

A Review Study of Mechanical Fatigue Testing Methods for Small-Scale Metal Materials

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ABSTRACT

A review on the mechanical characterization of materials at small- scale is presented. The concentration is on the different micro- and nano-scale testing techniques, classify, summarize, and compare all of these techniques that are currently available. The variety of materials investigated by the scientific and industrial communities and the mechanical quantities identified by such methodologies. Finally, the perspectives of studies on fatigue of the small scale metal materials are offered.

Keywords: Fatigue crack ; small-scale; nano-scale; MEMS; cyclic load; thin film.

Introduction

The past decade has seen the rapid growth of micro-electromechanical systems (MEMS) as an important area of technology, growth which is expected to continue well into the next century. The basic premise behind the concept of MEMS is that the efficiencies of high volume production and low unit cost achieved by the microelectronics industry over the past 50 years can be translated to devices in which mechanical and electrical components are integrated within a single silicon chip (or equivalent structure).

Small-scale components often have to withstand cyclic loads during their service life. Free-standing films, usually used to fabricate functional and/or structural components in MEMS, are subjected to mechanical fatigue loading at different frequency in different kinds of environments. Thin films on a substrate, widely used for fabricating microelectronic devices and integrated circuits, are subjected to temperature variation during service unavoidably leading to fatigue damage. In any case, fatigue causes continuous change in the properties of the material, which can be a cause of a possible failure mechanism. A knowledge of the dynamic properties of materials is a key issue for correct engineering design. During the last decades, many efforts have been made in order to develop fatigue tests suitable for micro- and nano-scale investigation. Fatigue tests are performed to characterize fatigue behavior of small-scale samples in terms of stress versus number of cycles to failure (S-N curves), fatigue damage, and fatigue crack initiation and growth (see figure 1). Metal materials were widely investigated, but also micrometer-sized specimens of both single-crystal and polycrystalline silicon have attracted attention [1-3].

Alsem *et al.*[4] carried out fatigue tests on resonator poly-Si specimens and confirmed the occurrence of cyclic stress assisted oxidation using high voltage transmission electron microscope. Also, all the tests showed a distinct independence of fatigue strength and number of cycles to failure from loading frequency [5], indicating that it was not merely stress corrosion cracking that was playing a role because then the time to failure would be constant irrespective of the frequency of loading.

Kahn *et al.*[6] suggest sub-critical cracking in the Si itself as the driving mechanism for cyclic failure in these specimens whereas Muhlstein *et al.*[7] have proposed reaction layer fatigue process wherein the cracking of the native SiO₂ scale covering the Si structure actually causes damage accumulation and drives fatigue failure.

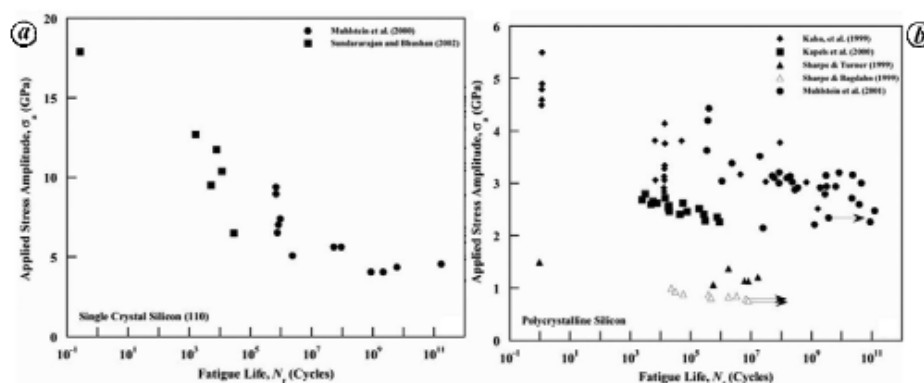


Fig.(1): Fatigue life determined by various research groups in (a) single crystalline and (b) poly-crystalline Si112.

Both tension-tension and tension-compression modes were used. Apparatus for fatigue tests under tension-tension loading were developed for testing thin metal wires [8-9], metal [10-14] or silicon [15] freestanding films or foils, while metal films deposited onto a substrate can be tested by tension and compression loading modes using the test method proposed in Ref. [16]. Tension compression cyclic loading tests can also be carried out on thin free-standing films.

