Development of Microtesting Systems: I. Tensile Testing of Metallic Microsamples

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Abstract

This paper describes procedures adopted for the development of microtesters, including microtensile and microfatigue testing systems. Small structures are known to exhibit superior mechanical properties attributed to their finer microstructure. This includes stress-life response at low and high cycle fatigue. The fatigue life of small structures involves crack initiation and crack propagation stages. While the crack initiation stage is known to be a significant component of fatigue life, experimental verification of its impact has not been performed. Using the compliance response of small LIGA Ni structures in load control mode and under cyclic loading conditions, the contributions of crack initiation and propagation to fatigue life were quantified. Instrumentation details of the development of microtesters, including the use of PID functions of Labview®, IMAQ Vision®, a piezoelectric actuator, and the results of fatigue tests will be discussed.

Introduction

The advent of micro- and nanotechnology and their emerging applications in various fields has prompted actions on the testing and characterization of such materials and devices used in micro and nanoelectromechanical systems (M/NEMS). The mechanical properties of materials have been shown to be different at the micro- and nano-scale, as compared to those at the macro-scale[1]. Variation of properties, across scales, originates mainly from the difference in microstructure; however, there are other contributing factors, such as size effect. Moreover, the mechanisms of failure at the micro-scale may differ from those at the macro-scale purely due to dimensional differences. For example, the fatigue crack growth component of fatigue life has been shown to be orders of magnitude smaller than that of the crack initiation component.

With the development of new microfabrication techniques, reliability becomes a critical issue for microdevices that have applications where human lives are at stake. Microdevices used in triggering weaponry systems, or those used in life-supporting devices are examples of such applications. Factors that affect reliability of such devices and structures must be explored to help optimize their design and manufacturing processes. Since properties do vary across scales, mechanical tests must be performed on structures that are used in real-size applications, e.g. at microscale. Development of microtesting systems is based on such a need. It addresses a range of issues such as micromechanical behavior of small structures, mechanisms operating at micro- and nanoscale, and helps provide mechanism-based models that can be used in robust design of micro- and nanodevices.

Microtesting of metallic and ceramic samples discussed here include the development and use of tensile, fatigue and room temperature creep testing systems that characterize mechanical properties of samples for which one or more dimensions are at micron scale. Instrumentation of microtesting systems used in configurations such as monotonic or cyclic loading in tension, compression, bending or indentation will be discussed.

Microtensile properties of MEMS structures

Instrumentation of microtensile testing systems entailed bringing together a number of subsystems including a drive mechanism for generating load, a load measurement unit, a displacement measurement subsystem, and their related controls. Fig. 1 shows the schematic of the microtensile tester developed after an original design from Johns Hopkins University[2] which was modified to accommodate measurement for large strains. The load train started with a motorized Velmex (Velmex , Inc., Bloomfield, NY 14469) unislide drive equipped with a gearbox of 1500:1 ratio, connected to a linear stage providing strain rates as low as 10^{-4}/s. As with nearly all variable speed motors, lower speeds could be achieved, however, load capacity dropped below the desirable levels. An "L" bracket machined and installed on the unislide drive made it possible to mount a micro-loadcell from Entran (Entran Devices, Inc. Fairfield, NJ 07004-3877) with capacities from 100 g to 22.4 kg in series with a Nelson (Nelson Air Corp., Milford, NH 03055) frictionless air bearing.

