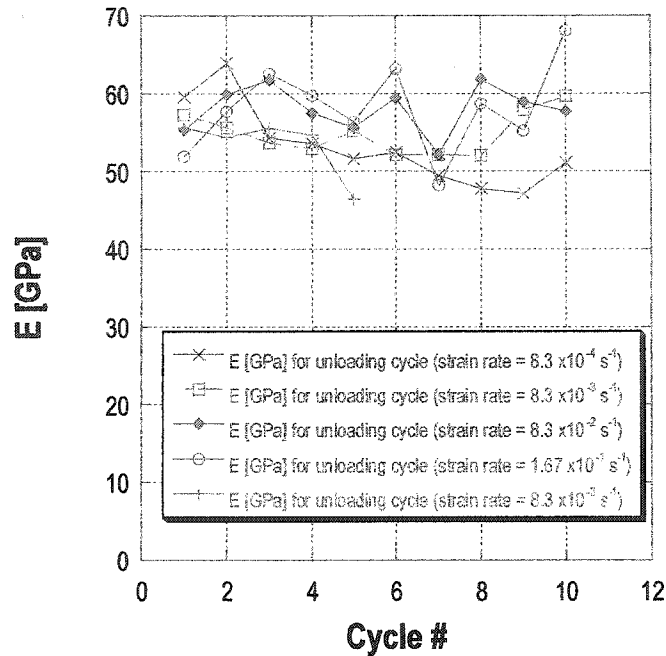


Figure 5-4: A plot of effective modulus vs. cycle # for all unloading cycles at each strain rate for a 0.50 μm thick Al microtensile beam (specifically microtensile beam 0.5A-1L-5-7).

5.1.1 Summary of the 1.00 μm thick Al microtensile beams

A different sequence of strain rates was performed for each of the three 1.00 μm thick microtensile beams. These three microtensile beams (1A-2L-1-6, 1A-2L-1-7, and 1A-2L-1-8) were located next to one another on the same wafer. Therefore, all three samples were subjected to the exact same deposition and fabrication process steps. As an example, the sequence of strain rates for microtensile beam 1A-2L-1-6 is outlined in Table 5-3; it was not low to high, but instead a random sequence. The 2nd strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ was repeated for the final 5 cycles to investigate a strain-history dependence. The effective modulus was measured for both the loading and unloading stress-strain curves at each strain rate for all three 1.00 μm thick microtensile beams. The average modulus for all 10 cycles (or 5 cycles for the

final strain rate) was determined and plotted against the log strain rate (Figure 5-5a); the strain rate sensitivity exponent was also determined (Figure 5-5b).

Table 5-3: Lists the sequence of strain rates, number of cycles, and the average effective modulus for microtensile beam 1A-2L-1-6 tested at a strain amplitude of 0.17%.

Strain rate [s ⁻¹]	Number of trapezoidal cycles performed at each strain rate	Average effective modulus, E [GPa]	
		loading modulus ± standard deviation	unloading modulus ± standard deviation
1.67 x10 ⁻¹	10	55.9 ± 8.3	59.6 ± 4.5
8.33 x10 ⁻²	10	59.4 ± 3.7	61.8 ± 6.3
8.33 x10 ⁻⁴	10	53.6 ± 4.8	59.7 ± 4.1
8.33 x10 ⁻³	10	58.2 ± 2.0	59.9 ± 2.1
3.33 x10 ⁻¹	10	N/A (not measured)	N/A (not measured)
8.33 x10 ⁻²	5	57.3 ± 5.0	60.7 ± 1.9

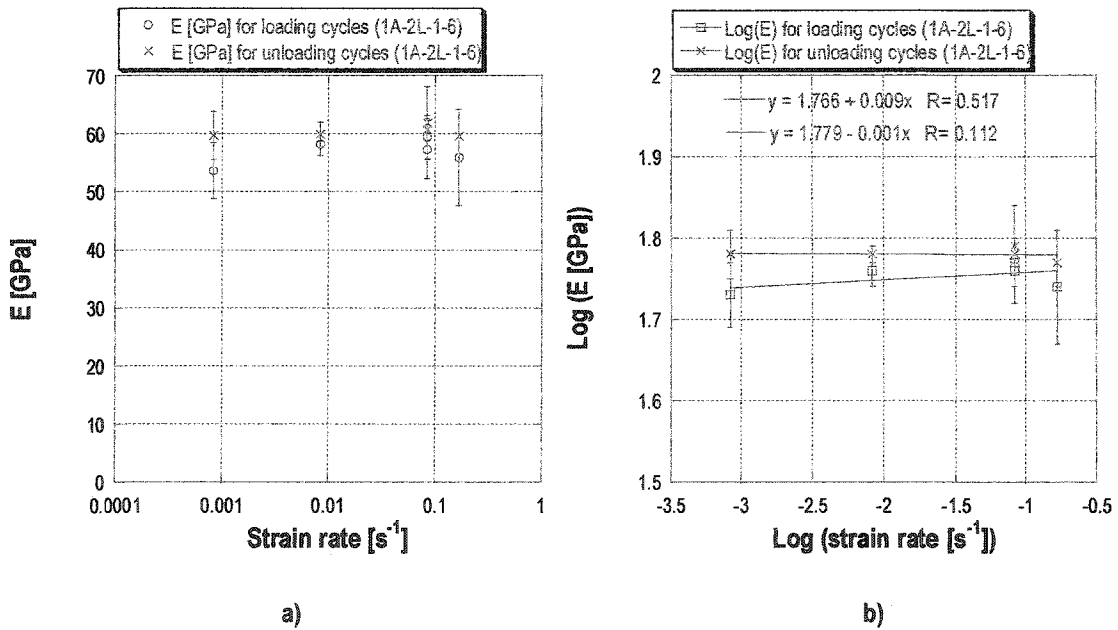


Figure 5-5: a) A plot of effective modulus vs. strain rate and b) a plot of the Log (E) vs. Log (strain rate) for both the loading and unloading cycles at each strain rate; this is for microtensile beam 1A-2L-1-6.

All three 1.00 μm thick microtensile beams show a similar behavior in that the effective modulus increases with increasing strain rate except at the highest strain rate (Figure 5-6). However, there is a large spread in the average effective modulus at each strain rate. Also, because the strain rate sequence varied for each microtensile beam it was difficult to establish a trend for the dependence of the effective modulus on the sequence of strain rates. It is interesting to note that even though these three microtensile beams were located next to one another on the same wafer, there is a substantial difference among their average effective modulus values. Additionally, no stress relaxation was observed during the hold at the maximum strain for all three 1.00 μm thick microtensile beams at all strain rates.

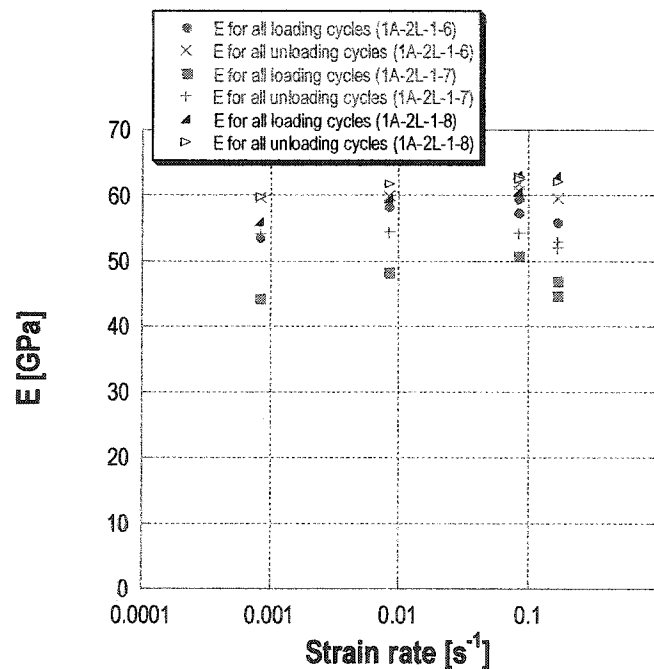


Figure 5-6: A plot of effective modulus vs. strain rate for both the loading and unloading cycles at each strain rate for all three 1.00 μm thick Al microtensile beams; error bars are not included for clarity.

5.1.2 Summary of the 0.75 μm thick Al microtensile beams

Similar to the 1.00 μm thick microtensile beams, the 0.75 μm thick microtensile beams showed no evidence of stress relaxation behavior during the hold at the maximum strain for all strain rates. Also, these two beams were located adjacent to one another on the same wafer yet produced substantially different values for the average effective modulus (Figure 5-7) and much lower than those for the 1.00 μm thick microtensile beams discussed in section 5.1.1. Both 0.75 μm thick microtensile beams were subjected to a different sequence of strain rates. Because the strain rate sequence was varied, it was difficult to establish a trend for the dependence of the effective modulus on the sequence of strain rates.

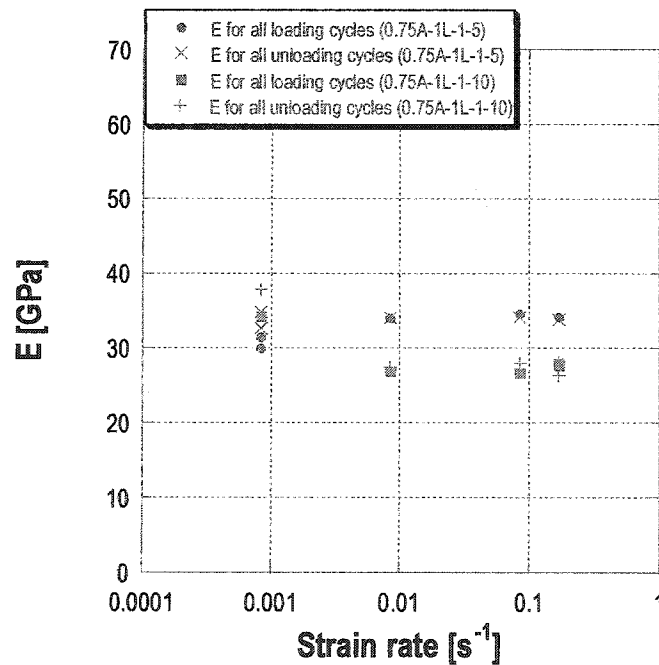


Figure 5-7: A plot of the effective modulus vs. strain rate for both the loading and unloading cycles at each strain rate for the two 0.75 μm thick Al microtensile beams; error bars are not included for clarity.

5.1.3 Summary of the 0.50 μm thick Al microtensile beams

The sequence of strain rates for microtensile beam 0.5A-1L-5-6 is outlined in Table 5-4. For all three 0.50 μm thick microtensile beams, the strain rates were performed in a low-to-high sequence with a repeat of the 2nd strain rate as the final strain rate. Similar to the 1.00 μm and 0.75 μm thick microtensile beams, these three 0.50 μm thick microtensile beams were also located next to one another on the same wafer.

Table 5-4: Lists the sequence of strain rates, number of cycles, and the average effective modulus for microtensile beam 0.5A-1L-5-6 tested at a strain amplitude of 0.17%.

Strain rate [s ⁻¹]	Number of trapezoidal cycles performed at each strain rate	Average effective modulus, E [GPa]	
		loading modulus $\pm$ standard deviation	unloading modulus $\pm$ standard deviation
8.33×10^{-4}	9	52.5 ± 6.6	53.3 ± 4.8
8.33×10^{-3}	10	56.6 ± 1.9	58.8 ± 1.3
8.33×10^{-2}	10	59.3 ± 1.4	59.9 ± 2.2
1.67×10^{-1}	10	60.8 ± 3.3	59.7 ± 4.2
3.33×10^{-1}	10	N/A (not measured)	N/A (not measured)
8.33×10^{-3}	5	56.9 ± 2.3	60.0 ± 1.9

The effective modulus was measured for both the loading and unloading stress-strain curves at each strain rate. The average modulus for all 10 cycles (or 5 cycles for the final strain rate) was determined and plotted against the log strain rate (Figure 5-8a). For microtensile beam 0.5A-1L-5-6, the strain rate sensitivity exponent on loading and unloading was 0.024 and 0.020, respectively (Figure 5-8b).

All three 0.50 μm thick microtensile beams were tested using the same sequence of strain rates. In addition, these microtensile beams show a higher strain rate sensitivity upon unloading

than both the 1.00 μm and 0.75 μm thick microtensile beams. The spread among the values for the average effective modulus is much less in the 0.50 μm thick microtensile beams than for the other thicknesses (Figure 5-9). Also, no stress relaxation behavior was observed during the hold at the maximum strain for all strain rates.

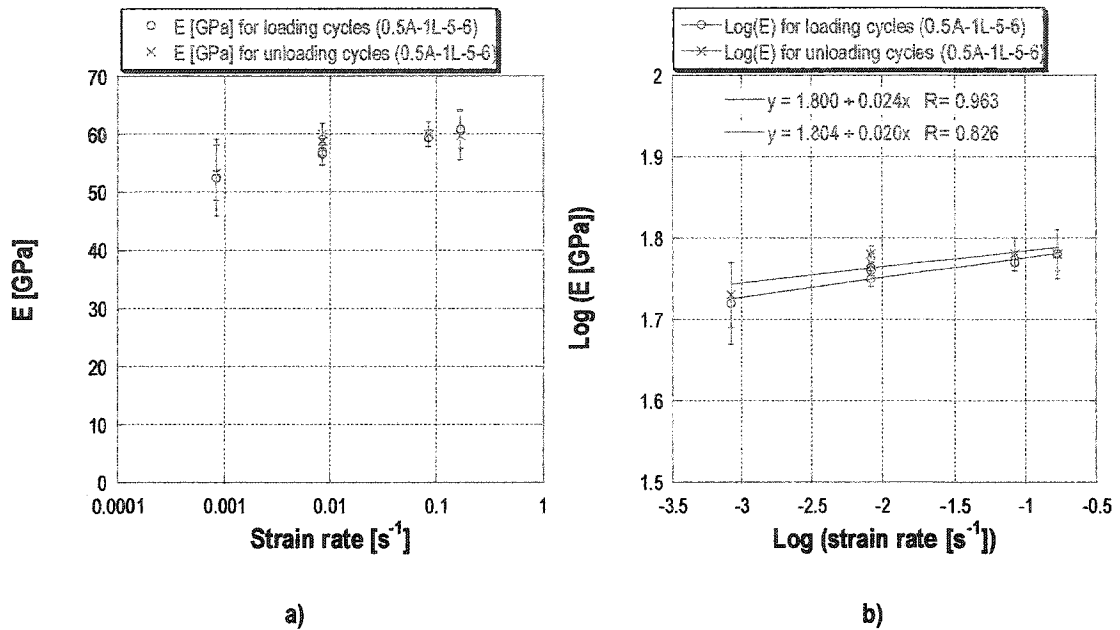


Figure 5-8: a) A plot of effective modulus vs. strain rate and b) a plot of the $\text{Log}(E)$ vs. $\text{Log}(\text{strain rate})$ for both the loading and unloading cycles at each strain rate; this is for microtensile beam 0.5A-1L-5-6.

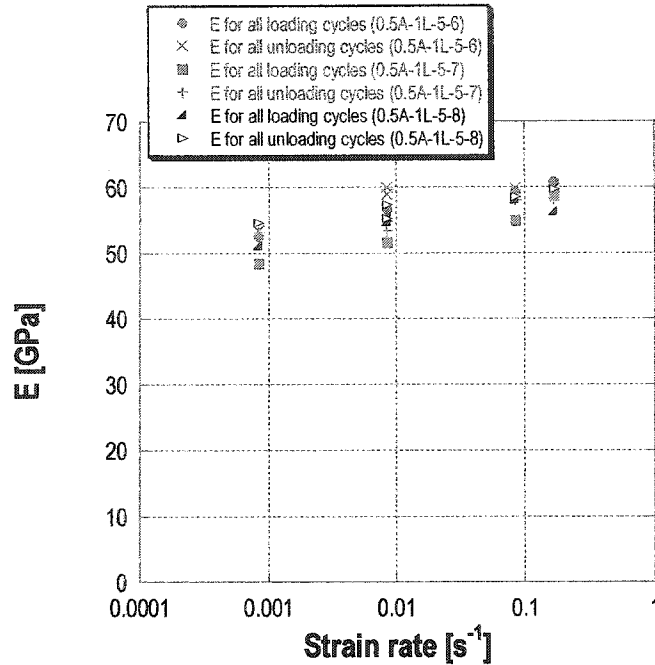


Figure 5-9: A plot of effective modulus vs. strain rate for both the loading and unloading cycles at each strain rate for all three 0.50 μm thick Al microtensile beams; error bars are not included for clarity.

5.1.4 Summary of the 0.25 μm thick Al microtensile beams

The order of strain rates for microtensile beam 0.25A-1L-1-1 were performed in a low-to-high sequence with a repeat of the 2nd strain rate as the final strain rate. A least-squares fit was carried out on the complete loading and unloading curves for microtensile beam 0.25A-1L-1-1 because they are linear up to failure. The effective modulus was measured for both the loading and unloading stress-strain curves at each strain rate. The average modulus for all 10 cycles (or 5 cycles for the final strain rate) was determined and plotted against the log strain rate (Figure 5-10a). Only one 0.25 μm thick microtensile beam was analyzed at the 0.17% strain amplitude; the results from the two other 0.25 μm thick microtensile beams tested were very noisy. Hence, it is difficult to say anything about a trend for these microtensile beams.

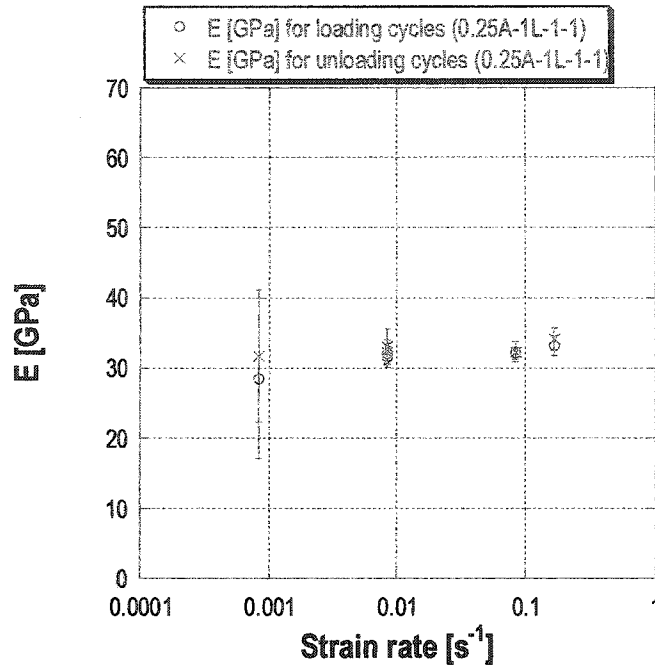


Figure 5-10: A plot of effective modulus vs. strain rate and for both the loading and unloading cycles at each strain rate; this is for microtensile beam 0.25A-1L-1-1.

5.1.5 Summary of the strain rate sensitivity experiments

Attention has been concentrated on the 0.50 μm thick microtensile beams because of their consistent behavior. Strain rate sensitivity exponents upon loading and unloading for only the 0.50 μm thick films are outlined in Table 5-5. For the 0.50 μm thick microtensile beams, the strain rate sensitivity exponent upon unloading is the same independent of the strain rate sensitivity exponent upon loading. The strain rate sensitivity exponents for the other film thicknesses were not included for two reasons: 1) the linear fit on the plot of Log (E) vs. Log (strain rate) was poor, and 2) a large spread of effective modulus values were measured for adjacent films on the same wafer. There seems to be major differences in film quality for the four thicknesses.

Table 5-5: Lists the strain rate sensitivity exponent upon loading and unloading for the 0.50 μm thick microtensile beams tested at a strain amplitude of 0.17%.