Muhlstein *et al.*(2002) have provided a fatigue failure map for thin film structures showing the critical oxide thickness vs. applied stress, clearly distinguishing domains of no cracking and sub-critical cracking as safe zones and reaction layer fatigue, as responsible for final failure (Figure 2). In thin films, the critical crack length required for device failure is less than the oxide layer thickness, which is why delayed failure becomes possible.

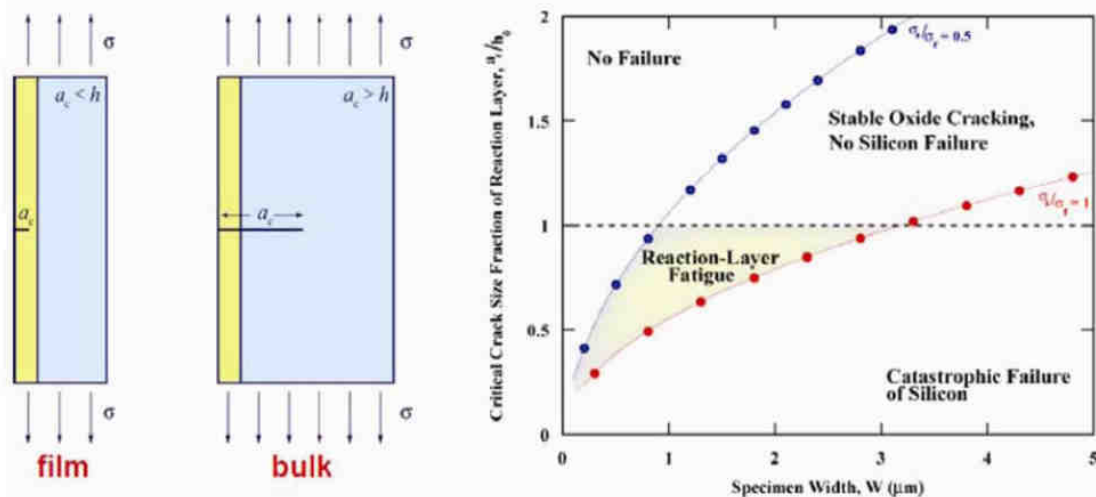


Fig. (2): Fatigue–fracture map in Si thin film systems showing domains of critical film thickness beyond which catastrophic fracture precedes fatigue failure[17].

Fatigue Damage Behaviors

For a thin metal film, there exist two length scales corresponding to film thickness and grain size. If the in-plane grain size of the film is larger than the film thickness, the film should have a columnar grain structure, while if it is less than the film thickness, the film may have an equal-axial grain structure. Fig. 3 presents an illustration of the relationship between film thickness and grain size of the thin Cu films investigated previously[18].

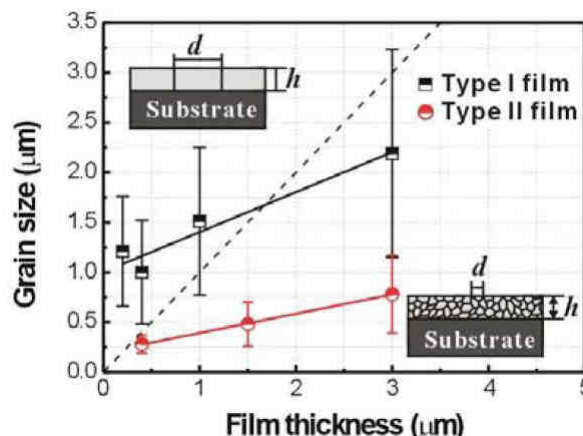


Fig. (3): Schematic illustration of the relationship between film thickness and grain size in the thin films studied by Zhang et al.

A comparison of the ratio of the extrusion area to the grain area in fatigued 200-nm-, 400-nm-, and 3.0- μ m-thick films as measured using plan-view a focused ion beam (FIB) images is presented in Fig. 4. Because the grain sizes for the three films did not vary strongly with film thickness (1.21, 1.00, and 1.45 μ m are the average grain sizes for increasing film thickness), the trend illustrated in Fig. 5 also applies to the extrusion widths. The significant decrease in the extrusion width with decreasing film thickness from 3.0 μ m to 200 nm clearly indicates that the local accumulation of cyclic plastic strain by the formation of extrusions/intrusions becomes more difficult with decreasing film thickness and is almost completely hindered in the 200-nm-thick films[19].