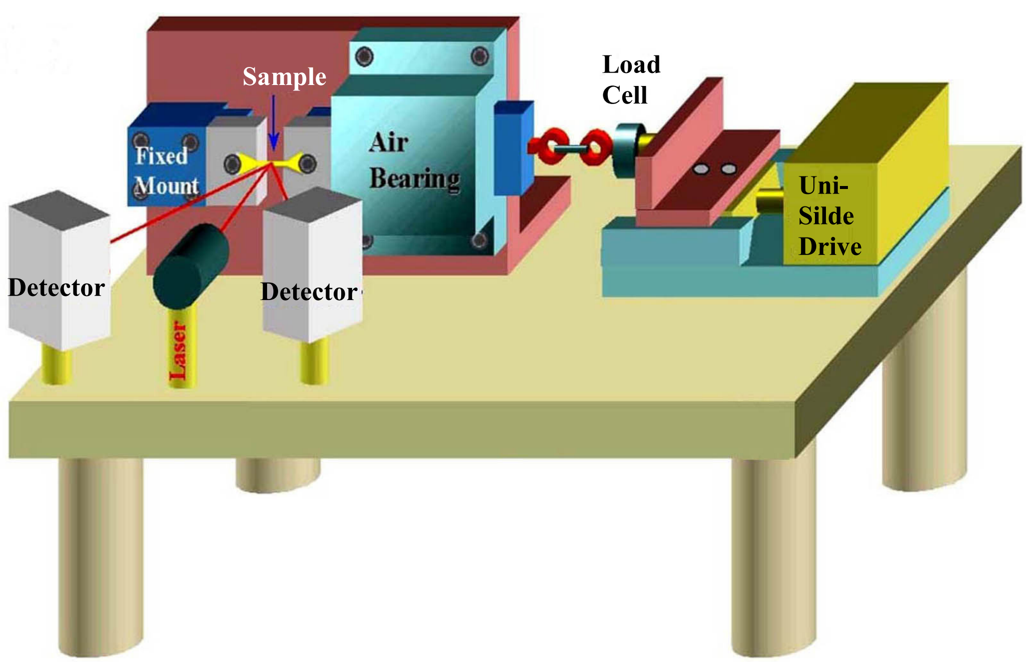

Fig. 1- Schematic of the microtensile testing system developed after Sharpe et al.[2]

The gas-powered, air bearing provided frictionless straining of the sample at a nitrogen pressure of about 40 psi. The sliding ram of the bearing was equipped with a specially made grip with a recessed area that matched the shape of the triangular end of the microsample. The depth of the recessed part was similar to that of the thickness of the sample. When sample's triangular end was embedded onto the recessed area of the grip, a washer overlapped part of the sample's grip area, keeping it in place during the test. The other end of the microsample was fitted into the recessed area of a second grip that was mounted on a fixed platform. Both the stationary platform and sliding grip were machined at the same time with the same mill bit so that perfect alignment was achieved. Further, the grips were made from one single plate, with the negative of the microsample carved out of it (recessed area). The single plate was then mounted on one side on the sliding air bearing ram, and on the other side on the stationary platform. After mounting screws were installed, pins were used to perfectly align the grips on their corresponding seats. Once the pins were in place, the single grip plate was cut into two halves right at the middle of the recessed area designed to hold the microsamples.

Several types of samples were tested by the microtensile tester, including Al and FeCrAlY samples fabricated by electro-discharge machining (EDM). The EDM was performed using a Uni-tek (Uni-tek Manufacturing Company, Frankfort, IL 60423) US-EDM Model D-55 system along with a special microsample-shaped copper die. The EDM samples were made by cutting a dog-bone shape sample out of Al or FeCrAlY alloy (bond coat material used for thermal barrier coating application) in a method similar to cookie cutting.

Microtensile LIGA Ni samples were of dog-bone shape (Fig. 2), and were supplied by Sandia National Labs of Albuquerque, NM. LIGA Ni samples were made by electrodeposition in PMMA molds realized by deep X-ray lithography[3], from a sulfamate bath with a current density of 50 mA/cm^2. SEM images on the cross section of the samples showed a columnar structure with grains that had a diameter of 5 μm and a length in the range of 5-25 μm.