Film thickness [μm]	Microtensile beam	Strain rate sensitivity exponent	
		Loading cycles	Unloading cycles
0.50	0.5A-1L-5-6	0.024	0.020
	0.5A-1L-5-7	0.036	0.019
	0.5A-1L-5-8	0.018	0.021

Overall, the 1.00 μm and 0.50 μm thick microtensile beams had a higher effective modulus than the 0.75 μm and 0.25 μm thick microtensile beams (Figure 5-11). The effective modulus for the 1.00 μm and 0.50 μm microtensile beams is around 55 GPa; in contrast, the 0.75 μm and 0.25 μm thick microtensile beams have an effective modulus around 30 GPa. However, the grain size (approximately 300 nm) of the 1.00 μm and 0.50 μm thick microtensile beams are similar while the grain size (approximately 400 nm) of the 0.75 μm and 0.25 μm thick microtensile beams are larger but also similar to one another. The effective modulus is suggested to be dependent on grain size and independent of film thickness; it is also not clear how impurities impact the observed mechanical behavior. However, XPS depth profiling suggests that all of these films have some level of impurities; N, O, C, or some combination (Figure 5-12). Therefore, taking grain size as the major factor influencing the effective modulus, it is suggested that a rapid relaxation mechanism is occurring in the 0.75 μm and 0.25 μm thick microtensile beams during loading resulting in the low effective modulus, and that the relaxation mechanism for the 1.00 μm and 0.50 μm thick microtensile beams may be influenced by impurities lying along the grain boundaries.

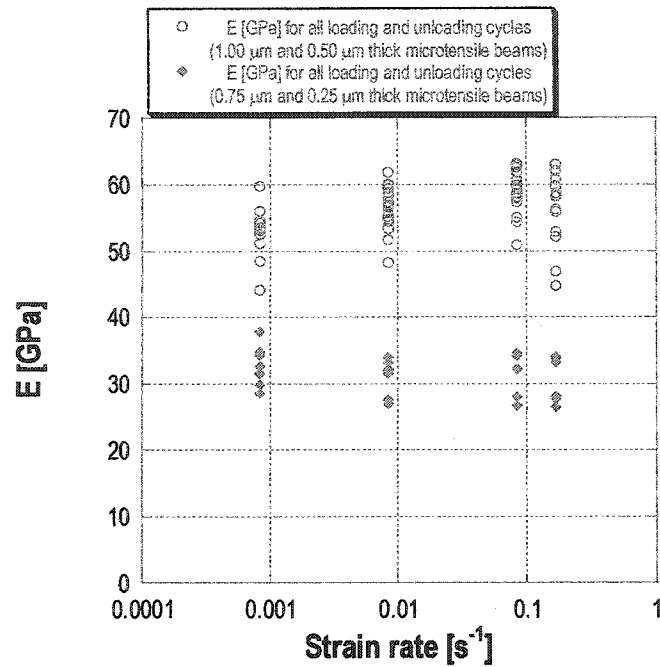


Figure 5-11: A plot of effective modulus vs. strain rate showing the average values upon loading and unloading for all nine microtensile beams.

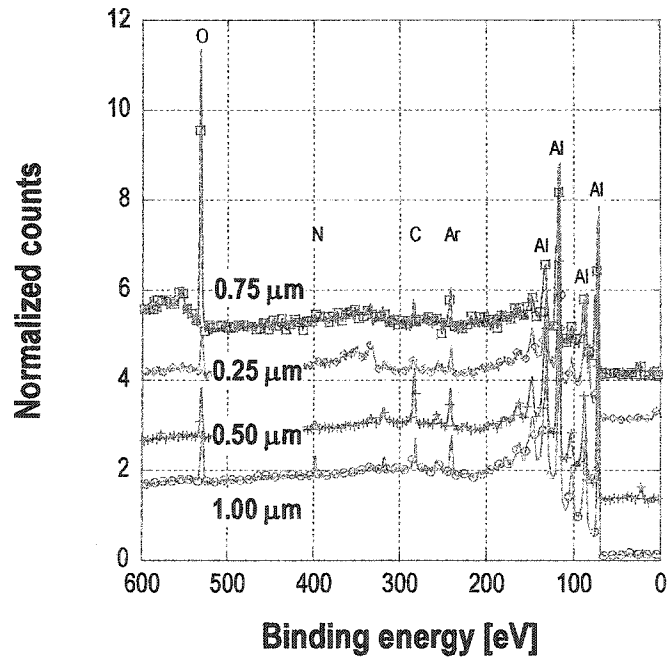


Figure 5-12: A plot of normalized counts vs. binding energy showing the results of the XPS depth profiling for all four microtensile beam thicknesses.

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These experiments show that even under seemingly purely elastic conditions, some of the microtensile beams exhibit a strain rate sensitivity. The strain rate sensitivity in the 0.50 μm thick microtensile beams is more consistent than for the other film thicknesses. At higher strain rates, less time is allowed for the anelastic mechanisms to relax thereby resulting in a higher effective modulus, and vice versa for low strain rates. Because the thicknesses are different and the grain sizes (and grain geometry) are similar (within each of the two sets; 1.00 μm , 0.50 μm ; and 0.75 μm , 0.25 μm), it suggests that the smaller grained films do not have the rapid relaxation mechanism that is present in the larger grained films. Because stress relaxation was not observed for any film thicknesses at all strain rates during the hold period at the maximum strain amplitude of 0.17%, the next set of experiments was performed to investigate the microtensile beams for stress relaxation behavior.

5.2 Stress relaxation experiments

A strain amplitude of 0.43% was used to investigate a transition region between the 0.17% strain amplitude and failure. Also, as in the 0.17% strain amplitude experiments a trapezoidal waveform was used. The strain rate was set at $8.33 \times 10^{-2} \text{ s}^{-1}$ for all experiments. The loading and unloading time is approximately 0.055 seconds and the hold time at the maximum strain is approximately 0.233 seconds making the complete time for the cycle 0.343 seconds. Four microtensile beams (one 0.75 μm and three 1.00 μm) were tested; they were 0.75A-1L-1-10, 1A-2L-1-8, 1A-2L-1-6, and 1A-2L-1-7. A gage length of 600 μm was used when calculating the strain.

5.2.1 Microtensile beam 0.75A-1L-1-10

Four cycles were performed to a strain amplitude of 0.43% followed by a strain-to-failure cycle. However, the data for the 1st cycle was lost, so only the data from 2nd, 3rd, and 4th cycles are shown in Figure 5-13. The effective modulus was approximately 36 GPa for all three cycles showing an excellent consistency among consecutive runs. Furthermore, very little stress relaxation was observed during the hold time (Figure 5-14). These results agree with the idea that a rapid relaxation mechanism is present during loading causing a lower effective modulus; therefore, when the hold occurs, the driving force for relaxation may have already been exhausted.

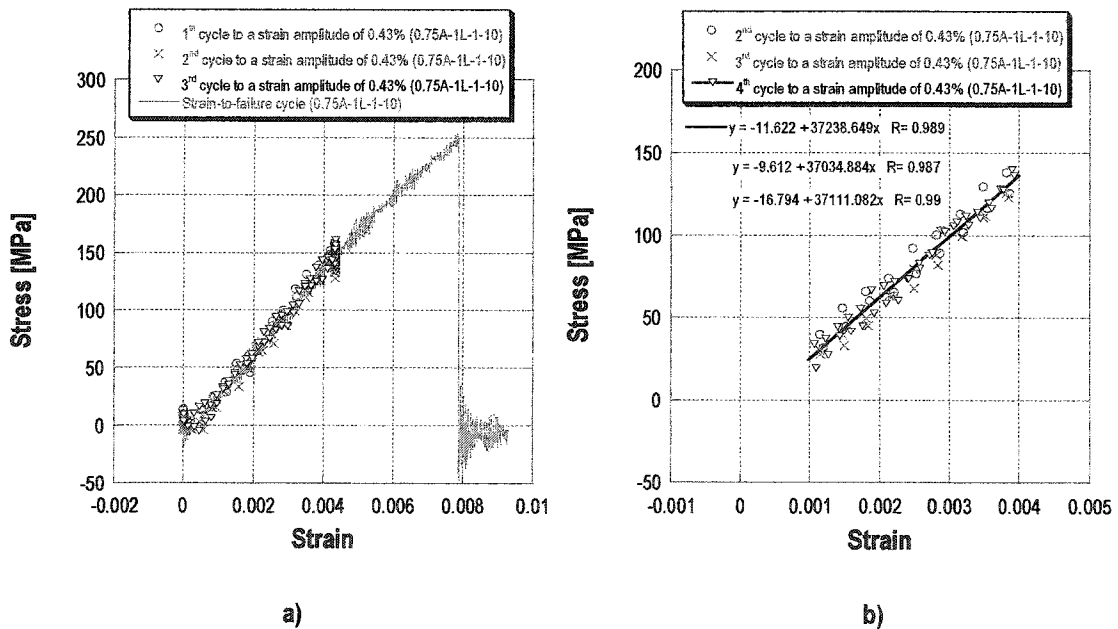


Figure 5-13: a) The stress-strain plot showing the loading and unloading cycles and b) showing the effective modulus for the three cycles to be approximately 36 GPa for microtensile beam 0.75A-1L-1-10.

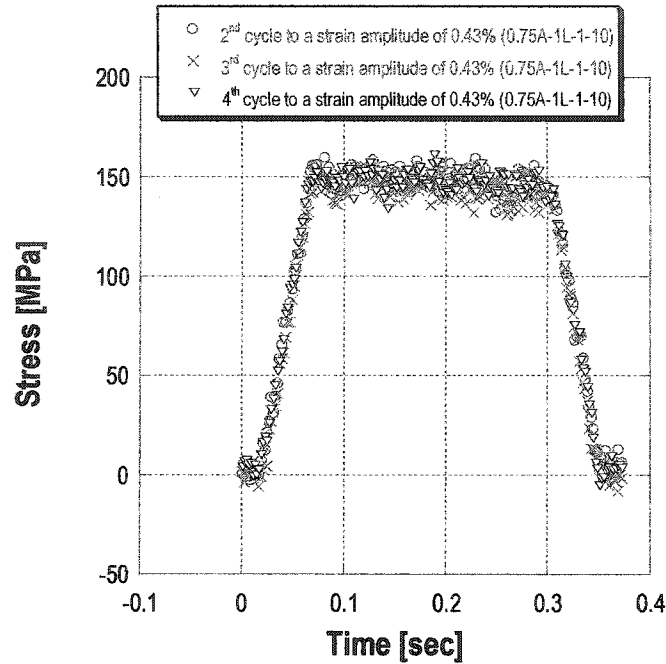


Figure 5-14: A plot of stress vs. time showing the absence of stress relaxation behavior in all three cycles for microtensile beam 0.75A-1L-1-10.

5.2.2 Microtensile beam 1A-2L-1-6

One cycle followed by a strain-to-failure cycle was performed. Both cycles are shown in Figure 5-15a. The effective modulus was approximately 59 GPa for both cycles showing an excellent consistency. Furthermore, a measurable amount of stress relaxation was observed during the hold time (Figure 5-15b). These results agree with the higher effective modulus measured for the 1.00 μm thick microtensile beams. During loading the relaxation mechanism is not completely exhausted; therefore stress relaxation is observed during the hold.

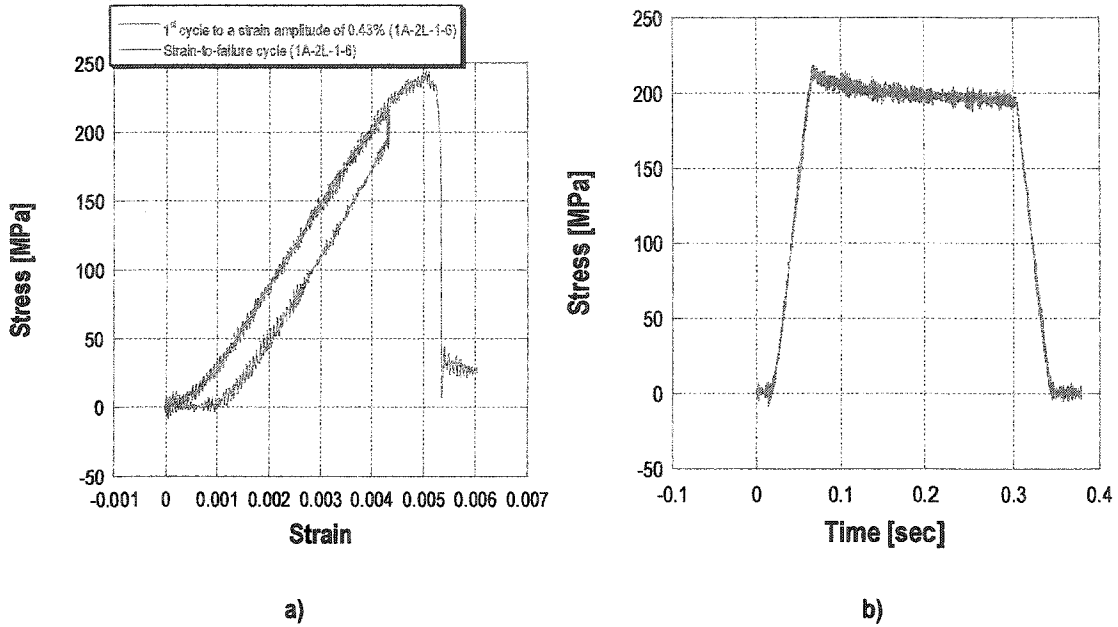


Figure 5-15: a) The stress-strain plot showing both the 1st cycle and the strain-to-failure curve, and b) showing the stress relaxation during the hold in the 1st cycle for microtensile beam 1A-2L-1-6.

In order to derive a time constant for the relaxation mechanism, the stress relaxation portion of the curve during the hold was fit using a double exponential decay (Equation 5-1), as shown in Figure 5-16. The double exponential decay describes a standard anelastic model containing two Maxwell components (Figure 1-2). The time constants, τ_1 and τ_2 , were determined to be 0.014 seconds and 0.147 seconds, respectively. In comparison to the 0.75 μm thick Al microtensile beam, the 1.00 μm thick microtensile beam experiences a slower relaxation process indicated by the higher effective modulus and the observed stress relaxation during the holding period at maximum strain.

$$\sigma(t) = \sigma_{\infty} + \Delta\sigma_1 * \left(\exp\left(\frac{-t}{\tau_1}\right) \right) + \Delta\sigma_2 * \left(\exp\left(\frac{-t}{\tau_2}\right) \right)$$

Equation 5-1: A double exponential decay used to model the stress relaxation behavior observed in freestanding Al thin films.

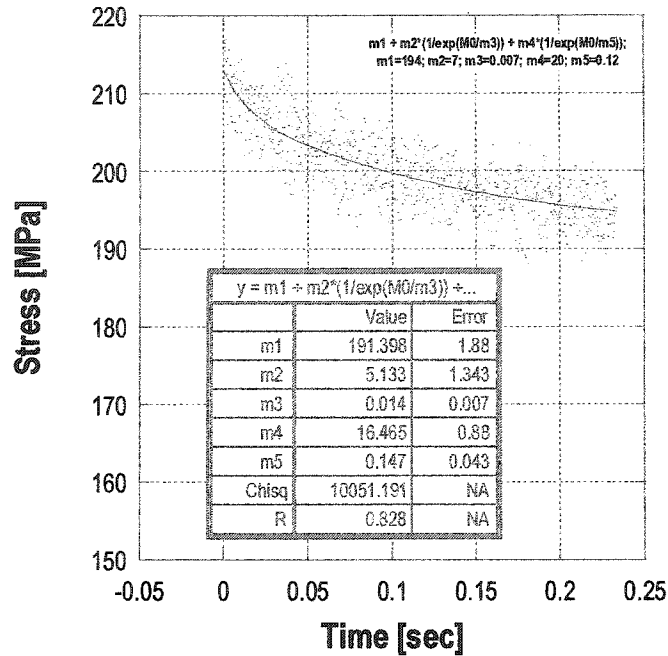


Figure 5-16: : A plot of stress vs. time showing the stress relaxation observed during the hold time for microtensile beam 1A-2L-1-6.

5.2.3 Microtensile beam 1A-2L-1-7

One cycle followed by a strain-to-failure cycle was performed. Both cycles are shown in Figure 5-17a. The effective modulus was approximately 56 GPa and 60 GPa for the 1st cycle and the strain-to-failure cycle, respectively. Between the two cycles, some recovery of the anelastic mechanisms occurred, but the recovery was not complete. Furthermore, a measurable amount of stress relaxation was observed during the hold time (Figure 5-17b). These results agree with the higher effective modulus measured for the 1.00 μm thick microtensile beams. During loading the relaxation mechanism is not completely exhausted; therefore stress relaxation is observed during the hold.

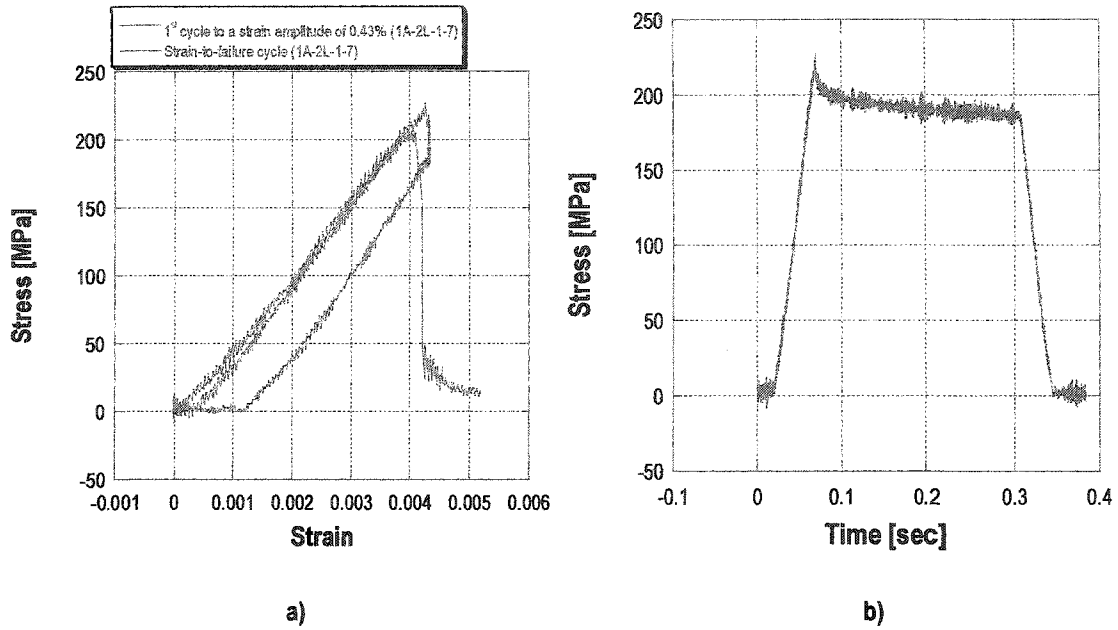


Figure 5-17: a) The stress-strain plot showing both the 1st cycle and the strain-to-failure curve, and b) showing the stress relaxation during the hold in the 1st cycle for microtensile beam 1A-2L-1-7.

In order to derive a time constant for the relaxation mechanism, the stress relaxation portion of the curve during the hold was fit using a double exponential decay, as shown in Figure 5-18. The time constants, τ_1 and τ_2 , were determined to be 0.004 seconds and 0.118 seconds, respectively.

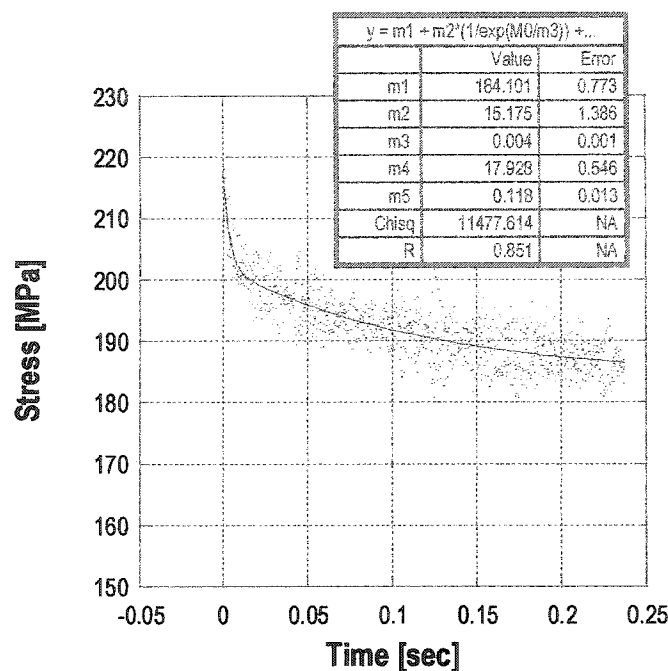


Figure 5-18: A plot of stress vs. time showing the stress relaxation observed during the hold time for microtensile beam 1A-2L-1-7.