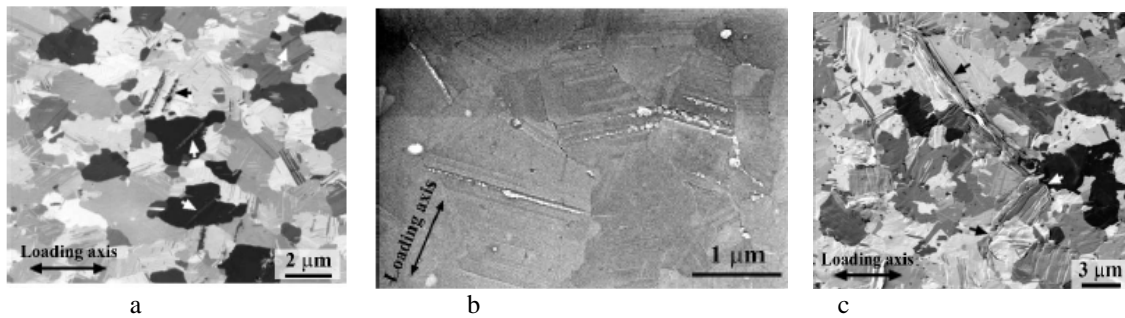


Fig. (4): Fatigue damage at the surface of the Cu thin films: (a) infrequent and narrow extrusions in the 200-nm film after 2×10^4 cycles (imaged by FIB at an angle of 45°); (b) cracking along twin and grain boundaries in the 200-nm film after 1×10^4 cycles (imaged by SEM); (c) extensive extrusions and cracking along extrusions/intrusions and at the intersection of extrusions with GBs in the 3.0- μm -thick Cu film after 5×10^3 cycles (imaged by FIB at an angle of 45°).

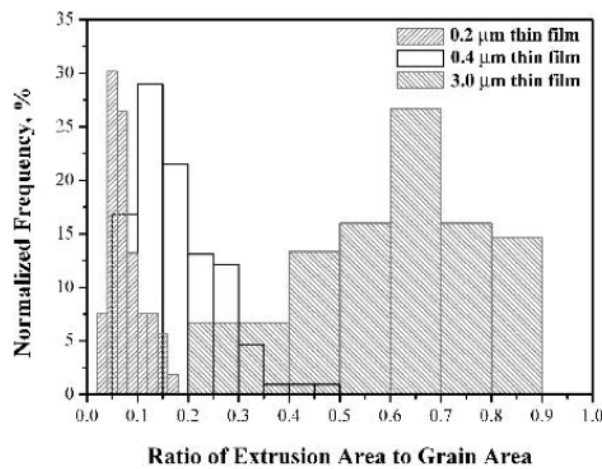


Fig. (5): A comparison of the ratio of the extrusion area to the grain area in different film thicknesses of 3.0 μm , 0.4 μm , and 0.2 μm .

The Micro and Nano Scales Effects

It has been known for quite some time that materials and structures with small-scale dimensions do not behave in the same manner as their bulk counterparts. This aspect was first observed in thin films where certain defect structures were found to have deleterious effects on the film's structural integrity and reliability. This became a significant concern because thin films are routinely employed as components in microelectronics and micro-electro-mechanical systems (MEMS).

Material mechanical properties are fundamental input parameters for structural design of micro- or nano-devices. The accuracy of numerical modeling and simulation results depends on the accuracy of the material properties provided as input. For this reason, during the last decades many suitable techniques have been developed to improve the measurement accuracy and/or to increase the number of the determinable mechanical quantities. We present a classification of these techniques in Fig. 6 [20].

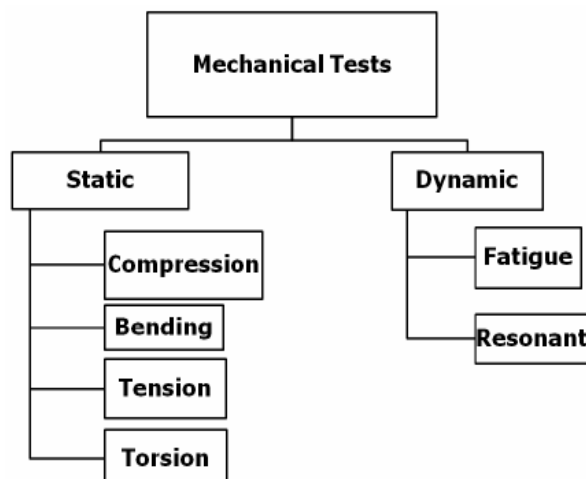


Fig. (6): Classification of the test methods for mechanical characterization of materials at micro- and nano-scales.