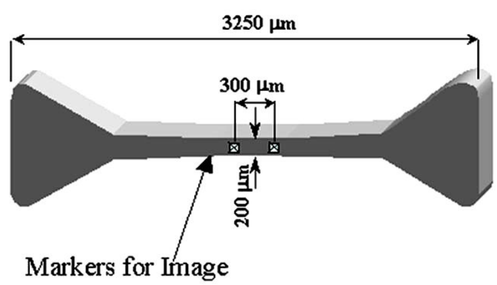

Fig. 2- Schematic of the dog-bone-shape LIGA Ni sample with thicknesses of 70 and 270 mm.

Dog-bone shape samples used for the microtensile studies had a gage length of 1000 μm, a width of 200 μm and thicknesses: 70 and 270 μm. The microsamples were designed such that failure would occur only at the middle 400 μm length of the sample. This was achieved by slightly tapering the sides of the gage area (Except for the uniform middle area) with a 10° inclination angle.

Strain measurements were performed in two ways: Laser interferometry and Vision-Based landmark matching. While the first method is more accurate at initial stages of deformation (e.g. elastic deformation), accurate measurement becomes challenging at large strains. The reason is roughening of the reflecting surfaces of the sample due to formation of slip/deformation bands. It is clear that for large strains; a different method must be employed that is less sensitive to the quality of the sample surface. Vision based landmark mapping may offer a better solution, as it finds and records coordinates of landmarks on the surface irrespective of their reflectivity.

To perform strain measurements by either method (interferometry-or vision-based), special markers were placed on the surface of the samples [4-6]. These markers were placed at a distance of about 300 μm. A microhardness tester was used to indent the surface of the sample using a pyramid shape diamond tip. The size of the indents was about 25 μm and the depth was about 4 μm. The indentation created inverted pyramids into the samples with reflecting surfaces that made interference of the reflected beams possible.

The surfaces of the samples were polished down to a mirror finish using colloidal silica prior to indentation. This assured high quality reflecting surfaces generated by indentation Samples were glued to a flat surface using crystal bond and later mounted on an x-y-twist combination stage of the microhardness tester. These indents were not only used for interferometry but also for landmark matching utilized in vision-based strain measurements. Occasionally the indenter was allowed to scratch the surface of the sample creating a groove. The scratching was achieved by moving the stage while the indenter was in contact with the surface of the sample. A pair of these grooves usually produced stronger interferometry patterns compared to a pair of square indents.

Both sides of the samples were indented. One pair of indents was used for laser interferometry while the other pair was used for image analysis. The platform on which the samples were tested was machined for a viewing window to expose the backside of the sample during deformation. While the back side of the sample was used for laser interferometry, the front side was photographed during the test. A SONY (Sony Corporation of America, New York, NY 10022), DFW-SX900 digital CCD camera was used to take images with resolutions as high as 960x1200 pixels. The camera was controlled by the Labview VI to select the area of interest, shutter speed, number of frames per second and time stamp for each image.

For detection of interferometry patterns, two photo-diode-array detectors were used at a distance of about 30 cm from the sample. Each array consisted of 512 pixels detecting about 10 pairs of light and dark bands. The voltage signal generated by the detectors was analyzed for periodicity using Fourier transform function of a Labview VI especially developed for this purpose. The change in the periodicity was then related to the local elongation of the sample during tensile loading.

The vision based measurement consisted of taking images of surface landmarks or surface features that persisted over a number of pictures taken from the sample during the test. Even if these features are transient and persist over a short period of time, they can be utilized to calculate local strains for that time period. Mapping of local strains will be possible if this process is performed for landmarks scattered over the area of interest. For this study images were obtained with frequencies of 0.1-5 Hz generating 6-300 images per minute.

Data and image acquisition was performed using Labview (Labview, IMAQ Vision and Vision Builder are trademarks of National Instruments, Austin, TX 78759), IMAQ Vision and Vision Builder to control software and hardware. Strain rate was selected manually at 5×10^{-4} resulting in a displacement rate of about 0.6 μm/s. Load cell data was recorded by a data acquisition card, DAQ from National Instruments, with a frequency of about 1000 data points per second which was later was reduced in number to match the number of data point obtained by laser interferometry or vision-based image analysis. Image analysis could be performed on the fly; however, it would greatly slow down the process. To avoid this, image analysis was performed post-mortem, correlating the image and load data results using time stamps associated with the two acquisition processes.