5.2.4 Microtensile beam 1A-2L-1-8

Two cycles followed by a strain-to-failure cycle were performed. The three cycles are shown in Figure 5-19a. The effective modulus was approximately 61 GPa for all three cycles. A measurable amount of stress relaxation was observed during the hold time for both cycles (Figure 5-19b). As in the previous 1.00 μm thick microtensile beams, during loading the relaxation mechanism is not completely exhausted; therefore stress relaxation is observed during the hold.

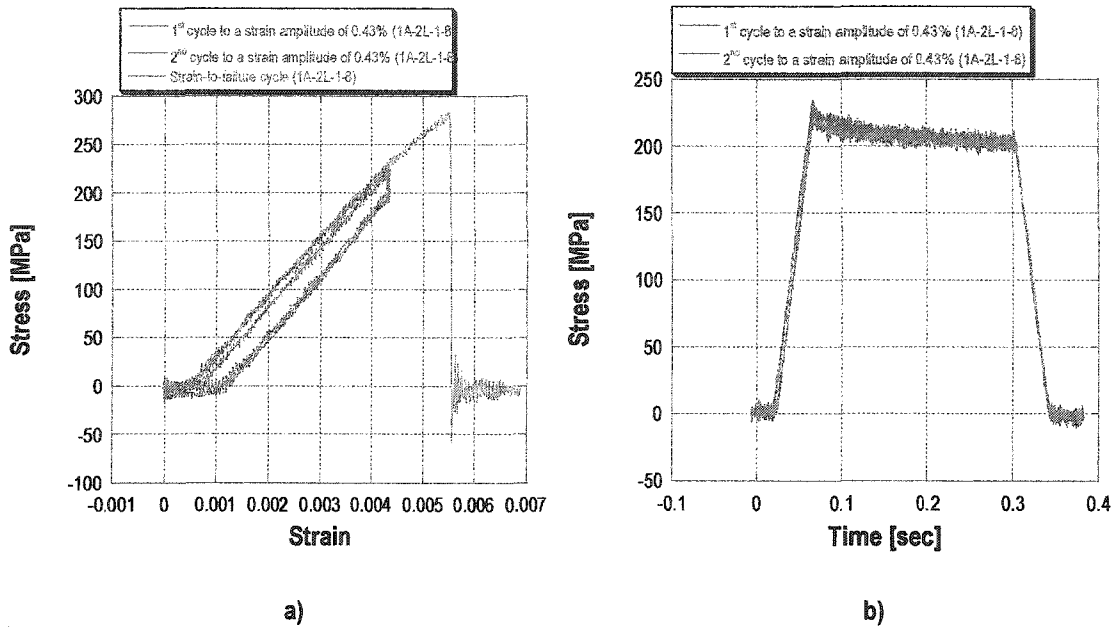


Figure 5-19: a) The stress-strain plot for two cycles to a strain amplitude of 0.43% and the strain-to-failure cycle, and b) a stress vs. time plot showing the stress relaxation occurring at the 0.43% strain.

In order to derive a time constant for the relaxation mechanism, the stress relaxation portion of the curve during the hold was fit using a double exponential decay, as shown in Figure 5-20. The time constants, τ_1 and τ_2 , were determined to be 0.011 seconds and 0.135 seconds for the 1st cycle and 0.017 and 0.499 seconds for the 2nd cycle. The time between cycles (i.e. “rest” or recovery time) was less than one second which may provide an explanation as to why 2nd cycle has a lower peak stress than the 1st.

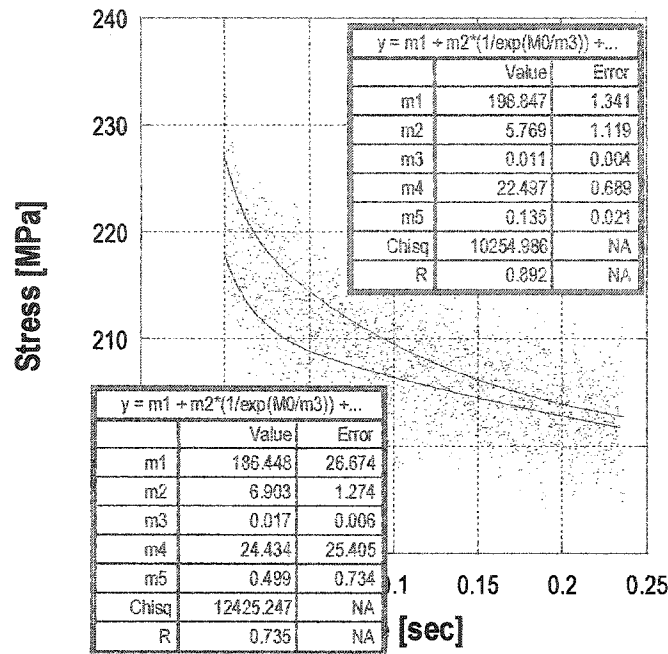


Figure 5-20: A plot of stress vs. time showing the stress relaxation curves and the fits with the appropriate time constants for microtensile beam 1A-2L-1-8.

5.2.5 Summary of the stress relaxation experiments

At a strain amplitude of 0.43% (also a higher stress level), the effective moduli are very similar to the values measured at a 0.17% strain amplitude; however, the 1.00 μm thick microtensile beams show measurable stress relaxation while the 0.75 μm thick microtensile beams do not. This result combined with a lower effective modulus suggests that the particular anelastic mechanisms operating in the 0.75 μm thick microtensile beams at these time scales activate at lower stresses and provide a faster stress relaxation process. These results imply that a smaller grain size and higher maximum stress level give rise to stress relaxation behavior during the hold periods at maximum strain. The 1.00 μm thick microtensile beams show stress relaxation during the hold at the higher strain amplitude of 0.43%, while the 0.75 μm thick

microtensile beams do not; thereby intimating that specific anelastic mechanisms may become active at certain higher stress levels in the 1.00 μm thick microtensile beams and not in the 0.75 μm thick microtensile beams.

5.3 *Strain-to-failure experiments*

Many microtensile beams of all four thicknesses were strained to failure at a number of strain rates. Some were strained to failure after performing many cycles while others having no prior history were also strained to failure. The 1.00 μm , 0.75 μm , and 0.25 μm thick microtensile beams show very brittle behavior upon failure and also show no strain rate sensitivity (Figure 5-21, Figure 5-22, and Figure 5-23). No trends are present for these three film thicknesses. On the other hand, the 0.50 μm thick microtensile beams exhibit a large amount of ductility and display a definitive strain rate sensitivity (Figure 5-24).

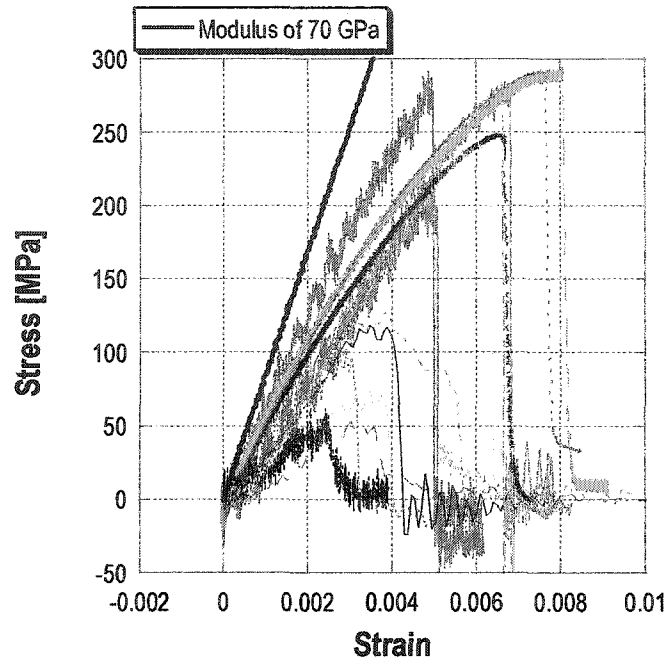


Figure 5-21: The stress-strain plot showing the strain-to-failure curves for all the 1.00 μm thick Al microtensile beams.

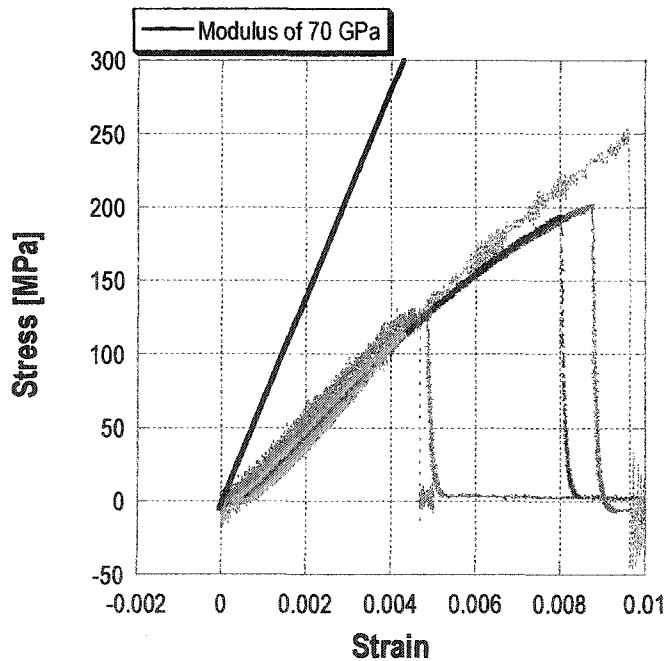


Figure 5-22: The stress-strain plot showing the strain-to-failure curves for all the 0.75 μm thick Al microtensile beams.

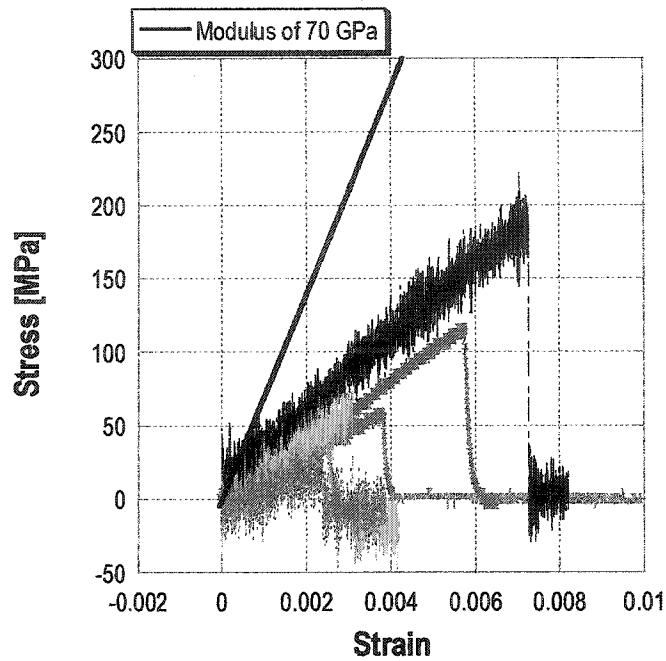


Figure 5-23: The stress-strain plot showing the strain-to-failure curves for all the 0.25 μm thick Al microtensile beams.

5.3.1 0.50 μm thick Al microtensile beams

The 0.50 μm thick microtensile beams can be strained to 2%, 3%, and 5% without failure. Also, a strain rate sensitivity is displayed in that the yield stress and ultimate tensile strength increase with increasing strain rate. Also, the failure strain decreases with increasing strain rate.

Table 5-6: Lists the results for the 0.50 μm thick microtensile beams strained to failure.

Strain rate [s ⁻¹]	Effective modulus [GPa]	Yield stress σ_{YS} [MPa]	Tensile strength σ_{UTS} [MPa]	Failure strain ε_f	Failure stress σ_f [MPa]
1.67 x10 ⁻⁵	67 (unloading)	134	200	> 3%	N/A
1.67 x10 ⁻⁵	63 (unloading)	115	210	> 5%	N/A
1.67 x10 ⁻⁵	68 (unloading)	126	N/A	N/A	N/A
6.67 x10 ⁻⁵	67 (unloading)	161	250	> 2%	N/A
8.33 x10 ⁻³	55 (loading)	280	410	0.12%	410
8.33 x10 ⁻³	54 (loading)	280	410	0.12%	410
8.33 x10 ⁻³	55 (loading)	310	410	0.12%	410

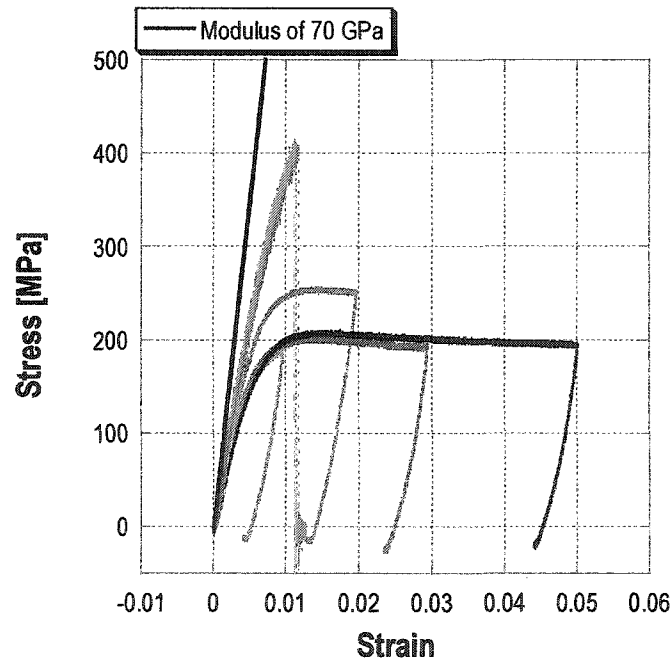


Figure 5-24: The stress-strain plot showing the strain-to-failure curves for all the 0.50 μm thick Al microtensile beams.

5.3.2 Summary of the strain-to-failure experiments

From these results, it was concluded that the 0.50 μm thick microtensile beams were behaving consistently and reliably. They demonstrated a higher ductility (i.e. they can be strained to 2%, 3%, and 5% without failure) at low strain rates than the three other film thicknesses. Also, for the other film thicknesses, the failure strain is only about 0.8% and does not depend on strain rate. The lower ductility may arise from microstructural or impurity effects that also may affect the stress relaxation behavior. Furthermore, anelastic mechanisms in the 0.75 μm and 0.25 μm thick films may require a higher stress level for activation. Because the 0.50 μm thick microtensile beams exhibited the most consistent and reliable behavior, the experiments discussed in Chapter 6 were designed specifically for the 0.50 μm thick microtensile beams.

5.4 *Microtensile superbams*

A final set of two 1.00 μm thick Al films provide extraordinary contrast in the behavior of microtensile beams made from them; the wafers are #8 and #9. To the best of our ability, these wafers were produced in exactly the same manner. The deposited Al was from the same source, the deposition process was the same (i.e. evaporation rate, base pressure, etc.), and the chemical baths and procedures used in the microtensile beam fabrication process were the same. However, the wafers gave completely different results from each other and from any previous films studied.

5.4.1 Microtensile beam 1A-3L-8-7

Anelastic recovery experiments were performed similar to those covered in Chapter 6 section 6.2.3. However, a completely different result was observed. The behavior in this microtensile beam was completely plastic upon the 1st cycle. The remaining cycles start picking up load from the plastic strain point of the 1st cycle (Figure 5-25). No anelastic recovery was observed at any of the rest times (1 hour was the longest rest time). No satisfactory explanation for this behavior exists, yet.

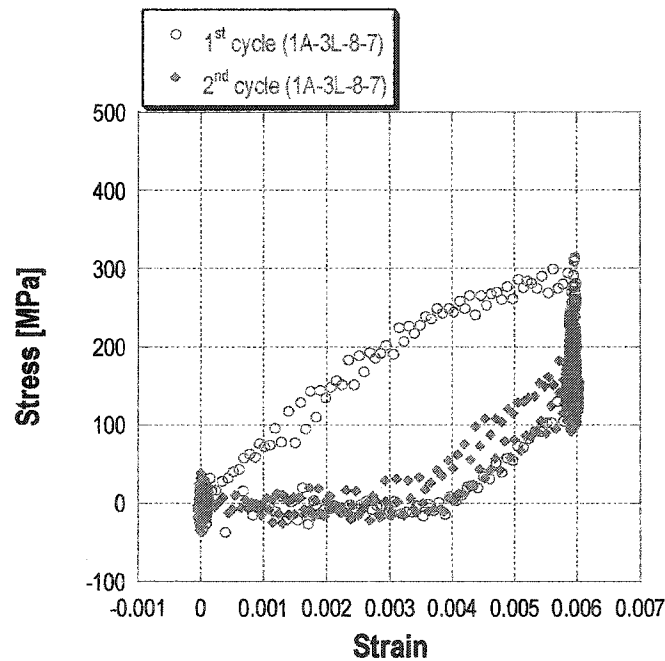


Figure 5-25: The stress-strain plot for the 1st and 2nd cycles at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ for a $1.00 \text{ }\mu\text{m}$ thick Al microtensile beam.

5.4.2 Microtensile beam 1A-3L-9-6, 1A-3L-9-10, and 1A-3L-9-7

In contrast, microtensile beam 1A-3L-9-6 is completely elastic for all cycles (Figure 5-26). This microtensile beam was nicknamed a microtensile superbeam. No relaxation was observed

and a modulus of 70 GPa was measured for each of the 16 cycles and the strain-to-failure cycle (Figure 5-27). Furthermore, a yield stress of 700 MPa was calculated. This yield stress is higher than that for any previously reported pure Al material and even than most bulk aluminum alloys.⁶⁴ The mechanistic reasons behind this result are currently being investigated. Even though microtensile beams 1A-3L-8-7 and 1A-3L-9-6 were deposited and processed in what were thought to be identical ways, and experienced the same experimental test conditions, they behaved extremely differently.

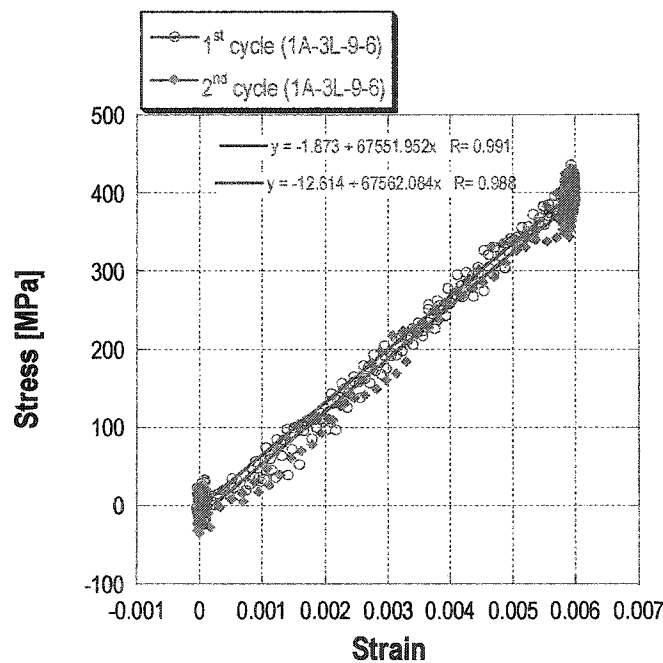


Figure 5-26: The stress-strain plot for the 1st and 2nd cycles at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ for microtensile beam 1A-3L-9-6.