Several pioneering studies have experimentally identified the existence of size effects on the plasticity of metals. These studies were able to obtain experimental nano-indentation data showing a strong size effect as evidenced by material hardness decrease as indentation depth increases [21–23]. Figure 7 is a reproduction of the data from Ma and Clarke of nano-indentation performed on epitaxially grown silver on sodium chloride and shows that the hardness increases by a factor of two to three as penetration depth decreases. Their results have been verified and expanded in subsequent studies [24–31].

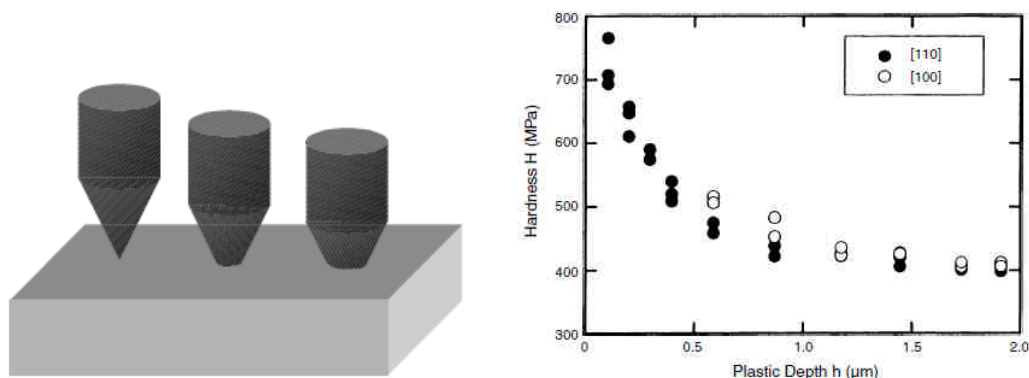


Fig. (7): Plot of hardness vs plastic depth illustrating how hardness increases with smaller plastic depth.

Reprinted with permission from [18], Q. Ma and D. R. Clarke, *J. Mater. Res.* 10, 853 (1995).
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The variation of flow stress as a function of grain size from the micro-crystalline (mc) to the nano-crystalline (nc) regime, which is schematically shown in Fig. 8. In many mc and ultrafine crystalline (ufc) metals and alloys with average grain size of 100 nm or larger, strengthening with grain refinement has traditionally been rationalized on the basis of the so-called Hall-Petch mechanism [32–34]. Here pile-up of dislocations at grain boundaries is envisioned as a key mechanistic process underlying an enhanced resistance to plastic flow from grain refinement. As the microstructure is refined from the mc and ufc regime into the nc regime, this process invariably breaks down and the flow stress versus grain size relationship departs markedly from that seen at higher grain sizes. With further grain refinement, the yield stress peaks in many cases at an average grain size value on the order of 10 nm or so. Further decrease in grain size can cause weakening of the metal. Although there is a growing body of experimental evidence pointing to such unusual deformation responses in nc materials, the underlying mechanisms are not well understood. Consequently, there is a concerted global effort underway using a combination of novel processing routes, experiments and large-scale computations to develop deeper insights into the various aspects of these phenomena.

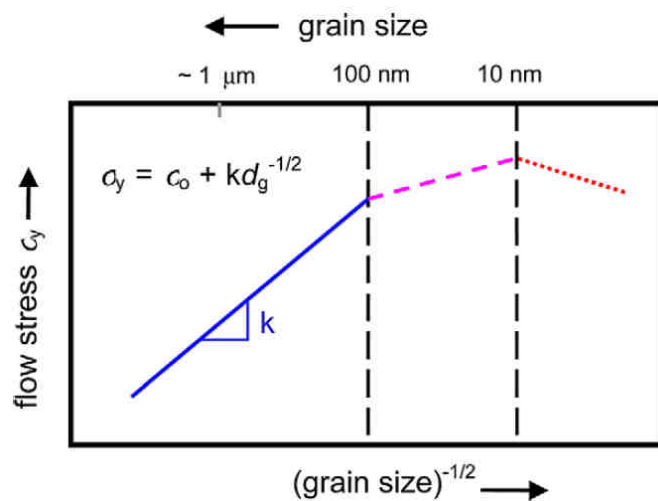


Fig. (8): Schematic representation of the variation of yield stress as a function of grain size in mc, ufc and nc metals and alloys.