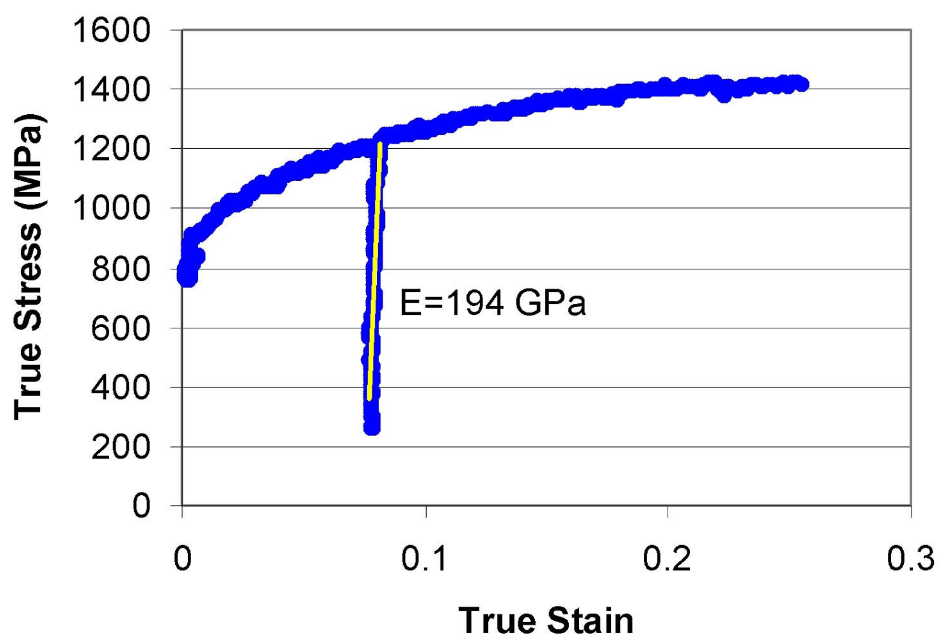

Fig. 3- Stress strain curve obtained by microtensile testing of dog-bone shape samples extracted from FeCrAlY foil.

The results of the tensile testing of the FeCrAlY microsample are presented in Fig. 3. In order to find Young's Modulus, the sample was unloaded and reloaded as seen from the stress-strain curve. A modulus of 194 GPa was obtained for the specimen. The yield strength and UTS were about 900 and 1400 MPa respectively. The results of microtensile tests performed on the thick and thin LIGA Ni samples are reported in Table 1. For comparison purposes, results of tensile testing of bulk samples are also reported in Table 1. Much higher strengths were obtained for our microsamples compared to bulk materials that have larger grain sizes. Tensile properties of materials are not expected to change with the size of the specimen. Nevertheless, the microstructure of these structures will have an effect on the mechanical properties such as yield strength, fracture strength, fatigue and creep. Detailed results of stress-strain curves, fracture surface analysis, electron microscopy of LIGA Ni samples tested by microtensile testers are discussed elsewhere [7-10].

Table1. Comparison of LIGA Ni Properties with Bulk Ni

Thickness/Strength	70-μm	270-μm	Hemker [15]	Others [16, 17]	Bulk Ni [18]
Yield Strength (MPa)	630	435	150	441	59
UTS (MPa)	735	575	170	555-2470	

The difference in the microstructure of small and large scale structures originate mainly from the fabrication methods. As an example, polysilicon structures have a fine nanoscale structure with grains that are a few tens of microns. While bulk specimens are usually polycrystalline and gage section of the specimen consists of many grains, this is not the case for small structures. When small structures are extracted from bulk samples, the entire small structures can fall within a few grains. It is now possible to make small samples (e.g. a dog-bone shape specimen) shown in Fig. 2 for which the entire gages section consists of one grain. This effectively leads to measuring mechanical properties similar to those of a single crystal.