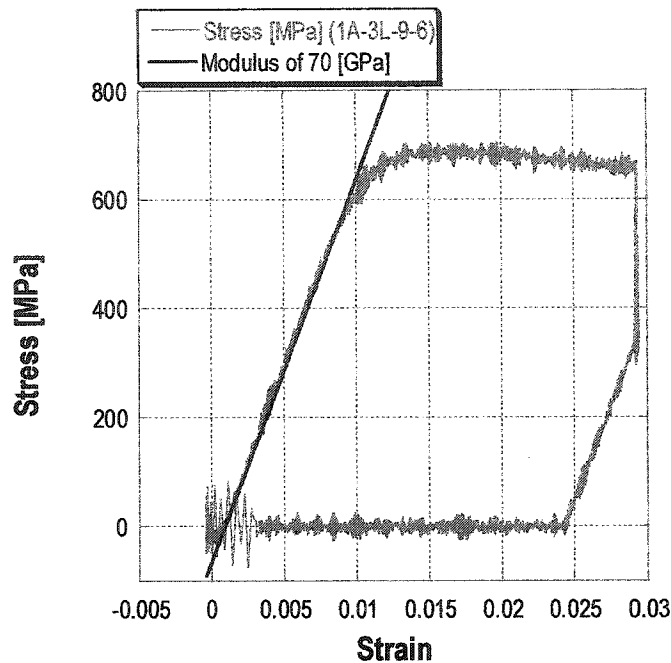


Figure 5-27: The stress-strain plot for the strain-to-failure experiment performed on microtensile beam 1A-3L-9-6 at a strain rate of $8.20 \times 10^{-2} \text{ s}^{-1}$; note the excellent agreement with the modulus of the bulk material.

Because it was suggested that this previous result may have been an anomaly, a second microtensile beam (1A-3L-9-10) from the same wafer #9 was strained to failure without performing any prior cycles. Both microtensile beams from wafer #9 behaved in a similar manner with a modulus of 70 GPa and a yield stress around 700 MPa (Figure 5-28). Additionally, both microtensile beams experienced about 3% strain without breaking. The ductility present in these microtensile beams can be compared to that observed in the $0.50 \mu\text{m}$ thick microtensile beams. However, even though these $1.00 \mu\text{m}$ thick microtensile superbeams and the previously discussed $0.50 \mu\text{m}$ thick microtensile beams are ductile, they do not display similar yield stresses nor do they have a similar relaxation behavior.

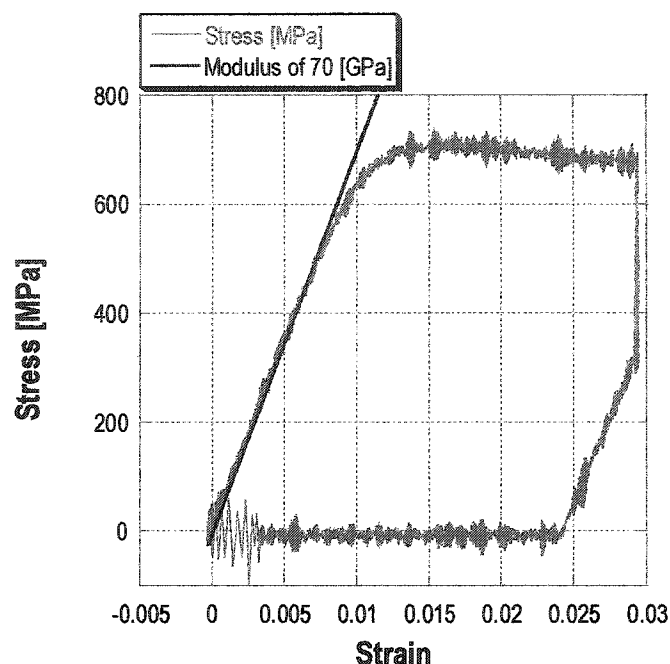


Figure 5-28: The stress-strain plot for the strain-to-failure experiment performed on microtensile beam 1A-3L-9-10 at a strain rate of $8.20 \times 10^{-2} \text{ s}^{-1}$; note the excellent agreement with the modulus of the bulk material.

A third microtensile beam from wafer #9 (1A-3L-9-7) was tested in a similar manner to that for the activation energy experiments covered in Chapter 6 section 6.2.4. No relaxation was observed at any of the five strain amplitudes at any of the four temperatures. The modulus was measured as 70 GPa for all four temperatures. Only XPS has been performed to characterize these microtensile beams; results show that they are indeed very pure Al and contain an immeasurable amount of impurities (Figure 5-29).

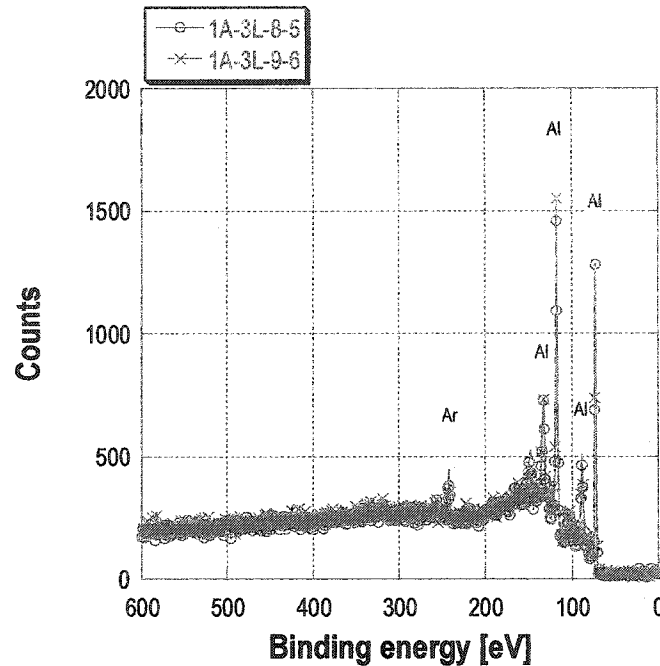


Figure 5-29: A plot of counts vs. binding energy showing the results of the XPS profile at a depth of 400nm from the surface for the 1.00 μm thick Al microtensile beams fabricated from wafers #8 and #9.

5.5 Summary of the microtensile beam quality effects

These results provide an extraordinary account for how the film quality affects the mechanical properties. Even though the microtensile beams were produced in a seemingly identical manner, they displayed incredibly different mechanical behavior. Clearly, impurity content is a factor, but not the only factor. The superbams were indeed purer than the other microtensile beams. But, this is probably not the only parameter that gives rise to their remarkable properties. Also, the location of impurities within the microstructure most likely impacts the mechanical behavior. Impurities sitting at the grain boundaries will surely lead to a different behavior than those located within the grain. To understand reasons for this behavior, a detailed characterization analysis (i.e. texture, grain size, etc.) is necessary. However,

because of the consistent and reliable behavior of the 0.50 μm thick microtensile beams, supplementary experiments can be performed to better understand their anelastic properties.

6 Anelasticity experiments, results, and discussion

The strain rate sensitivity exponent provides one measure of the anelastic processes occurring in the samples during straining events. Thus, preliminary strain rate sensitivity experiments carried out on commercial (Goodfellow Co.) 99.99% pure 10 μm diameter Al and Cu wires were performed on microtensile system #1. These experiments are presented first. Stress relaxation, strain rate sensitivity, anelastic recovery, and activation energy experiments performed on freestanding Al microtensile beams fabricated at Lehigh University and carried out using microtensile system #4 comprise the second part of this chapter. Four thicknesses (1 μm , 0.75 μm , 0.50 μm , and 0.25 μm) of Al thin films were tested using microtensile system #4 as noted in Chapter 5. However, only experiments for the 0.50 μm thick microtensile beams will be included in this section because they seem to be “higher quality” films than those of the other thicknesses. Furthermore, microtensile system #4 was enclosed in a temperature-controlled chamber. With this experimental setup, an activation energy for a stress relaxation mechanism operating over the time scale of 0.5 seconds was determined.

6.1 10 μm diameter Al and Cu wire experiments (microtensile system #1)

These experiments were developed to examine how grain structure, dislocation density, and geometry affect anelastic behavior, or more specifically the strain rate sensitivity of the modulus. Al and Cu wires 10 μm in diameter and 99.99 % pure were heat treated in vacuum and tested under uniaxial tension. To study a geometry effect, the Al wires were annealed in vacuum at 350°C and compared to previous results obtained from 99.99% pure Al thin films

with a similar grain size.^{37,39} Plotting the measured elastic modulus (i.e. the effective modulus) against strain rate illustrated the strain rate sensitivity of the wires.

6.1.1 Experimental setup

Micro-wire test specimens were prepared in one of four categories; as received, annealed at 150°C, and annealed at 350°C. The wires were annealed in a vacuum system at a base pressure below 5×10^{-7} torr. For annealing method A, one end of the wire was pinned between two glass slides while the larger remaining segment of the wire was freestanding. The two glass slides were then clamped onto a 2 cm thick Inconel™ plate. In the vacuum system, the Inconel™ plate was located below a quartz heating element. The wires annealed with method B were pinned between the metal plate and one glass slide. Because the heating was through conduction, the wires in contact with the metal plate received the greatest amount of heat and were expected to grow larger grains than the freestanding wires.

After removing the wires from the vacuum system, they were mounted with Krazy Glue® onto Si frames (not containing microtensile beams) similar to those used in previous experiments.^{4,37,39} The wire was glued directly across the gap where the microtensile beam would have been, therefore making the gage length of the wire approximately 600 μm . Following clamping and cutting of the Si frame, the wire was visually inspected using the stereomicroscope to determine if it was intact (and if buckled or taut) or broken. If the wire was buckled, the piezo translator was retracted until the wire was visibly taut, or until a small load was registered. If the wire was taut, the piezo translator was extended until the load leveled off at zero. The stress on the wire was calculated by dividing the measured load by the starting cross-sectional area (i.e. for 10 μm diameter wires, the cross-sectional area was 78.5 μm^2 , or

78.5 x10⁻¹² m²). For microtensile system #1, strain was determined by dividing the grip displacement by the gage length of the wire, or 600 μm. Although this type of strain measurement was not directly measuring sample strain, it did improve the accuracy of the strain measurement compared to an earlier study without loss of high strain-rate capability.³⁷ Three strain rates were performed in a random sequence on the as-received wires and the 150°C (thermal treatment A) annealed wires; six strain rates were performed on the 150°C (thermal treatment B) and 350°C (thermal treatment B) annealed wires (Table 6-1). By applying the various strain rates in a random sequence, the dependence of elastic moduli on strain rate history was removed.

Table 6-1: Strain rates performed on the Al and Cu micro-wires for their specific thermal treatments.

Strain rate [s⁻¹]	
As-received and 150°C annealing method A	150°C and 350°C annealing method B
2.6 x 10 ⁻⁵	6.7 x 10 ⁻⁶
4.5 x 10 ⁻⁵	1.3 x 10 ⁻⁵
2.5 x 10 ⁻⁴	2.6 x 10 ⁻⁵
—	4.5 x 10 ⁻⁵
—	2.5 x 10 ⁻⁴
—	4.5 x 10 ⁻⁴

6.1.2 Wire microstructure, experimental results, and discussion

The wires have similar cross-sectional areas and purities to the thin films measured in previous studies.^{37,39} Conversely, several differences are implicit: initial dislocation density, texture (preferred orientation along the loading axis vs. perpendicular to it), grain shape, absolute grain size, and the ratio of grain size-to-film or wire thickness. By annealing, the first

four differences between grain structures are significantly reduced. The grain size-to-film or wire thickness ratio difference remains, allowing a comparison between micro-wires and thin films. This ratio may be a significant source of low modulus values typically measured in microtensile studies on freestanding thin films.

Using an FEI XP dual beam focused-ion beam scanning electron microscope (FIBSEM, or just FIB), a cross-sectional cut along the length of the wire was made (Figure 6-1). With this instrument, the transverse, longitudinal, and cross-sectional grain sizes can be imaged. The cutting process uses focused gallium ions; the process can be monitored by periodically capturing electron or ion beam images. Because the wires were manufactured using a drawing process, the as-received condition reveals an elongated grain structure along the drawing axis, as shown in Figure 6-3 and Figure 6-2. A grain structure like this with small, imbedded grains provides multiple layers of constraint for the inner grains from free-surface interactions. With a columnar structure, most grains have a free surface, as shown in Figure 6-5. Al thin films upon deposition (i.e. physical vapor deposition, or PVD, techniques) typically exhibit columnar grains whereas the grain structure of the wires can be altered from a drawn to a columnar structure by annealing. Figure 6-4 shows how the elongated grain structure of the micro-wires approach, but do not reach the columnar grain structure of thin films.

For comparing the grain structure of the wires to the grain structure of the thin films, the transverse grain size measurement was the most appropriate. For determining grain size, the line intercept method, as shown in Figure 6-6, was employed. Table 6-2 summarizes grain size measurements for both the Al and Cu wires. In the FIBSEM, the sample was positioned normal to the ion beam (I-beam), and at the same time, it was positioned at a 52° angle to the electron beam (E-beam). In these images, the point of view is looking from the normal I-beam;

therefore, the real grain size was directly measured from the image and no correction of foreshortening was necessary. It should be noted that in the wires annealed at 350°C, voids are present along some grain boundaries.



Figure 6-1: A cross-sectional FIB cut of a 10 μm diameter Al wire.

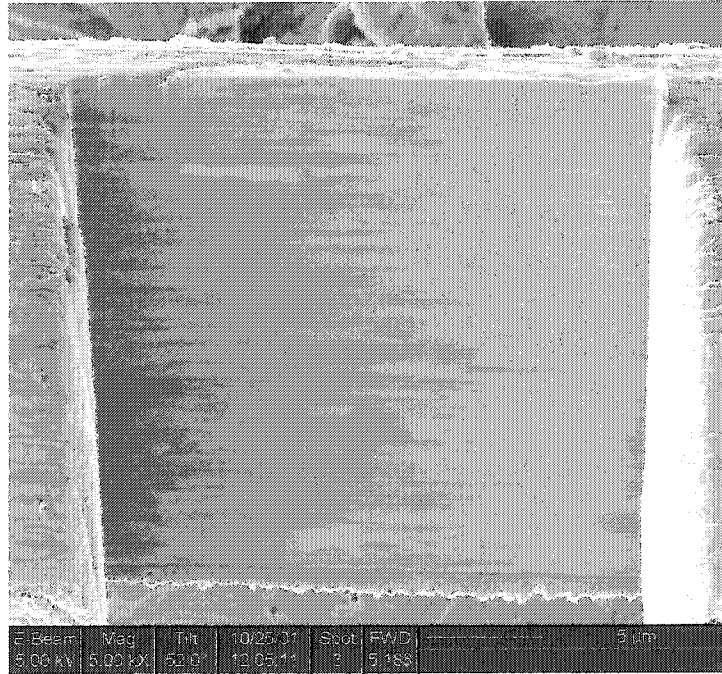


Figure 6-2: A cross-sectional FIB cut of a 10 μm diameter Cu wire showing the as-received microstructure.

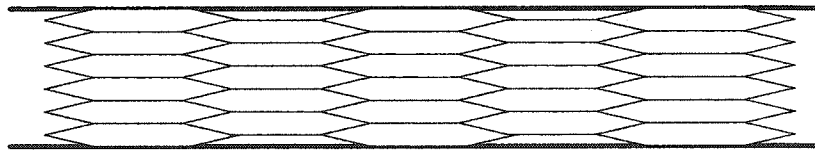


Figure 6-3: Elongated grain structure for the as-received commercial micro-wires showing clear evidence of a drawing process.

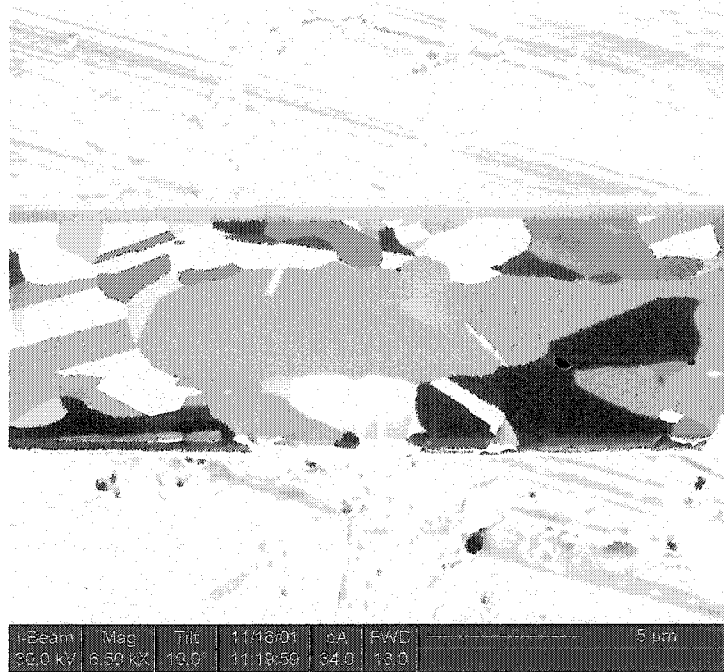


Figure 6-4: A cross-sectional FIB cut showing the annealed microstructure approaching a columnar grain structure for a 10 μm diameter Cu wire; the Cu gives a higher grain contrast than the Al.

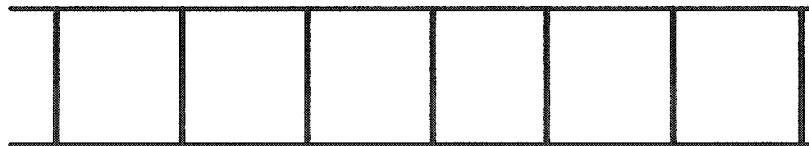


Figure 6-5: Columnar grain structure typically found in physically vapor deposited Al thin films.

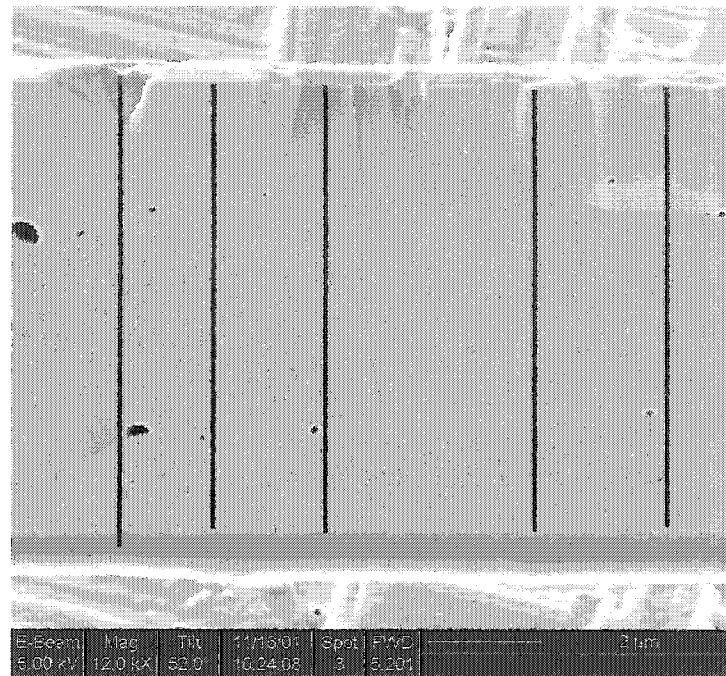


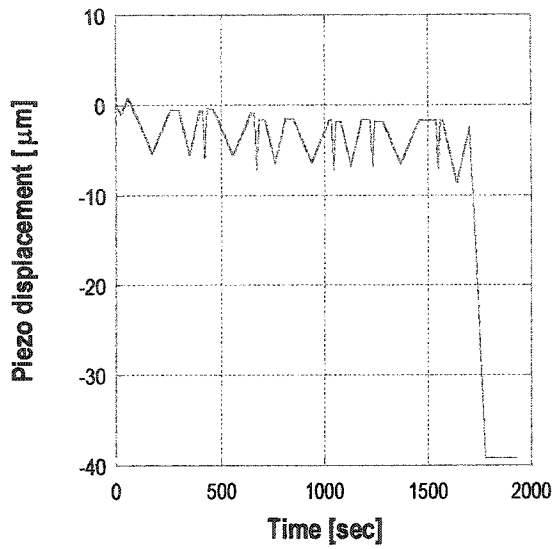
Figure 6-6: The line intercept method was used for measuring grain size; this is a 10 μm diameter Al wire. Note the voids (black spots) present along some grain boundaries.

Table 6-2: Thermal treatments and the associated transverse grain size for the Al and Cu micro-wires.