Micro-tensile testing

Micro-tensile tests were performed on the dog-bone specimens (Fig. 9). The tests were carried out under displacement control, using an Instron Model No. 5848 Micro tester (Instron, Canton, MA). The specimens were deformed continuously to failure at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. The local strains/displacements were monitored using an in situ video camera with a $3000\text{pixel} \times 4000\text{pixel}$ resolution. The incremental images of gauge section deformation and the loads were monitored with an automated image acquisition system[35].

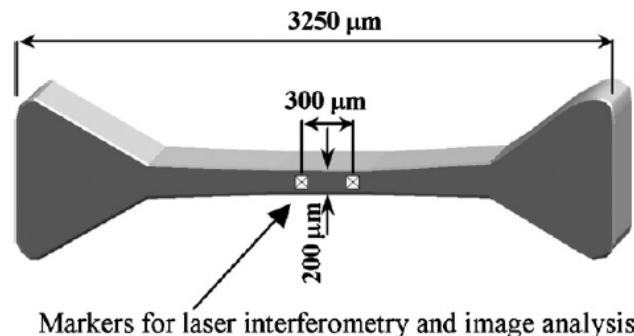


Fig. (9): Schematic of the dog-bone shape sample used for micro-tensile and micro-fatigue testing experiments.

Micro-fatigue testing

The (Lithographic, Galvanoformung, Abformung) LIGA fatigue samples had the same dog-bone shape as the micro-tensile samples (Fig. 3). The tests were performed under load control at a cyclic frequency of 5 Hz. The load and cross-head displacement data were recorded continuously along with the number of cycles to failure.

In situ fatigue experiments were performed in a micro-fatigue testing system that was developed at Princeton University [36]. These were used to monitor surface crack growth and incremental specimen deformation during fatigue. A photograph of the micro-fatigue system is shown in Fig. 4a. A schematic of this system is also presented in (Fig. 10).

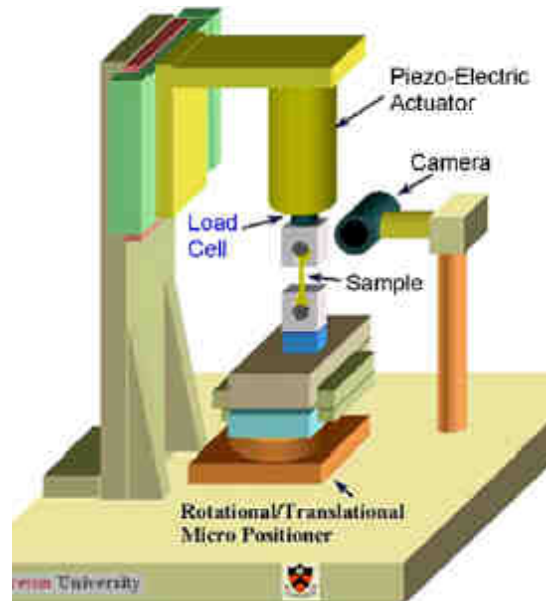


Fig. (10): the schematic of the micro-fatigue testing system.

Fig. 11 shows a plot of engineering stress vs. displacement for all the specimens of nano-twinned copper (NT Cu). To normalize the force variations due to sample thickness variations, nominal engineering stress (P/Bw) is used instead of the loading force P . Fig. 12 shows a plot of the change in crack length vs. the number of cycles when multiple load-controlled cycles with an initial ΔK of $6 \text{ MPa}\sqrt{m}$ are imposed on the specimens. This leads to the inference that a decrease in twin lamellar spacing leads to smaller crack length change over the full range of cycles[37].

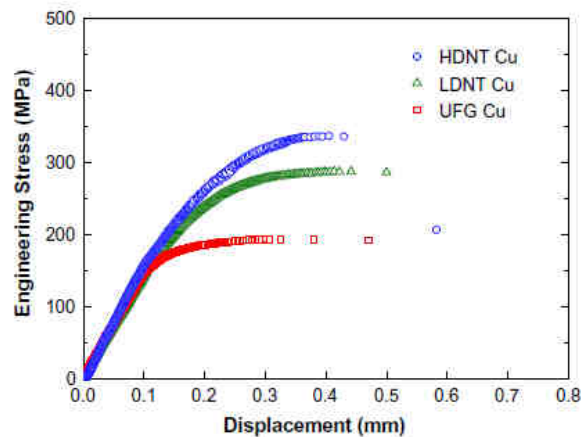


Fig. (11): Engineering stress vs. displacement plot of HDNT (High density nano-twinned), LDNT (Low density nano-twinned) and ultrafine-grained (UFG) specimens which were pre-cracked to 1.5 mm prior to the fracture experiment.