Fatigue properties of LIGA Ni MEMS

The main objective in the fatigue testing of LIGA Ni Samples was to study the mechanisms of crack nucleation and crack propagation under cyclic loading. It was also desired to establish relative fractions of fatigue crack initiation versus crack propagation life. These findings would help develop mechanism-based mechanics models that would predict fatigue life in LIGA Ni MEMS components.

The samples used for fatigue testing of LIGA Ni had the same shape and geometry as those used in our microtensile testing experiments presented in Fig. 2. Both thick (70 μm) and thin (270 μm) samples were used under cyclic loading conditions with a frequency of 10 Hz in tensile-tensile fatigue with a load ratio of 0.1. The details of microfatigue tester are discussed here.

The microfatigue system used for testing our dog-bone shape samples is shown in Fig. 4. At the heart of the tester lies a Polytec PI piezo actuator with a 180-μm travel range. This is a high voltage linear actuator with ultra-high resolution made of multiple thin PZT ceramic disks. The pushing force on these actuators is 4500 N and pulling force is 500N. Response of the actuator is sub-millisecond making it possible to actuate it with frequencies of up to 1000 Hz. This actuator was mounted in series with a l0-lb loadcell from Entran for load measurements. The load cell was connected to an end piece having recessed triangular grip area similar to that of the microtensile tester mounts. One side of the microsamples was mounted in the grip area of this end piece while the other end was mounted in a similar end piece connected to an x-y rotational stage fixed to the optical table.

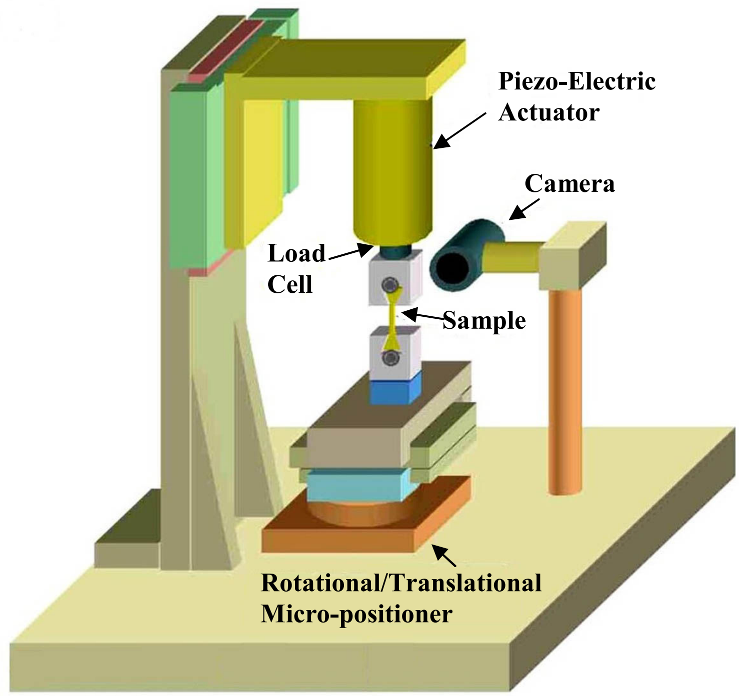

Fig. 4- Schematic of the microfatigue testing system used in this study

The piezo actuator was mounted on a Newport motorized stage for quick z-adjustments. The motorized stage was capable of micron-resolution motion with jog or continuous motion capabilities. Alignment of the sample was achieved with an optical scope mounted in front of the tester, imaging the specimen, measuring its displacement. The images were used to calibrate and confirm the extent of displacement of the sample as well as calibrating the position signal obtained from the PI control box. The data from the load cell were recorded along with the images of the sample and the position monitor signal from the PI control box. Data were recorded by the data acquisition (DAQ) board of the National Instruments using Labview as well as image acquisition u software of IMAQ vision and Vision Builder. Images were captured using SONY SX-900 camera for displacement measurement/calibration purposes.