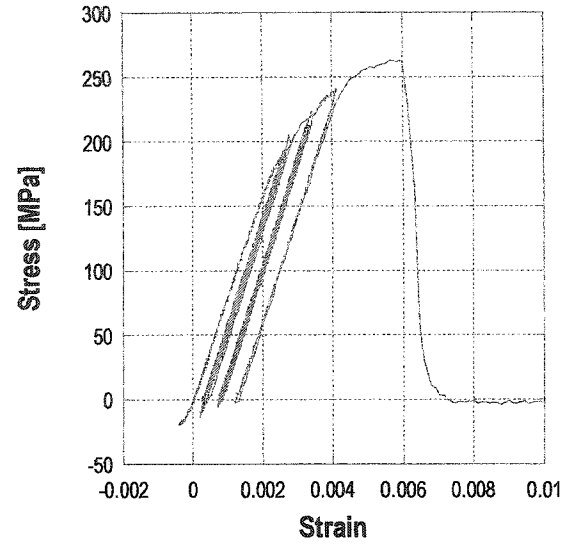
Heat treatment	Transverse grain size [μm]	
	Aluminum	Copper
As-received	0.36	0.28
150°C thermal treatment A	0.51	0.34
150°C thermal treatment B	0.81	0.58
350°C thermal treatment B	2.36	1.70

Effective modulus values were determined by measuring the slope of the unloading curve for each strain rate. Figure 6-7 and Figure 6-8 show typical stress-strain plots for an as-received Al wire and a Cu wire annealed at 150°C with thermal treatment A, respectively. The effective modulus values measured at lower strain rates were then subtracted from the effective modulus

value measured at the highest strain rate and then divided by the elastic modulus of the bulk material, 70 GPa; the normalized change in effective modulus, $\Delta E/E$ is plotted against $\dot{\epsilon}$ [s⁻¹] (where $\dot{\epsilon}$ is plotted on a logarithmic scale). Figure 6-9 shows the plot of $\Delta E/E$ vs. $\dot{\epsilon}$ for the small-grained Al wires (as received and 150°C anneal). Only one 150°C (thermal treatment B) annealed Cu and Al wire was tested, therefore no error bars are present. The as-received Al wires do not demonstrate rate effects because all data points are scattered around 0. However, a small amount of annealing at 150°C induces the beginning of some strain rate sensitivity. At the lower strain rates, the strain rate sensitivity was stronger as seen by a rapidly falling $\Delta E/E$. If the strain rate were to approach zero, $\Delta E/E$ would eventually become constant. Figure 6-10 shows $\Delta E/E$ vs. $\dot{\epsilon}$ for small-grained Cu wires, and Figure 6-11 shows $\Delta E/E$ vs. $\dot{\epsilon}$ for the large-grained Cu and Al wires, as well as for the Al thin film. No error calculations were available for the Al thin film results.

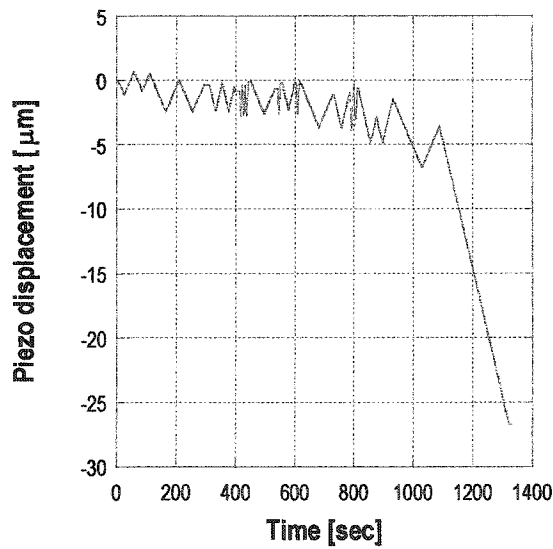


a)

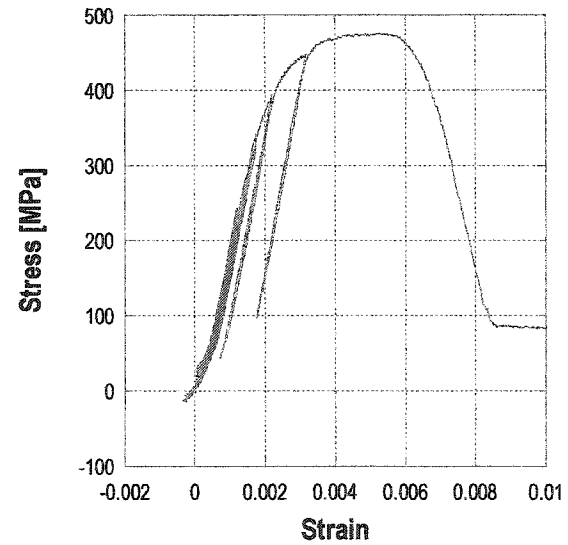


b)

Figure 6-7: a) A plot of piezo displacement vs. time showing multiple loading and unloading events, and b) the stress-strain plot for an as-received Al micro-wire.



a)



b)

Figure 6-8: a) A plot of piezo displacement vs. time showing multiple loading and unloading events, and b) the stress-strain plot for a Cu wire annealed at 150°C with thermal treatment A.

As seen in Figure 6-9, the as-received Al wires do not show a dependence of effective modulus on strain rate, or a strain rate sensitivity. In contrast, the wires annealed at higher temperatures show noticeable strain rate sensitivity. Therefore, in the Al wires a dependence of effective moduli on strain rate appears to increase with increasing grain size and decreasing dislocation density. The dislocation density in the as-received wires was very high because of the prior cold working taking place throughout the drawing process. By annealing, the dislocation density was reduced.⁷¹ If GBS was the primary relaxation mechanism then smaller grains would result in a lower effective modulus because there is a higher density of grain boundaries, or more channels for atomic diffusion resulting in a higher stress relaxation. Thus, dislocation density appears to be the critical factor in determining the strain rate sensitivity of Al and Cu micro-wires, although, even if below the detection limit, a contribution from voids located along the grain boundaries cannot be discounted.

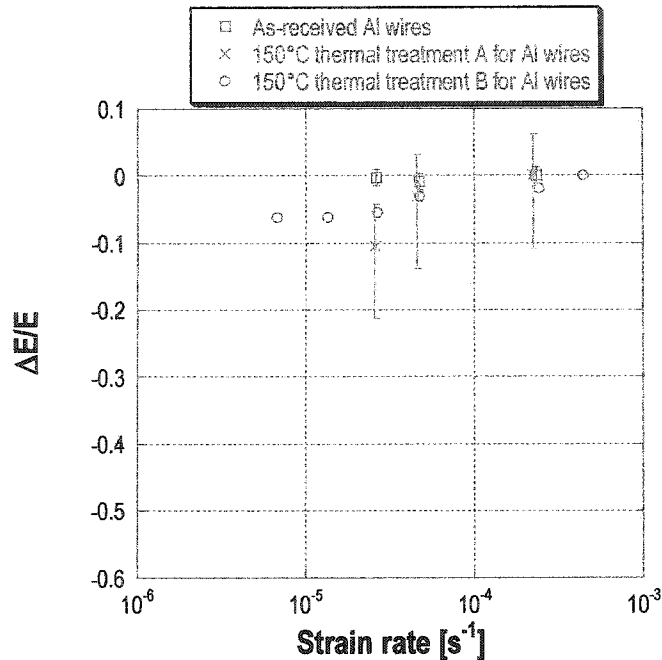


Figure 6-9: A plot of $\frac{\Delta E}{E}$ vs. $\dot{\epsilon}$ for small grained 10 μm diameter Al wires.

Similar to the results from the Al wires, the Cu wires illustrate a strain rate sensitivity after annealing. A material dependence of strain rate effects can be observed when comparing the results from the Al and Cu wires to one another. The Al shows a stronger dependence than the Cu (Figure 6-11). This was expected because the melting point of Al (660°C) is lower than Cu (1083°C). It also suggests that the strain rate sensitivity is more strongly dependent on microstructural characteristics such as grain size and dislocation density than grain boundary characteristics (possible void formation or micro-cracking on the grain boundaries) that appear in both the 350°C annealed Al and Cu wires. Finally, in Figure 6-11, the Al thin film data shows a large degree of relaxation, which agrees strongly with the Al wire results, suggesting that the effects of specimen geometry, scale, and grain size-to-thickness ratio are negligible.

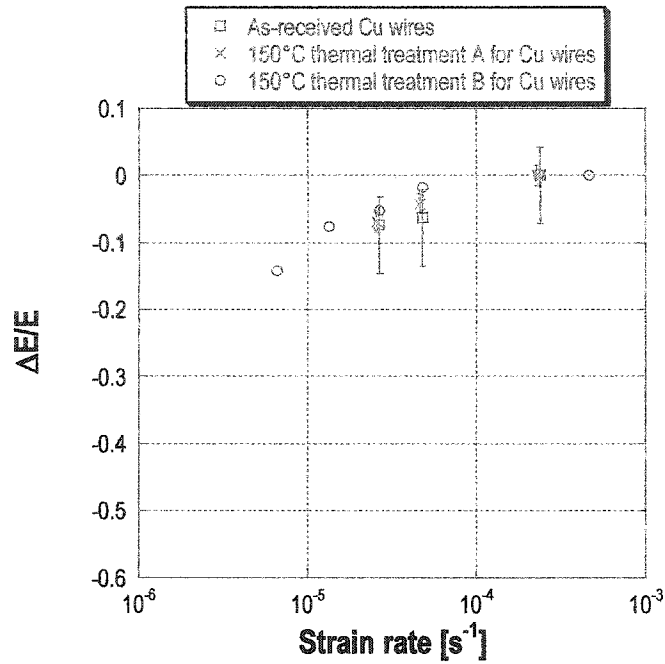


Figure 6-10: A plot of $\frac{\Delta E}{E}$ vs. $\dot{\epsilon}$ for small grained 10 μm diameter Cu wires.

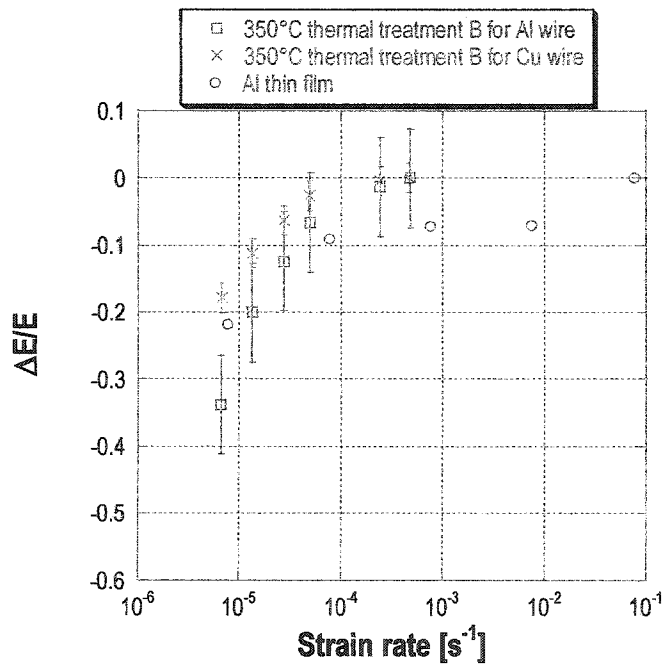


Figure 6-11: A plot of $\frac{\Delta E}{E}$ vs. $\dot{\epsilon}$ for large grained 10 μm diameter Al and Cu wires; the results from the wires are compared to an Al thin film.

These micro-wire experiments served as a baseline study for better understanding the mechanical behavior of thin films. In this study, a transition from bulk mechanical behavior to thin film mechanical behavior was observed in both the Al and Cu micro-wires. The large-grained Al wires compare closely with the results from the Al thin film suggesting that a microstructural change associated with annealing was a strong variable for the dependence of the effective modulus on strain rate. Geometry constraints do not appear to have as large an influence on moduli values as microstructure, mainly because both the cylindrical micro-wire and the rectangular thin film show similarities in effective modulus vs. strain rate behavior. With an annealed wire, however, not only was the grain size increased, but it was inferred that the dislocation density was reduced. Therefore, in the annealed wires, the effective moduli values as a function of strain rate depend on grain size and dislocation density. Also, in agreement with the expected results, Al shows a stronger strain rate dependence than Cu.

6.2 *Al microtensile beam experiments*

The 0.50 μm thick microtensile beams exhibited consistent behavior (e.g. strain rate sensitivity and stress relaxation experiments, as described in Chapter 5); therefore, additional experiments were carried out only with these microtensile beams. Four types of experiments were performed; stress relaxation, strain rate sensitivity, anelastic recovery, and activation energy experiments. Stress relaxation experiments were performed at a variety of different strain amplitudes; for increasing strain amplitudes, a larger amount of stress relaxation was observed. Strain rate sensitivity experiments were performed to a strain amplitude of 0.6%; four different strain rates were employed and two runs were performed. Results from this experiment

pointed to a finite time associated with a recovery process, which was investigated in the anelastic recovery experiment. The final set of experiments performed allowed determination of the activation energy for the anelastic mechanism responsible for the observed stress relaxation and recovery behaviors. Additionally, it was determined that this activation energy was independent of stress level.

6.2.1 Stress relaxation experiments

Stress relaxation experiments using a single strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ and five different strain amplitudes (0.20%, 0.30%, 0.40%, 0.50%, and 0.60%) were performed on three $0.50 \text{ }\mu\text{m}$ thick microtensile beams. Three runs were performed where the strain amplitudes were performed in a low-to-high sequence for the 1st and 2nd runs, and a random sequence for the 3rd run. The goal of these experiments was to investigate a transition from elastic to anelastic behavior in the stress-strain curve.

Using an arbitrary waveform specifically designed for this experiment and the parameters listed in Table 6-3, a $0.50 \text{ }\mu\text{m}$ thick microtensile beam was tested under the same strain rate to five different strain amplitudes. Two other $0.50 \text{ }\mu\text{m}$ thick microtensile beams tested exhibited similar results.

The fraction of stress relaxation observed was measured by subtracting the stress at approximately $t = 1.5$ seconds, $\sigma_{\min}$, from the maximum stress reached upon loading, and then

dividing by the maximum stress, or $\frac{\Delta\sigma}{\sigma_{\max}} = \frac{\sigma_{\max} - \sigma_{\min}}{\sigma_{\max}}$. The value of 1.5 seconds was

chosen because this is the shortest amount of time allowed for relaxation. As shown in Figure 6-12, as the time allowed for relaxation increased, the amount of stress relaxation continuously

increased. However, as shown in Figure 6-13, the fraction of stress relaxation taking place was independent of stress level. This indicates that the sample is displaying linear anelastic behavior.

Table 6-3: Lists the parameters for the stress relaxation experiments performed on the 0.50 μm thick Al microtensile beam; arbitrary waveform named "wave_G.csv".

Strain [%]	Strain rate [s^{-1}]	Frequency setting [Hz]	Amplitude setting [mV]	Hold time [sec]
0.2	8.33×10^{-2}	21,897.57	99	≈ 1.44
0.3	8.33×10^{-2}	14,598.38	199	≈ 2.16
0.4	8.33×10^{-2}	10,948.78	299	≈ 2.88
0.5	8.33×10^{-2}	8,759.03	398	≈ 3.60
0.6	8.33×10^{-2}	7,299.19	498	≈ 4.32

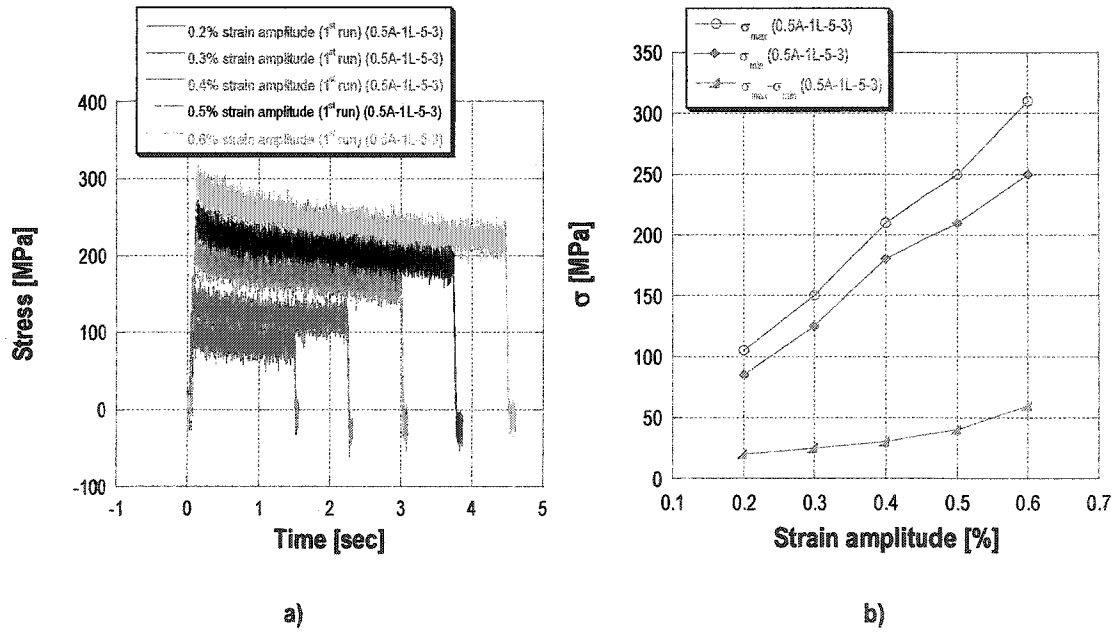


Figure 6-12: a) A plot of stress vs. time showing the stress relaxation taking place at each strain amplitude for the 1st run and b) the maximum stress ($\sigma_{\max}$), minimum stress ($\sigma_{\min}$), and difference in stress ($\sigma_{\max} - \sigma_{\min}$) for microtensile beam 0.5A-1L-5-3.

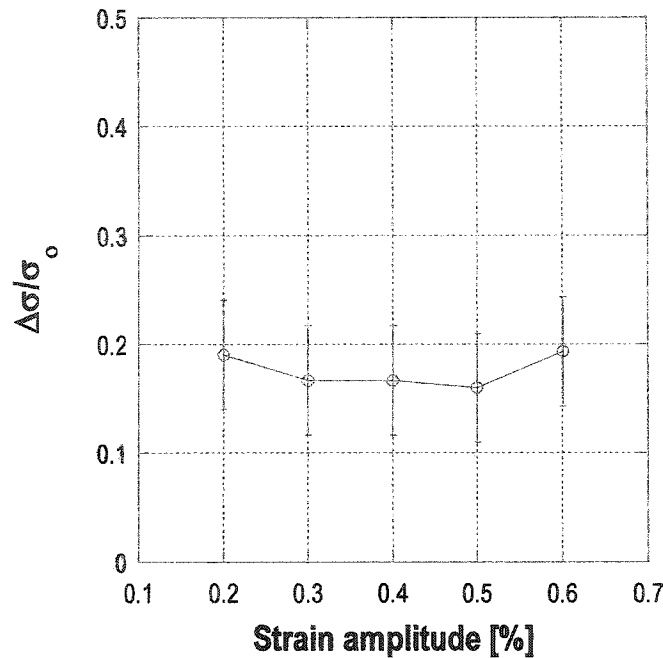


Figure 6-13: A plot of $\Delta\sigma/\sigma_0$ vs. strain amplitude for the stress relaxation experiment (1st run) performed on microtensile beam (0.5A-1L-5-3).

6.2.2 Strain rate sensitivity experiments

When a material is anelastic, the stress relaxation and strain rate dependence are related. To examine this relationship, at the largest strain amplitude of 0.6%, measurements at different strain rates were performed in order to investigate the strain rate sensitivity. Strain rate sensitivity experiments were performed using four different strain rates and one strain amplitude. Two sequences (i.e. runs) of strain rates (Table 6-4 and Table 6-5) were performed where one cycle was carried out at each strain rate and approximately 30 seconds elapsed between successive cycles. The strain rate sensitivity exponents were approximately the same values as for the strain amplitude of 0.17% indicating that between 0.17% and 0.60% strain, the anelastic mechanisms operate similarly (Figure 6-14). Figure 6-15 shows the stress vs. time for

the highest strain rate comparing the 1st and 2nd runs. A dependence of strain rate history is observed in that the peak stress reduces from the 1st to the 2nd run for the same strain rate. This implies that a finite recovery time is associated with the anelastic mechanisms. Additionally, the sequence of strain amplitudes performed does not affect the total amount of stress relaxation as indicated by the agreement among the stress relaxation curves for the 1st, 2nd, and 3rd runs. These results provide a basis for designing the anelastic recovery experiments in an attempt to determine the recovery time for the anelastic mechanisms in the 0.50 μm thick microtensile beams.

Table 6-4: List the parameters for the strain rate sensitivity experiments in the 1st run performed on the 0.50 μm thick Al microtensile beams at a strain amplitude of 0.6%.

Order of strain rates	Strain [%]	Strain rate [s^{-1}]	Frequency setting [Hz]	Amplitude setting [mV]	Hold time [sec]
1 st	0.6	8.33×10^{-1}	72991.90	598	≈ 0.435
2 nd	0.6	8.33×10^{-2}	7299.19	598	≈ 4.350
3 rd	0.6	8.33×10^{-3}	729.91	598	≈ 43.500
4 th	0.6	1.67×10^{-1}	14598.38	598	≈ 2.175

Table 6-5: List the parameters for the strain rate sensitivity experiments in the 2nd run performed on the 0.50 μm thick Al microtensile beams at a strain amplitude of 0.6%.

Order of strain rates	Strain [%]	Strain rate [s^{-1}]	Frequency setting [Hz]	Amplitude setting [mV]	Hold time [sec]
1 st	0.6	8.33×10^{-3}	729.91	598	≈ 0.435
2 nd	0.6	8.33×10^{-2}	7299.19	598	≈ 4.350
3 rd	0.6	1.67×10^{-1}	14598.38	598	≈ 43.500
4 th	0.6	8.33×10^{-1}	72991.90	598	≈ 2.175

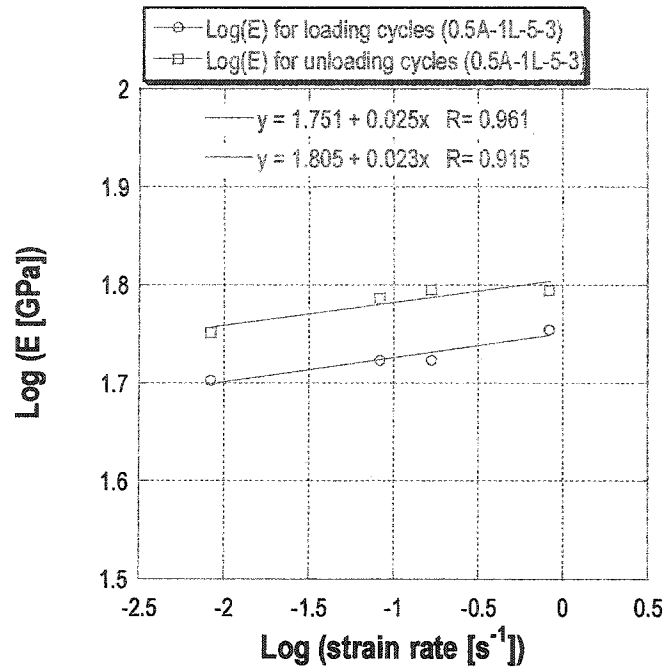


Figure 6-14: A plot of the $\text{Log}(E)$ vs. $\text{Log}(\text{strain rate})$ for both the loading and unloading cycles for the 1st run at each strain rate; this is for microtensile beam 0.5A-1L-5-3.

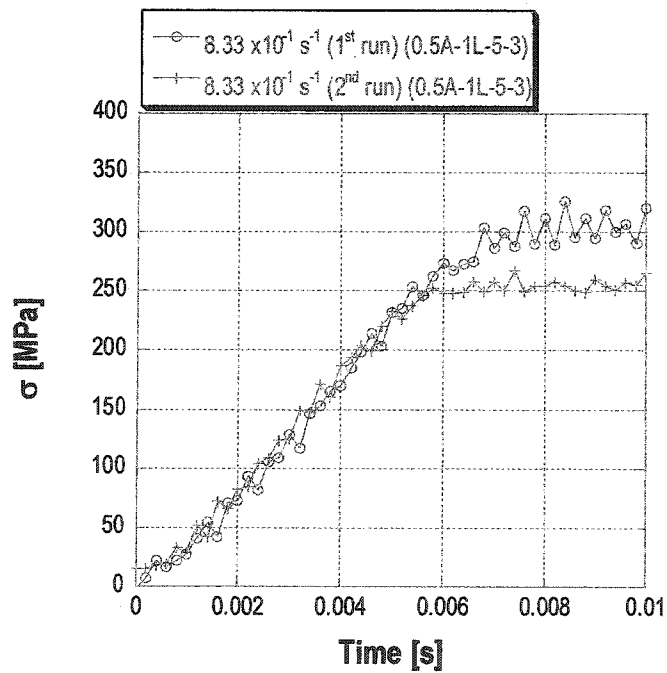


Figure 6-15: The stress vs. time plot for the highest strain rates comparing the maximum stress attained for the 1st and 2nd runs performed on microtensile beam 0.5A-1L-5-3.

Created by Paul El-Deiry

6.2.3 Anelastic recovery experiments

This experiment was performed using a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$. After the 1st cycle was performed, the sample was allowed to rest 10 minutes; the rest time was 9 minutes after the 2nd cycle (Figure 6-16). The experimental procedure is outlined in Table 6-6. A total of 19 cycles were performed. The purpose of this experiment was to determine a specific recovery time (or times) for the anelastic mechanisms. After each cycle and during the rest times, the feedback for the grip sensor was turned on. This guaranteed that the grip displacement was kept the same for each cycle. Additionally, the temperature was held constant at 38°C. Before testing, the system was allowed to sit for several hours at 38°C in order for all the thermal expansion to settle out. It turned out that for a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$, finite recovery times existed. With decreasing rest times, the amount of anelastic recovery was reduced.

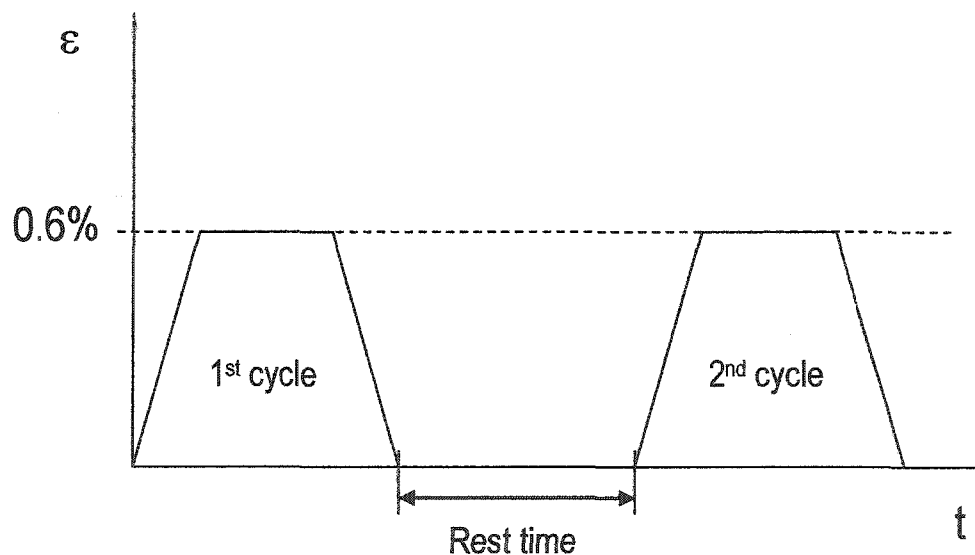


Figure 6-16: Schematic showing the consecutive cycles performed for the anelastic recovery experiment; the rest time between the 1st and 2nd cycle was 10 minutes.

Table 6-6: Lists the cycles and the rest time between cycles for the anelastic recovery experiment performed on microtensile beam 0.5A-1L-2-3 at a strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$.

Cycle #	1	2	3	4	5	6	7	8	9	10
Rest time after cycle [minutes]	10	9	8	7	6	5	4	3	2	1

Cycle #	11	12	13	14	15	16
Rest time after cycle [seconds]	30	10	10	5	2	1

Cycle #	17	18	19	strain-to-failure
Rest time after cycle [minutes]	35	60	20	N/A

Consider the stress-strain curves: the 2nd and 3rd cycles are similar, but the 4th and 5th cycles exhibit a narrower stress-strain loop indicating an incomplete recovery for a rest time of 8 minutes (Figure 6-17). For rest times of 9 and 10 minutes, the recovery is similar and nearly 100%. The 6th and 7th cycle agree with the 4th and 5th cycles. The 8th, 9th, 10th, 11th, and 12th cycles are also similar to one another, but exhibit a narrower stress-strain loop than the 4th, 5th, 6th, and 7th cycles. Between the 1st and the 12th cycles, two distinct recovery time constants are represented. The first ranges between 6 and 8 minutes, and the second ranges between 30 seconds and 3 minutes. This recovery time is best revealed by measuring the value of strain where the microtensile beam begins to take on load (i.e. the pick-up of load). Figure 6-18 displays the strain where the load pick-up occurs during the loading cycle vs. the rest time prior to that cycle. Two additional recovery time constants exist between the 13th and 17th cycles; the

first ranges between 5 and 10 seconds and the second between 1 and 2 seconds (Figure 6-18b). Another recovery time constant is present for rest times of 35 and 60 minutes; this 5th time constant is a function of the strain history. Overall, 5 distinct time constants for the recovery process are resolvable in this experiment (Figure 6-19).

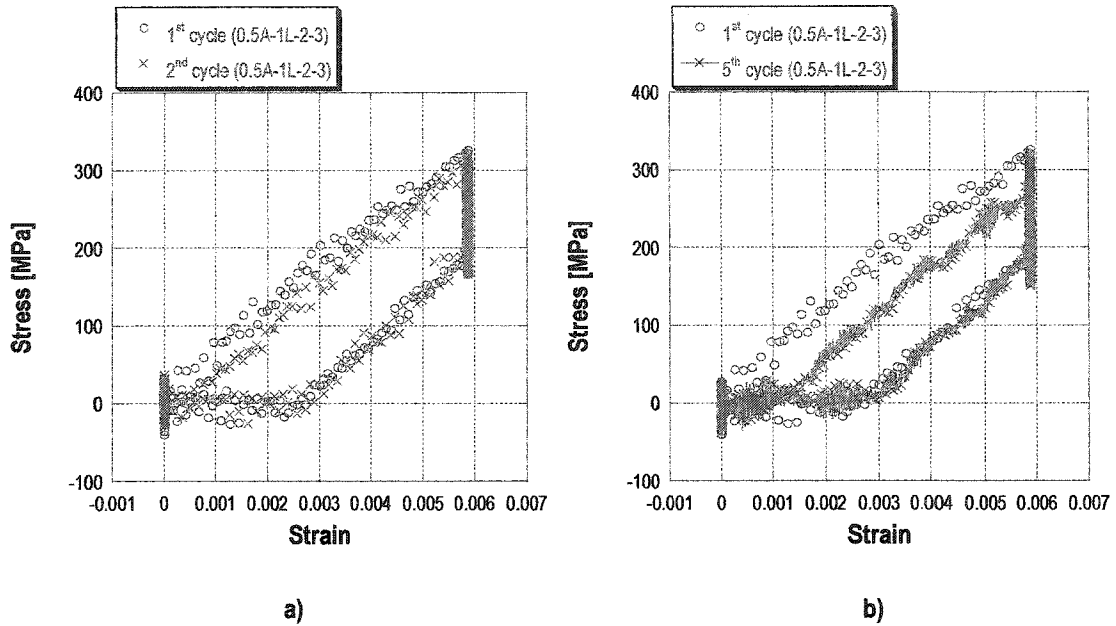


Figure 6-17: a) The stress-strain plot for the 1st and 2nd cycles and b) the 1st and 5th cycles at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ for a 0.50 μm thick Al microtensile beam.

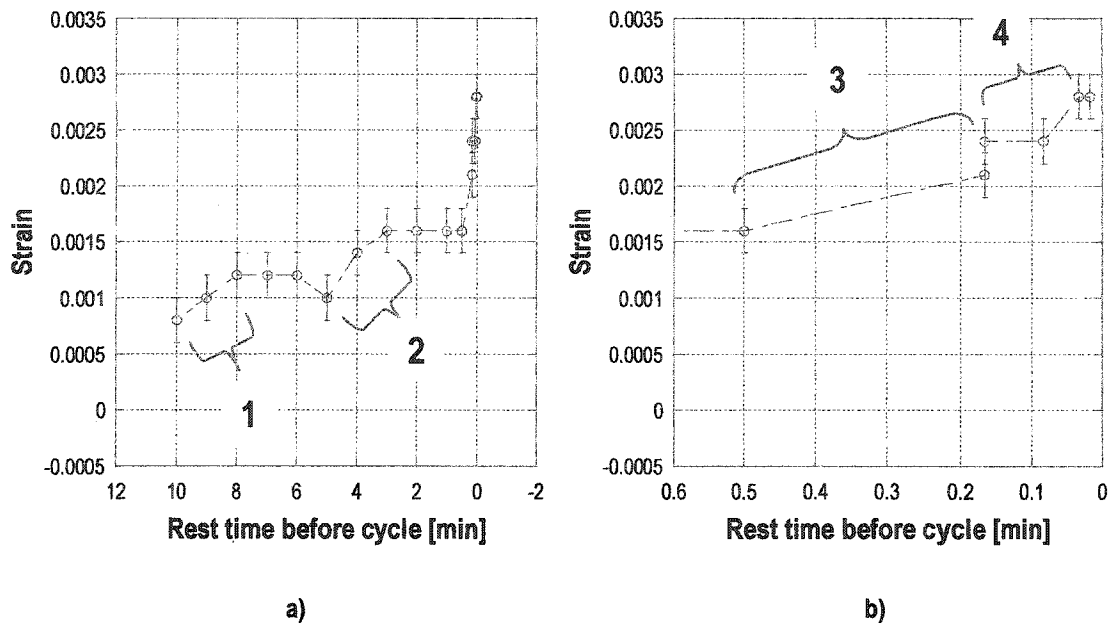


Figure 6-18: A plot of strain (at pick-up of the load) vs. rest time showing 4 of the 5 distinct regions of anelastic recovery and b) a close-up view of the shorter rest times.

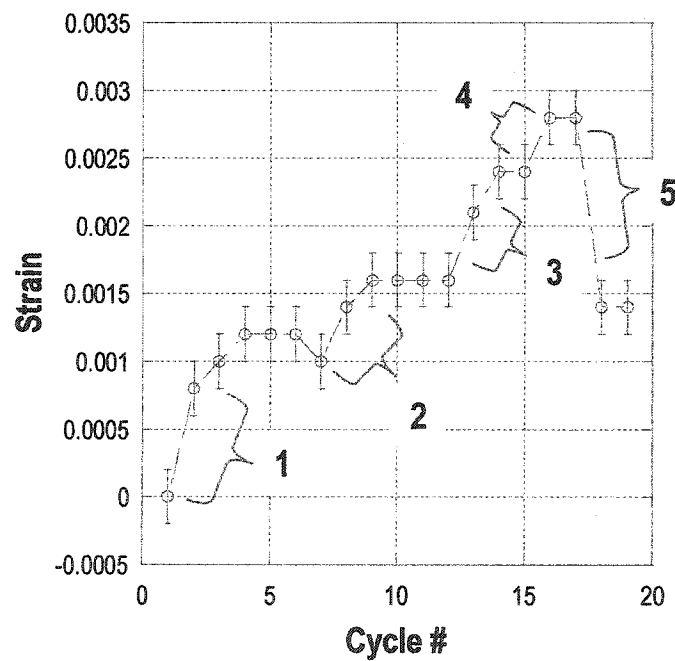


Figure 6-19: A plot of strain (at pick-up of the load) vs. cycle # showing the 5 distinct regions of anelastic recovery in the 0.50 μm thick Al microtensile beams.

A 35 minute rest time was performed between the 17th and 18th cycles. For a rest time of 1 second after the 16th cycle, the 17th cycle shows no recovery (Figure 6-20). A 60 minute rest time was performed between the 18th and 19th cycles. Because there is excellent agreement between the 18th and 19th cycles, it is suggested that the anelastic mechanisms (i.e. gliding dislocations) are no longer fully reversible. However, the 18th and 19th cycles exhibit more recovery than the in the 17th cycle (Figure 6-21). This indicates that by waiting 35 minutes, there is a partial recovery occurring. It also indicates that by performing the cycles without waiting for a long period of time (i.e. the 12th, 13th, 14th, 15th, 16th, and 17th cycles), a permanent deformation has taken place (i.e. the dislocations are becoming locked in place and are then unable to recover). This experiment provides insight into the most likely anelastic mechanism (i.e. dislocation glide) responsible for the recovery process. It is difficult to imagine that permanent deformation would be noticeable if diffusion-controlled GBS was the responsible mechanism.

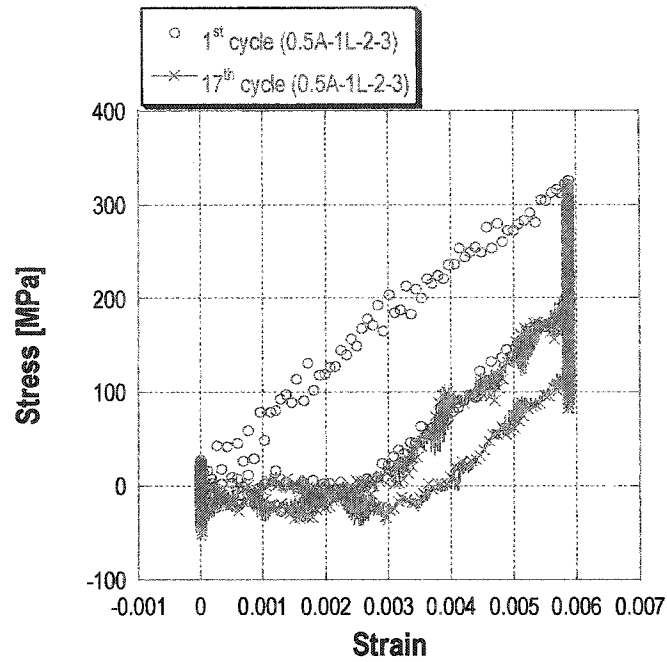


Figure 6-20: a) The stress-strain plot for the 1st and 17th cycles at a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ for a $0.50 \text{ } \mu\text{m}$ thick Al microtensile beam.

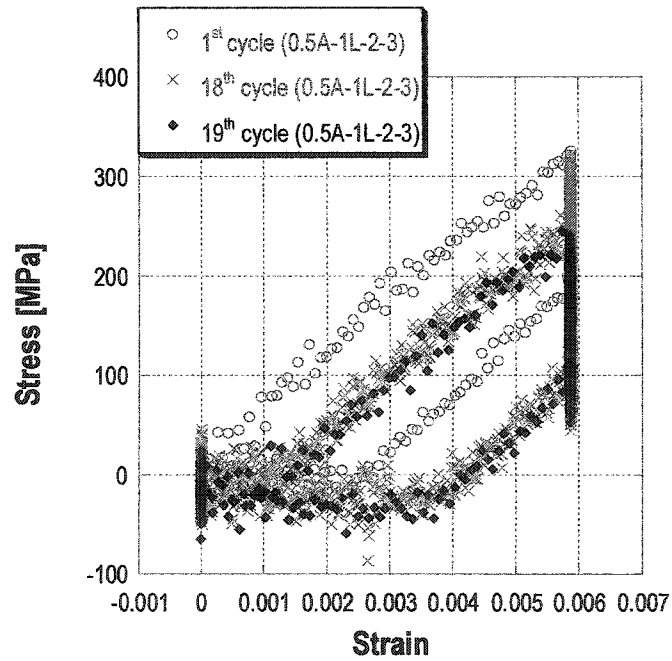


Figure 6-21: The stress-strain plot for the 1st, 18th, and 19th cycles for a $0.50 \text{ } \mu\text{m}$ thick Al microtensile beam; the 18th and 19th cycles were performed at the strain rate of $8.33 \times 10^{-3} \text{ s}^{-1}$.