All fatigue tests were performed in load control mode. This was achieved by developing a Labview VI using PID functions built in the software. The feedback signal from Loadcell was used in order to regulate the control signal that fed the piezo actuator control box. The piezo controller was energized by a signal between 0-5 volts which then was amplified in the range of 0-1000 V before being sent to the piezo actuator. When this signal is sinusoidal, piezo will oscillate similar to a reciprocating jig saw. Maximum and minimum loads were controlled by the displacement of the actuator relative to its mean position.

The Labview VI that was developed for microfatigue testing generated a sinusoidal signal between 0-5 volts which was used to control the piezo actuator. While the frequency of this signal was maintained at 0.1 Hz, the amplitude and the mean value were adjusted by the PID loop in the Labview PI. The feedback signal from the loadcell was forced to conform to a designed sinusoidal signal generated by the PID function through adjustments of the actuator's mean position and its displacement amplitude. The designed sinusoidal load signal was generated using a 0.1 Hz frequency, desired levels of maximum load, and the load ratio. Although for initial quarter wavelength, the desired sine path was not exactly followed, the rest of the signal was closely followed.

The adjustment of the load, achieved by moving the actuator, made changes to the mean position of actuator over the fatigue life span of the sample. As the sample deformed, its compliance changed. It became necessary to adjust the displacement in order to achieve same level of load. For an un-cracked specimen, the change in the compliance, and consequently the change in the mean position of the actuator, is small. However, when a crack was initiated, compliance changed significantly. This was captured by the significant change that occurred in the mean position of the actuator. The change in the displacement rate of the mean position stands out as a landmark in the displacement vs. life cycles curve marking the onset of crack growth.

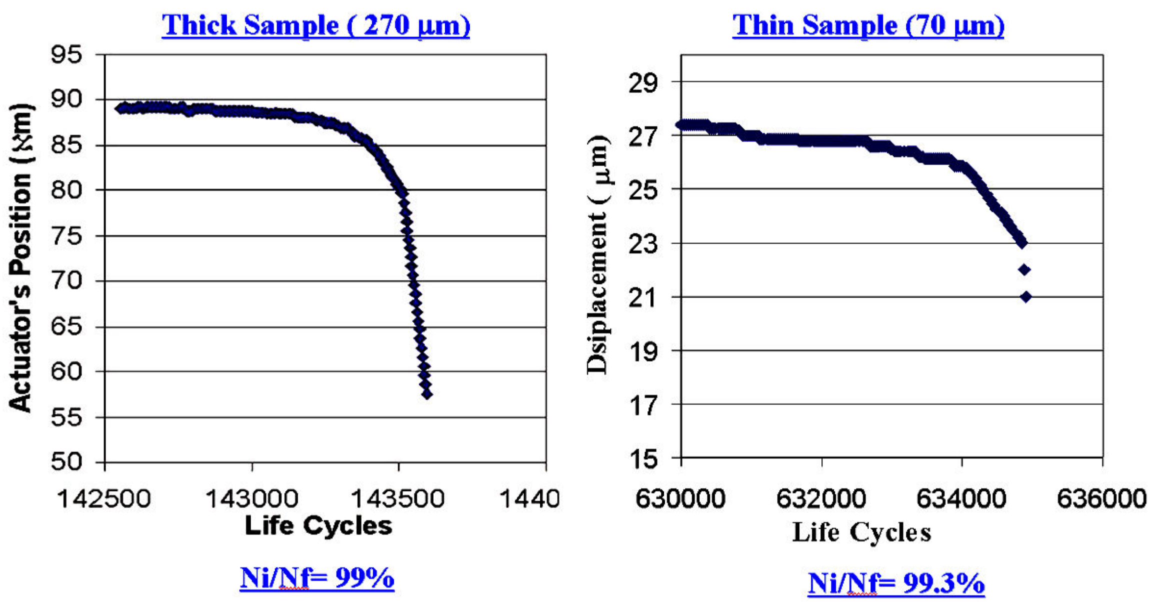

Fig. 5- Crack initiation and crack propagation components of life captured by the change in compliance of the sample resulting in a change in the rate of motion of the actuator