6.2.4 Activation energy experiments

The purpose of these experiments was to determine the specific anelastic mechanism (i.e. dislocation glide or GBS) responsible for the observed relaxation behavior over the time interval of 0.5 seconds in the 0.50 μm thick microtensile beams. Stress relaxation experiments were performed at four temperatures (38°C, 50°C, 60°C, and 70°C). Five strain amplitudes (0.2%, 0.2%, 0.4%, 0.5%, and of 0.6%) using a strain rate of $8.33 \times 10^{-2} \text{ s}^{-1}$ were performed at each temperature with approximately 7 minutes between cycles. Only the 0.6% strain amplitude at each temperature was analyzed; two specific time constants were determined because each stress relaxation curve was fit using a double exponential. Using the values of the time constants and the well known Arrhenius relationship, $\tau = \tau_o \exp\left(\frac{-Q}{RT}\right)$, the activation energy, Q , was calculated.

6.2.4.1 Experimental procedure

Because these experiments are difficult, the procedure for temperature testing is included here. After cutting the side supports of the Si frame and adjusting the piezo translator to give zero load, the feedback control from the capacitance grip sensor to the piezo translator was turned on. This kept the grips in the same relative position to one another while allowing the piezo translator to automatically adjust in order to compensate for the thermal expansion in the microtensile system as the temperature changed. When the system stabilized after several hours, the relay to the waveform generator was turned off while the appropriate waveform was downloaded to the arbitrary function generator. There are a total of 5 waveforms for each strain

amplitude (Figure 6-22 and Figure 6-23). Additionally, the hold time at the maximum strain was kept the same for all strain amplitudes; approximately 5 seconds.

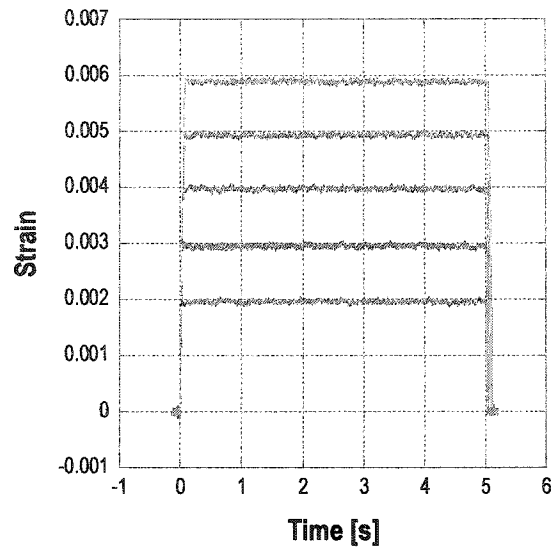


Figure 6-22: A plot of strain vs. time displaying the complete waveform for each strain amplitude.

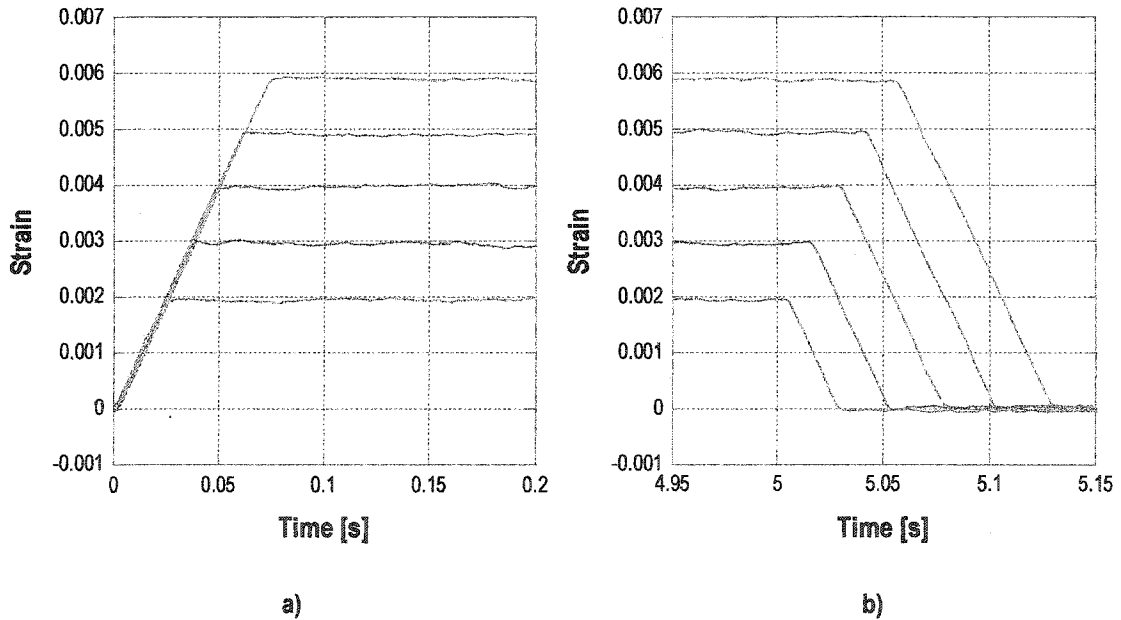


Figure 6-23: A plot of strain vs. time showing the beginning and b) end of the waveforms.

When increasing the temperature from 38°C to 50°C, for example, the grip displacement feedback to the piezo translator is turned on to keep the grip displacement constant. However, it turned out that the Al microtensile beam was itself expanding. Therefore, because the grip position was held constant with the feedback controller, the expansion of the microtensile beam caused it to buckle. Hence, when successive strains were performed, the maximum stress was always lower; and with that, the stress relaxation was less. The thermal expansion of the microtensile beam was estimated to be approximately 0.18 µm with an increase in temperature of 12°C by applying the following method:

$$\sigma = E\alpha\Delta T = (70GPa) \cdot (23 \times 10^{-6} / ^\circ C) \cdot (50^\circ C - 38^\circ C) = 19.3MPa ;$$

Equation 6-1: The stress associated with the thermal expansion in the Al microtensile beam.

$$E = \frac{\sigma}{\varepsilon} \text{ and also } \varepsilon = \frac{\sigma}{E} = \frac{19.3MPa}{70GPa} = 0.00027 = 0.03% ;$$

Equation 6-2: The strain associated with the stress calculated in Equation 6-1.

$$0.0003 \cdot 600\mu m = 0.18\mu m .$$

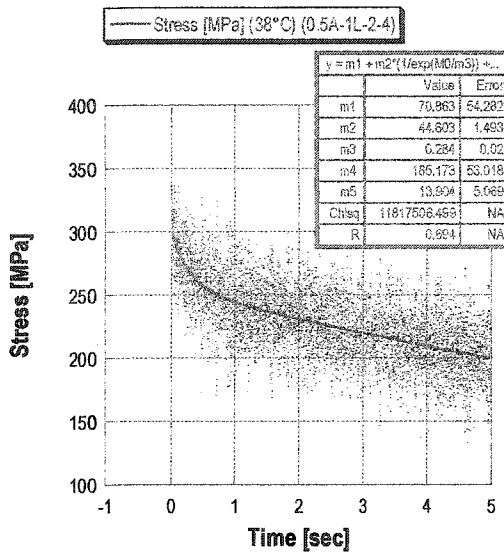
Equation 6-3: The expansion in the microtensile beam with a 600 µm gage length.

This value of 0.18 µm only accounts for the expansion in the microtensile beam. However, parts of the microtensile system are also expanding. Calculating the expected expansion for the microtensile system is a complicated problem. Therefore, to solve this problem and ensure the microtensile beam is under the same stress state, the zero load was determined at each new

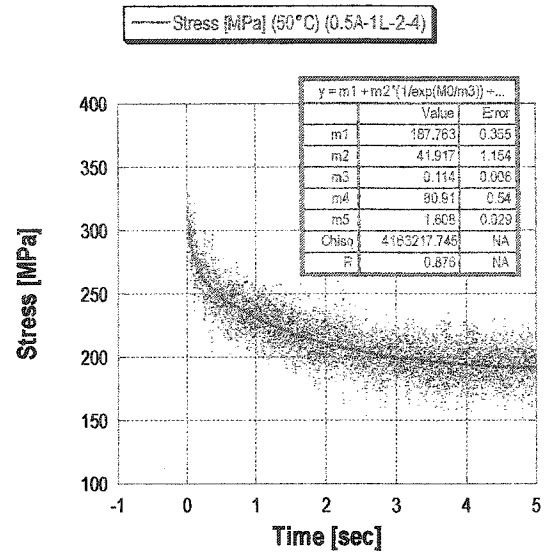
temperature. Furthermore, when increasing to a new temperature, it was necessary to wait approximately 5 to 7 hours for the thermal expansion to settle out.

6.2.4.2 Experimental results: an activation energy

Two time constants were determined for the 0.6% strain amplitude at each of the four temperatures, but an activation energy was calculated only using the first time constant. The reason for concentrating on the short time constant is because the data for the second time constant is not yet entirely understood. This becomes clear when looking at the shape of the curves for each temperature. In the middle two temperatures (i.e. 50°C and 60°C), the relaxation mechanism appears to exhaust around 5 seconds while at the two end temperatures (i.e. 38°C and 70°C) the relaxation did not show signs of slowing down within 5 seconds. Each curve was fit with a double exponential using the graphical program Kaleidagraph®. A function with five unknowns was created and the program then determined the unknowns by providing the best fit to the data. Figure 6-24 and Figure 6-25 show the double exponential fit for the strain amplitude of 0.6% at each of the four temperatures.

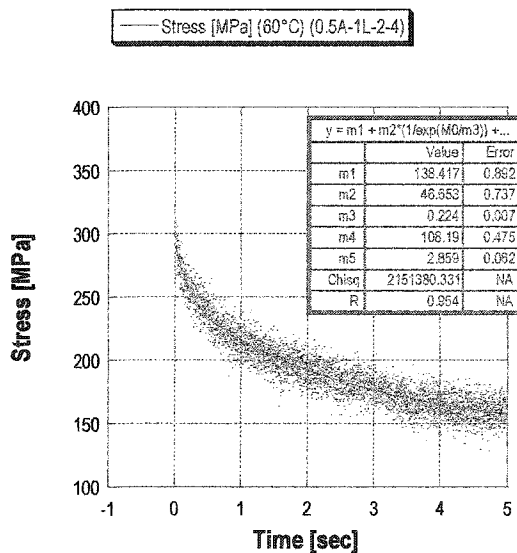


a)

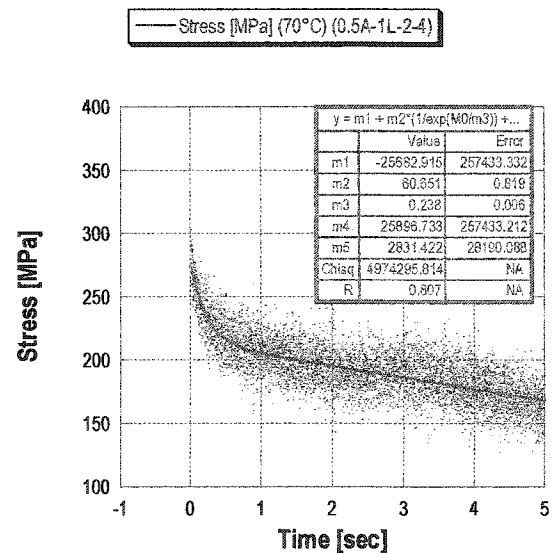


b)

Figure 6-24: A plot of stress vs. time showing the stress relaxation at a) 38°C and b) 50°C.



a)



b)

Figure 6-25: A plot of stress vs. time showing the stress relaxation at a) 60°C and b) 70°C.

Once the time constants were determined for the two 0.50 μm thick microtensile beams (Table 6-7), the activation energy was calculated using the Arrhenius equation. The natural

logarithm ($\ln$) of the time constant (i.e. $\ln \tau_1$) was plotted against $1/T$, where T is the temperature in Kelvin. The slope of the line is then the activation energy, Q , divided by the universal gas constant, R , which is 8.314 J/mol-K . For both the $0.50 \text{ }\mu\text{m}$ thick microtensile beams, the activation energy agrees with a dislocation glide mechanism (Figure 6-26). The activation energies for cross slip and dislocation climb are much higher than that observed for the microtensile beams indicating that the dominant mechanism over a 0.30 second time interval is dislocation glide.⁶⁵

Table 6-7: The first time constant, τ_1 , for the stress relaxation mechanism in the $0.50 \text{ }\mu\text{m}$ thick Al microtensile beams.

Temperature [°C]	τ_1 [s]	
	0.5A-1L-2-4	0.5A-1L-4-7
38	0.284	0.153
50	0.114	0.266
60	0.224	0.191
70	0.238	0.318

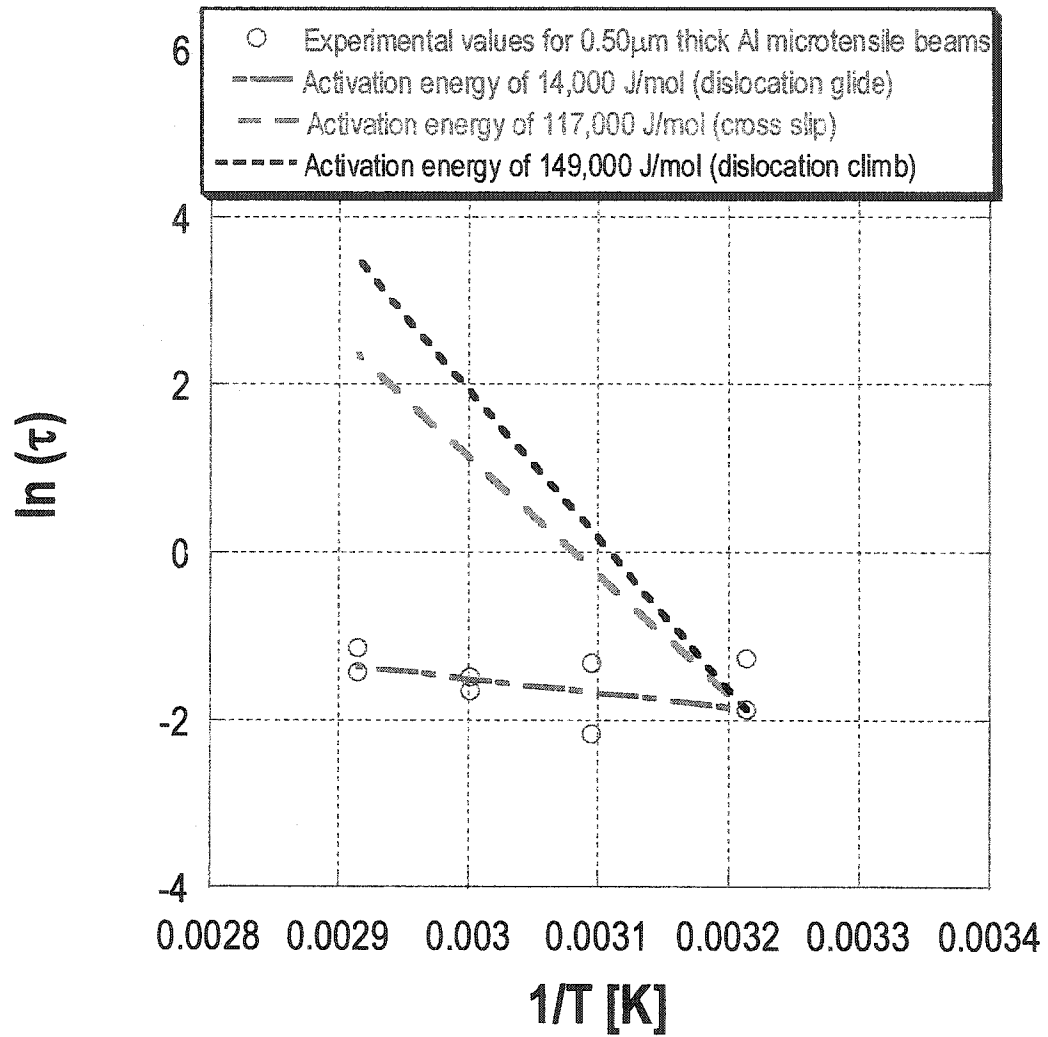


Figure 6-26: A plot of $\ln \tau$ vs. $1/T$ where the slope is $\frac{Q}{R}$; Q is the activation energy and R is the universal gas constant 8.314 J/mol-K.

7 Conclusions and future experiments

7.1 Conclusions

- Strain rate sensitivity experiments performed on Al and Cu micro-wires indicate that larger grains and a lower dislocation density lead to a stronger strain rate sensitivity. This result agrees with the findings from the activation energy experiments (i.e. a dislocation glide mechanism). Weak strain rate sensitivity is observed for the small grained micro-wires with a high dislocation density; mobile dislocations do not need to travel very far before being inhibited by other dislocations and grain boundaries.
- The strain rate sensitivity and the amount of stress relaxation are independent of strain amplitude (or stress level) for the 0.50 μm thick Al microtensile beams. This result agrees strongly with the linear anelastic model proposed in Chapter 1 section 1.1.4. Furthermore, this model can be used for qualitatively predicting the modulus deficit and the stress relaxation behavior of freestanding Al thin films.
- When given enough rest time (6 to 8 minutes), the recovery of deformation was complete in the 0.50 μm thick Al microtensile beams. For consecutive cycles performed with short rest times of 1 or 2 seconds, permanent deformation was observed. The anelastic mechanism that provided the best explanation for this permanent deformation was dislocation glide (i.e. because the rest time was short, not enough time was allowed for the

dislocations to return to their original positions, and the dislocations were most likely becoming interlocked with one another).

- In this study, no connection was made between the long time constants observed in the anelastic recovery experiment and the short time constants observed in the activation energy experiment. A spectrum of time constants has been shown to exist for various mechanisms.^{11;12} This is most likely the case for these freestanding Al thin films.
- The activation energy for the rapid anelastic mechanism responsible for the observed stress relaxation and recovery behavior in the 0.50 μm thick Al microtensile beams was determined; this mechanism is dislocation glide.

7.2 Future Experiments

This study has brought attention to the difficulty in reproducing the mechanical properties of freestanding Al thin films from sample to sample and wafer to wafer even when the processing conditions are similar. For films having the same thickness, elastic modulus values vary from 55 GPa to 70 GPa. Also, the ductility can vary from 0.3% to 3%. Reproducibility is a major factor when measuring the mechanical properties of thin films. Even within the same laboratory, reproducibility is difficult. Therefore, when researching the mechanical behavior of thin films, comparing results across different laboratories is often a complex situation. Also, comparing results from different types of mechanical testing systems and techniques (i.e. microtensile testing, nanoindentation, bulge testing, internal friction, etc.) is also not straightforward.

7.2.1 Microtensile testing vs. nanoindentation

Results from a nanoindentation experiment performed on the film/substrate area of the Si frame of one 1.00 μm thick Al microtensile beam gave a modulus of approximately 70 GPa. In contrast, the freestanding microtensile beam resulted in a modulus of approximately 55 GPa. Because the superbeams resulted in a modulus of 70 GPa, the microtensile system is known to be capable of measuring the true modulus of freestanding thin films. From this short study, both nanoindentation and microtensile testing have been shown to be capable of measuring Young's modulus of Al thin films. A more thorough investigation is necessary to determine the detailed differences between freestanding thin films and films on substrates and reasons why the differences exist.

7.2.2 Microtensile superbeams

On the subject of the superbeam results, more characterization is necessary to determine the reasons behind their superior mechanical properties compared to the other microtensile beams tested. The superbeams produced a modulus of 70 GPa and a yield stress of around 700 MPa. However, the reasons behind these values are still mostly unknown. A more thorough characterization is underway; texture and grain size analysis are necessary but may not be sufficient.

7.2.3 Stress relaxation experiments for different materials

This study has provided insight into the mechanism responsible for rapid stress relaxation behavior in 0.50 μm thick freestanding Al thin films. With the current setup, mechanical testing of different materials can be performed. The fabrication process may need to be slightly altered,

but it was designed to be compatible for many different material systems. Alloying the Al with Ti is suggested in order to observe the influence Ti has on the stress relaxation mechanisms over short time intervals. The stress relaxation behavior has already been observed over long (10 minute) time intervals.⁴ In addition, copper microtensile beams may be fabricated and tested. Results from the copper microtensile beams can be compared to those of the Cu micro-wires and the Al microtensile beams. Silicon dioxide, gold, and gold-vanadium microtensile beams have already been fabricated and tested.⁶⁸

7.2.4 Microtensile system on-a-chip (in-situ TEM experiments)

A next step in this project is to visually observe the mechanisms responsible for relaxation in these thin films. This will be accomplished by designing a microelectromechanical device that can be situated into a TEM. Using a straining stage, the thin film on this device can then be strained. This structure is based on the design by Saif et al. at the University of Illinois.⁵³ Currently, fabrication of such a device is underway. Figure 7-1 shows a schematic of the structure; the holes located at the ends of the Si frame are where the straining stage will hold the device. Figure 7-2 shows a stereo microscope image and Figure 7-3 shows a scanning electron microscope image of a partially fabricated structure.

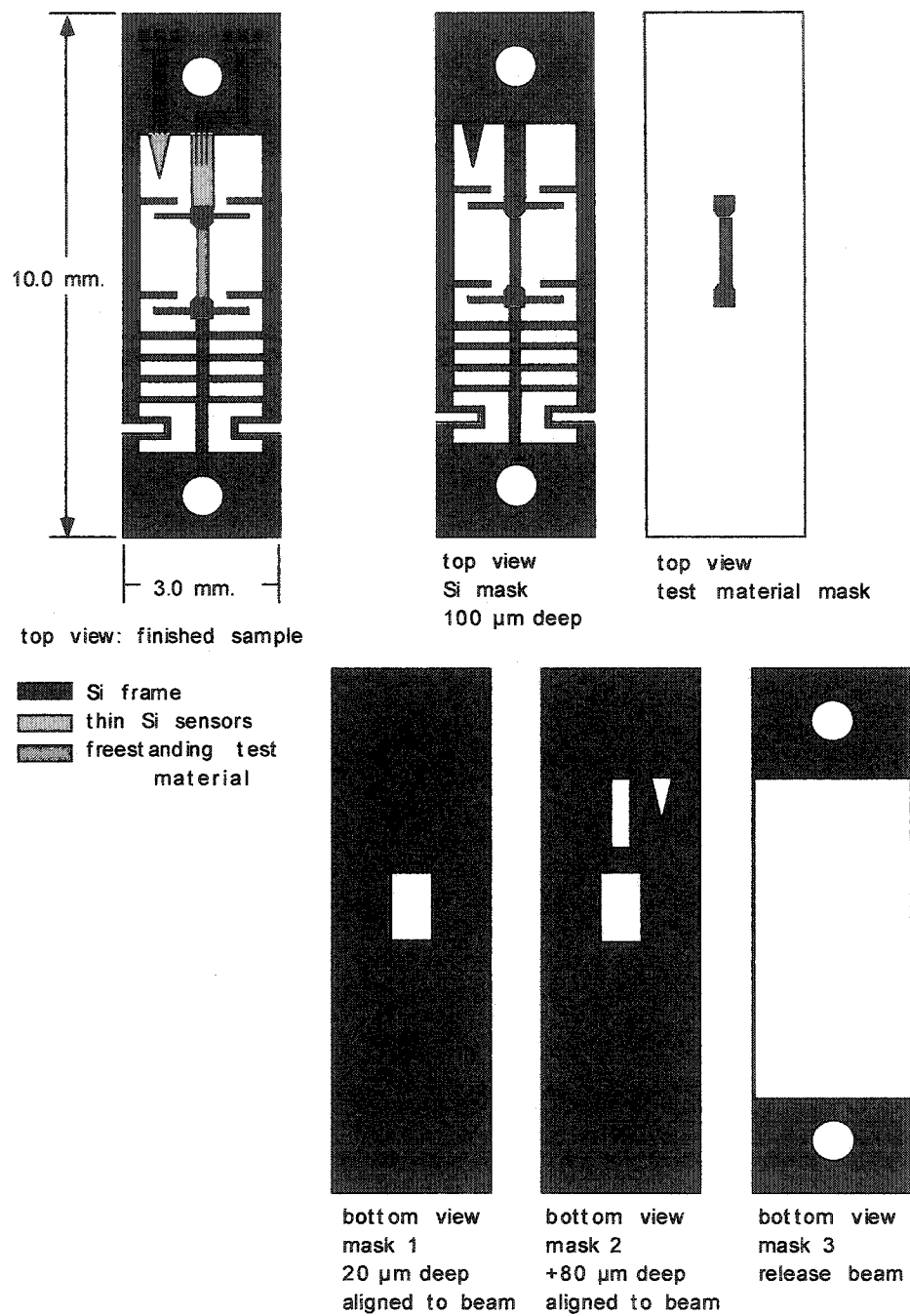


Figure 7-1: A schematic of the in-situ TEM microtensile device (courtesy of R. Vinci).

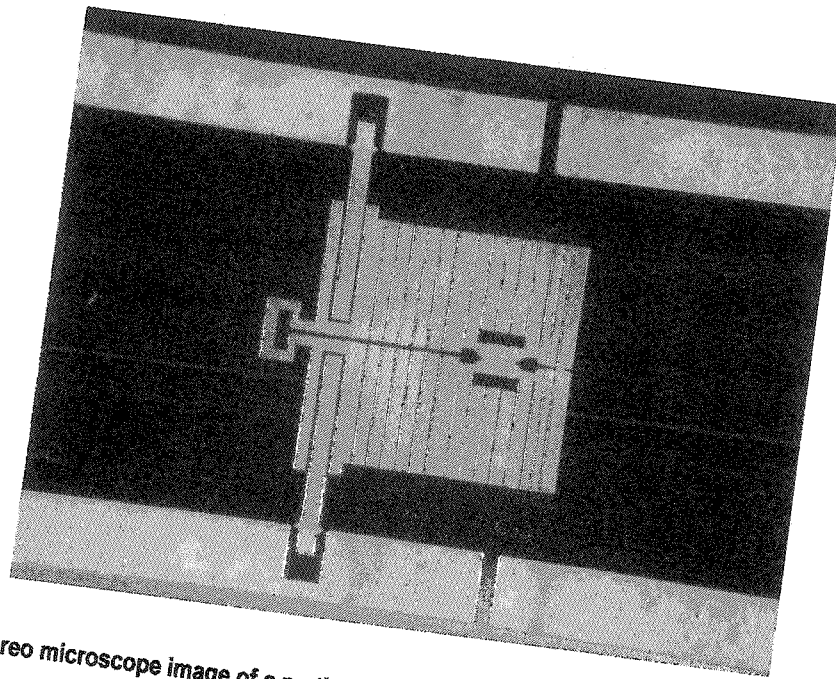


Figure 7-2: A stereo microscope image of a partially fabricated device (Mag 60x) (fabricated by J. Stanescu at the Cornell University Nanofabrication facility).

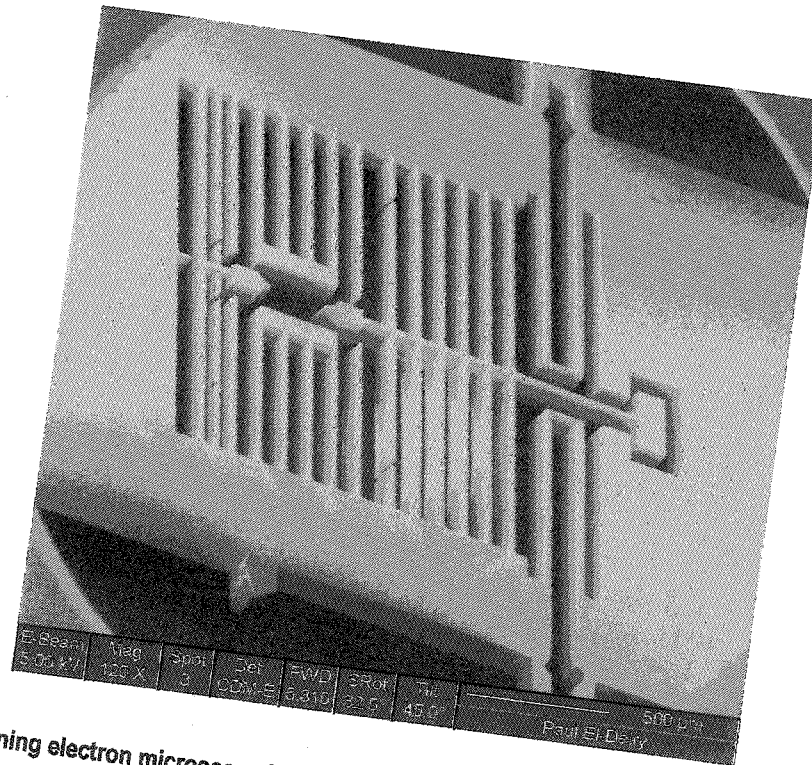


Figure 7-3: A scanning electron microscope image of a partially fabricated device (Mag 120x) (fabricated by J. Stanescu at the Cornell University Nanofabrication facility).

Created by Paul El-Deiry

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Appendix

This appendix contains the procedure for clamping and cutting the microtensile beam samples, the procedure for designing and downloading waveforms for use with the arbitrary function generator, a procedure for calibrating and tuning the piezo translator, materials used for the structural members of microtensile system #4, and the manufacturer's documentation for products used in this research.

Cutting and clamping microtensile beam samples

As discussed in Chapter 2, the microtensile beams were fabricated using well-known IC processing steps. Once the silicon frames were diced from the wafer, they were mounted into the grips. Typically, the method of mounting was as follows (the silicon frame was mounted into the grips with the film side facing the grips – this allowed the sample to be closest to the system's central axis):

1. First, place the clamp on the bottom grip.
2. The Si frame with the microtensile beam should be placed on a glass slide with the film side down.
3. Next, pick the Si frame up with the vacuum tweezers and place it between the face of the bottom grip and the clamp; with the other hand, push the clamp onto the Si frame holding it tight against the grip face.
4. Once the frame is secure, then the two screws can be tightened into the grip (these screws tighten the clamp/Si frame/grip face assembly and hold it together – before tightening the screws, small alignment adjustments can be made while the clamp is holding the Si frame).

5. At this point, the load is zeroed, and the top clamp is placed on the top grip. The top clamp is then pushed against the top part of the Si frame. When this is done, a load is usually registered (sometimes compressive and sometimes tensile).
6. When the top clamp is tightened, additional changes in load may be registered; this is a result of creating a stress on the Si frame.
7. Before cutting the frame to release the microtensile beam, this load on the frame needs to be zeroed. This is accomplished by adjusting the piezo translator.
8. Once the load is zeroed, the Si frame is cut using an AC-powered (not battery powered – it does not provide enough saw speed) Dremel® saw with a 3" Norton diamond saw blade. The left side is usually cut first followed by the right.

Designing and downloading waveforms for specific experiments

Each waveform is written in a ".csv" format in Excel where the numbers are floating point. In the first column is the number defining the point in the waveform while the second column is the waveform value for that point. The waveform values range between 0 and 1 and there are 32,768 (or 2^{15}) points that define the waveform. The function generator is connected to the computer using an R-232 serial connection. Using this and a specific program called "Waveform download", specific waveforms can be downloaded from the computer to the function generator. The file that will be downloaded can only be a maximum 8 characters long and must be located in the root directory of the drive (i.e. G:\wave_G.csv). Otherwise, the download program will not be able to find the file. The time of the download is approximately 5 to 7 minutes. If you are waiting longer than this, something is awry. Each waveform used in this research has specific parameters for producing specific strain rates and strain amplitudes. To run the downloaded

waveform, switch to the arbitrary mode on the function generator and enter the appropriate frequency and voltage.

Procedure for calibrating and tuning the piezo translator

Calibrating the piezo translator:

1. Make sure power is off. For safety purposes, remove power cable from wall or from the chassis.
2. Remove piezo servo card (E509.S1) from the amplifier chassis.
3. Plug in extender card into chassis and then plug piezo servo card into extender card. This allows power to be transferred to the piezo servo card while at the same time allowing access to the potentiometers on the piezo servo card.
4. Plug in both piezo wires (one goes to the "monitor signal" on the piezo servo card and the other goes to the amplifier) and power up the chassis. Power switch (rocker switch) is in the back of the chassis. Let it sit for 5 minutes or longer so the electronics can warm up.
5. Leave the BNC "control input" plug open (don't plug anything in there yet).
6. Make sure you plug in the BNC "monitor" plug; this should be already connected to the computer through the DAQ (data acquisition) card.
7. Turn on the appropriate LabVIEW program so that you can see the voltage coming in from the piezo signal.
8. Observe the piezo voltage. Adjust the "zero potentiometer" on the front of the piezo servo card until the voltage reads between -0.3 and -0.5 volts.

9. Make sure the "servo" switch is off (the piezo is now in open loop control).
Then, slowly introduce a rising voltage to the piezo translator by turning the offset knob. The voltage should increase. Once you finish "warming up" the piezo, turn the knob until the piezo reads its initial voltage value and, more importantly, until the knob no longer turns.
10. Next, switch the "servo" to on. This puts the piezo in a closed loop control. The piezo voltage should read zero.
11. Plug the BNC cable into the "control input". Introduce a 0-10 DC rising voltage to the piezo through the "control input." Before moving on to the next step, make sure the "control input" is 0 volts.
12. Connect top grip to bottom grip by a stiff piece of Si (clamp in the grips). This will allow you to measure how much the piezo is actually moving by monitoring the displacement of the load sensors (for microtensile systems #1 and #2), or the output of the load cell (microtensile systems #3 and #4). You can also calibrate the displacement of the piezo translator to the grip capacitance sensor. For either situation, you have to infer that the load sensors or the grip sensor are absolutely correct.
13. Now, slowly introduce a 0-10volt DC rising voltage (like step 11) through the "control input". Stop every 5 or 10microns of displacement (in the load sensors) and adjust the "gain" potentiometer (pot. #8) on the piezo servo card so that the piezo reads the nominal displacement.

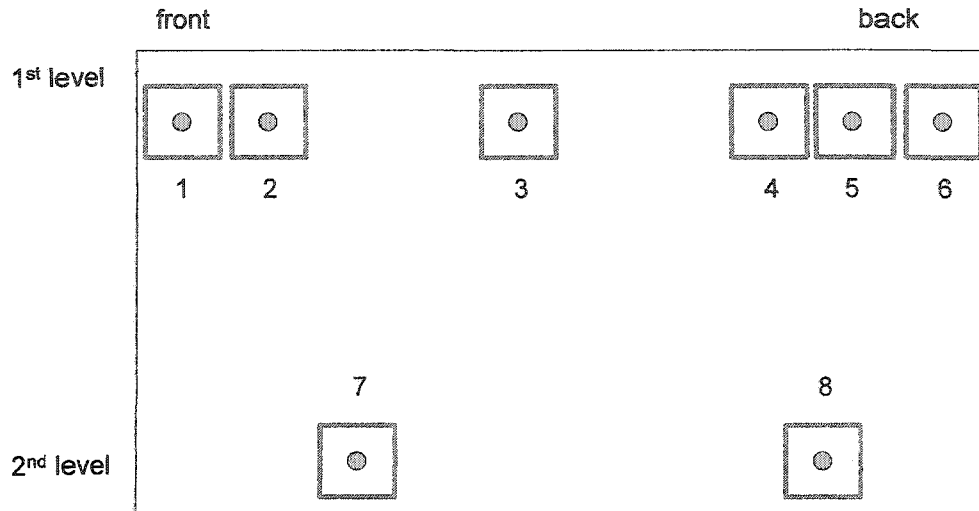


Figure A 1: Schematic of the servo controller card; note the locations of the potentiometers.

#1 and #2: Notch filter ("D" - differential in the PID control loop)

#3: "I" - integral in the PID control loop

#4: Feed forward gain

#5: "P" - proportional in the PID control loop

#6: slew rate $\left(\frac{dV}{dt}\right)$

#7: Adjustment for the zero potentiometer on the front of the piezo servo card (I'm not sure what this does, so if it is adjusted, I would suggest calling Polytec PI)

#8: GAIN – this is the potentiometer that needs to be adjusted.

Tuning the piezo translator:

1. Send a square waveform to the piezo translator through the "control input" BNC plug. Then, adjust the "P", "I", and "D" potentiometers until the piezo response is as close to matching the input square wave as possible.

2. After this step is done, a triangular waveform can be sent to the control input to simulate fatigue tests.
3. Also, frequency sweeps can be performed running from 0 – 60Hz and 0 – 100Hz. At 60Hz (according to a Polytec PI engineer), the amplitude drop should only be approximately 2.5% of the full scale. If you are unable to achieve this value, contact Polytec PI.

Materials used for the structural members of microtensile system #4

Top and bottom grips w/clamps – Type 316 stainless steel

Shoulder bolts – Type 316 stainless steel

Alignment block – Type 6061 aluminum

System #1 and #2 load cells – sensors are aluminum (from Polytec PI), blocks that secure the ends of the force sensing beam are type 316 stainless steel.

Remaining parts of system can be found in G. Cornella's dissertation: Side rails, top and bottom blocks are type 316 stainless steel. The adjustment portion of the top block is type 6061 aluminum.

Manufacturer's documentation

1. Polytec PI, Inc.

16 Albert Street
Auburn, MA 01501
www.polytecpi.com
Phone: 508.832.3456
Fax: 508.832.0506

2. Capacitec, Inc.

87 Fitchburg Road
P.O. Box 819
Ayer, MA 01432
www.capacitec.com
Phone: 978.772.6033
Fax: 978.772.6036
sales@capacitec.com

3. Honeywell Sensotec

2080 Arlingate Lane
Columbus, OH 43228
www.sensotec.com
Phone: 614.850.5000
Phone: 1.800.298.9228
Fax: 614.850.1111
sales@sensotec.com

4. Kistler Instrument Corp.

75 John Glenn Drive
Amherst, NY 14228
www.kistler.com
Phone: 716.691.5100
Phone: 1.800.kistler (547.8537)
Fax: 716.691.5226
sales.us@kistler.com

5. minus k Technology

420 South Hindry Avenue
Unit E
Inglewood, CA 90301
www.minusk.com
Phone: 310.348.9656
Fax: 310.348.9638
sales@minusk.com

6. Eurotherm Controls Inc. and Barber-Colman Industrial Instruments

741-F Miller Drive
Leesburg, VA 20175
www.eurotherm.com
www.barber-colman.com
Phone: 703.443.0000
Fax: 703.669.1300

7. Dell Inc.

One Dell Way
Round Rock, TX 78682
www.dell.com
Phone: 1.800.624.9897

8. National Instruments Corporation

11500 N. Mopac Expressway
Austin, TX 78759
www.ni.com
Phone: 888.280.7645
Fax: 512.683.8411

Vita

Paul Andrew El-Deiry, son of Adel and Lorraine El-Deiry, was born on October 20th, 1977. He attended Banner Elementary Grade School in Dunlap, IL from 1st through 6th grades. He then moved on to Pioneer Junior High School in Dunlap, IL for 7th and 8th grades. He graduated 8th grade in June 1991. From there, he attended Dunlap High School located in Dunlap, IL and graduated in June 1995. Throughout his tenure at Dunlap High School, he was active in the marching band (drums), orchestra (percussion), jazz band (piano), pep band (percussion), and played on the varsity tennis team all four years. In the summer of 1993, he attended Wolfeboro Camp School in Wolfeboro, NH where he took classes in Algebra II and English. In the summer of 1994, he attended Cornell University summer school in Ithaca, NY where he took classes in Pre-calculus and English. After graduating from Dunlap High School in 1995, he attended Lehigh University in Bethlehem, PA. In his first semester at Lehigh, he walked-on to the NCAA Division I varsity tennis team and continued as a team member for all four years. During the summers of 1998 and 1999, he worked in the Microelectronics Division at Lucent Technologies in Allentown, PA. In June 1999, he received a Bachelors of Science (B.S.) degree in Materials Science and Engineering. He then continued at Lehigh University for graduate studies where in January 2004 he received his Masters of Engineering (M.E.) degree in Materials Science and Engineering with a focus on the mechanical properties of bulk metallic glasses. In May 2005, he received his Doctor of Philosophy (Ph.D.) degree in Materials Science and Engineering with a focus on the mechanical properties of aluminum thin films. Throughout the final four years (2001-2005) of his graduate school career, he attended Lehigh Valley Taekwondo and on November 5th, 2004 he received a red belt.