For LIGA Ni samples this method was used to quantify the contributions of fatigue crack initiation and fatigue crack propagation to the overall fatigue life of the sample. For both thin and thick samples, fatigue crack initiation stage constituted 99% or more of the fatigue life. This is clearly seen from Fig. 5 for thick and thin LIGA Ni microsamples. Samples with life cycles shown in Fig. 5 were examined in an FEI XL-30 scanning electron microscope. All samples had cracks that had grown to some extent depending on their location on the compliance curve of

Fig. 5. This clearly proves the usefulness of compliance method in determining the contributions of each one of the crack initiation and crack propagation components to the total fatigue life. Detailed results of microfatigue testing of LIGA Ni samples are presented elsewhere [11, 12].

Fatigue Properties of Foam Struts

Metallic foams have been of special interest for a variety of applications including light weight structural components subjected to dynamic loads. Since open cell foam structures consist of arrays of cells formed by ligaments, it is always desirable to model mechanical behavior of foams based on micromechanical properties of the ligaments (struts). The dimensions of these struts are of micro-scale making it necessary to test them with microtensile testers. In order to achieve this, the microtensile tester was equipped with the special grips shown schematically in Fig. 6. These grips allowed mounting of microsamples extracted from aluminum open cell foams.

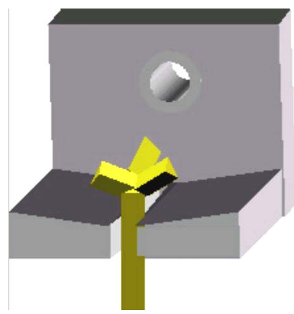

Fig. 6- Schematic of the grips used for the microtensile and microfatigue testing of Al foam struts

Details of the tests are similar to that of LIGA Ni samples except for the fact that laser interferometry was not used. The rough surfaces of the ligament that are fabricated by investment casting process could not be polished without damaging the struts. Further, indentation of these surfaces without bending them was not possible. It was concluded that vision-based measurement was the only practical solution to obtain strains. Since the surfaces were rough, surface features could be used as landmarks. However, foam struts were marked with a focused ion beam microscope for landmark matching. This was performed using an FEI (FEI Company, Hillsboro, OR 97124 USA) Strata TM DB-235 dual beam focused ion beam microscope. Results of microtensile testing of the struts are reported elsewhere[13].

In addition to tensile strength, fatigue behavior of foam struts is also of interest. Fatigue properties of struts shed light into the mechanism of fatigue crack initiation and fatigue crack propagation of foam blocks. It may also help develop mechanism based models that predict the fatigue life of open cell metallic foams under various load levels and loading conditions. Three types of open cell struts were extracted from Al foam blocks in as-received, annealed and annealed and aged conditions. The composition of the alloy is presented in Fig. 7. Foam struts were extracted using EDM machining from the foam blocks.

Specimen	Cu	Mg	Mn	Si	Fe	Zn	B	Al
Foam	0.03	0.29	.01	0.25	0.14	0.01	0.03	99.22

Fig- 7. Chemical composition of Al foam struts

The microfatigue testing system used for LIGA Ni samples were slightly modified to test Al foam struts. The fatigue tests were conducted under load control using the feedback signal of a load cell and the actuator position data from the position monitor of the piezoelectric actuator. Samples were mounted on the same special grips (Fig. 6) used for microtensile loading of foam struts. A series of images taken from a foam strut during deformation are shown in Fig. 8. It is interesting to note that deformation starts at lower half of the strut Fig. 8(a-b); nevertheless, the main fracture gets initiated (Fig. 8-c) and proceeds in the upper half of the strut (Fig. 8d-f). The results of fatigue testing of these aluminum foam struts are presented elsewhere [14].

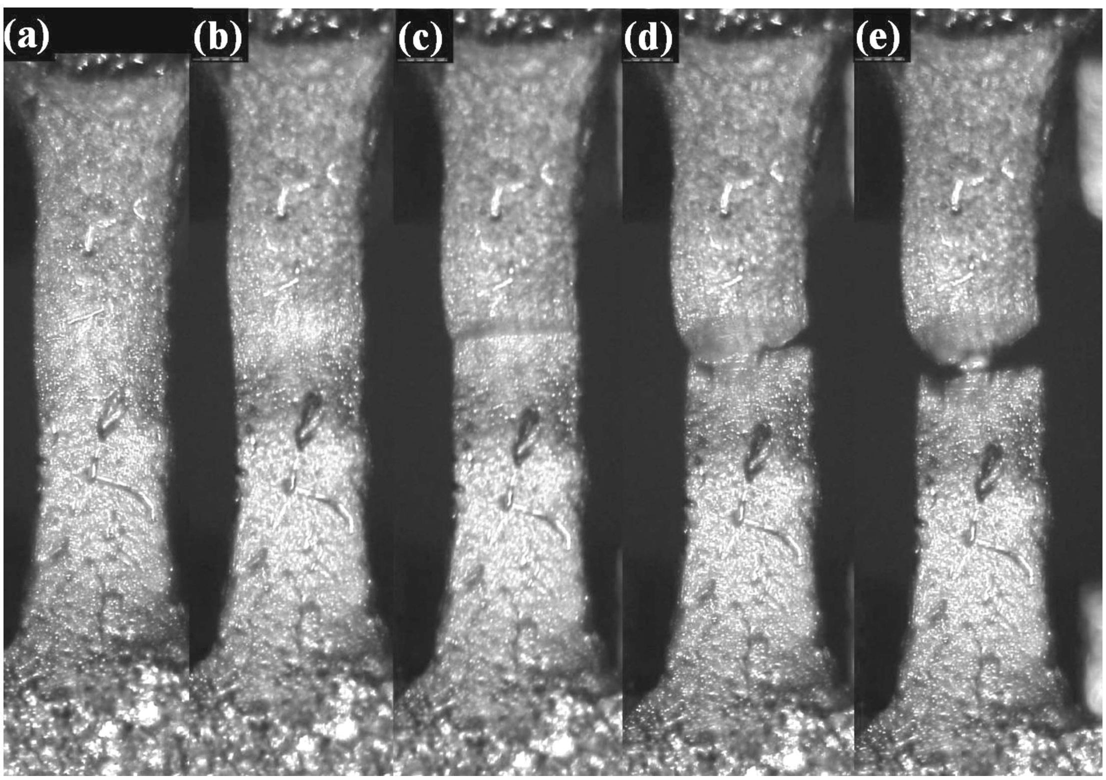

Fig. 8- Deformation of a foam strut during fatigue. The thickness of the sample is about 400 mm

Conclusions

Microtesters were developed to evaluate micromechanical properties of metallic materials in tension under monotonic and cyclic loading conditions. The microtensile tester used laser interferometry and vision-based metrology to determine displacements. The microfatigue tester used a piezoelectric actuator for cyclic loading of the sample resulting in frequencies of up to 1000 Hz. The change in the mean position of the actuator marked the onset of fatigue crack propagation in the sample. Results of tests performed with the microtensile testers were consistent with the results obtained by other investigators using similar materials. The novel finding of this study is the ability to determine the fatigue life components due to fatigue crack initiation and fatigue crack propagation.

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