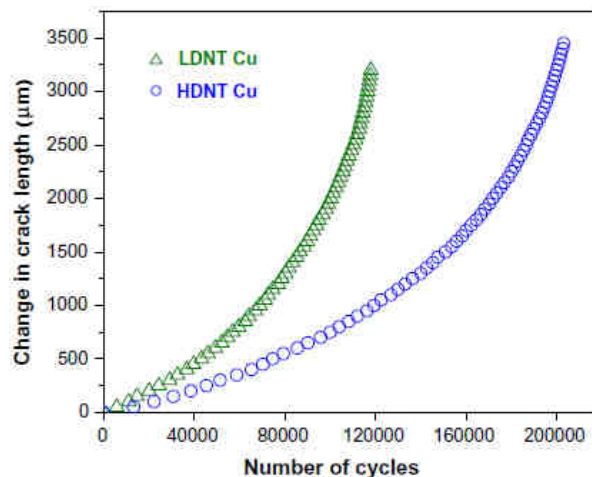


Fig.(12): A plot of the change in crack length vs. the number of cycles with an initial ΔK of $6\text{MPa}\sqrt{m}$ for the HDNT and LDNT specimens, demonstrating that increasing twin density leads to a decreased rate of fatigue crack growth.

However, using micromechanical resonant fatigue characterization structures similar to that shown in Fig. 13, a number of investigations have demonstrated that micron-scale silicon exhibits “metal-like” stress-life ($S=N$) fatigue behavior in ambient air. Our recent studies have indicated that the fatigue degradation process occurs via mechanisms confined to the SiO_2 native oxide layer that forms upon exposure to the atmosphere; specifically, this reaction-layer fatigue process involves the sequential, stress-assisted oxidation and stress-corrosion cracking of the native oxide, as described in Muhlstein et al. (2002b). However, to understand how such fatigue damage accumulates in these $S=N$ fatigue tests, it is necessary to fully characterize the micromechanical fatigue testing structure, and in particular to calculate the driving force for crack advance under resonant loading conditions. During micromechanical fatigue characterization of silicon films, the compliance of the specimen increases as fatigue damage accumulates in the form of incipient crack growth. This degradation induces a corresponding decrease in the natural frequency of the structure[38].

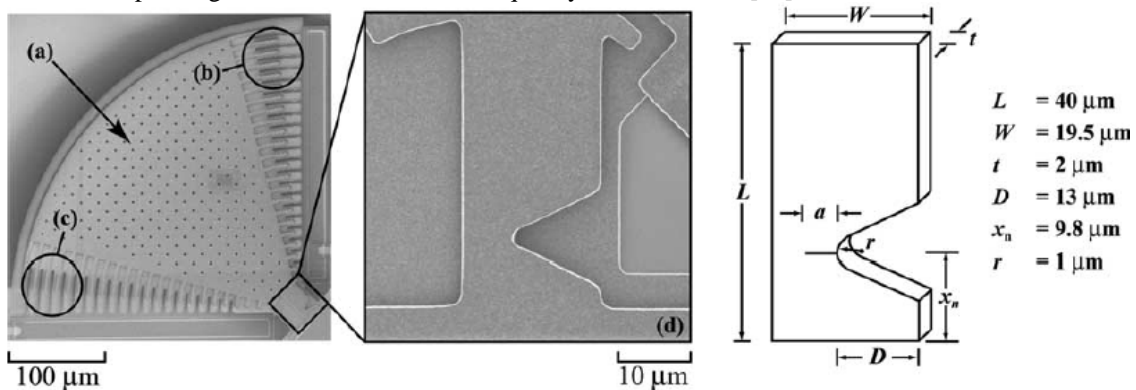


Fig. (13): SEM of the micron-scale fatigue characterization structure containing a notched cantilever-beam specimen. The (a) mass, (b) comb drive actuator, (c) capacitive displacement sensor, and (d) notched cantilever-beam specimen are shown. The nominal dimensions of the specimen are as indicated in the schematic.

Thin Film Fatigue

In metallic thin films this translates to plastic yielding occurring at increased stresses over their bulk counterparts. Although this phenomenon was observed as early as 1959 [39], no consensus or common basic understanding of it yet exists. In addition to plastic behavior, other mechanical properties can exhibit size effects, such as fracture toughness and fatigue resistance. Each of these properties operates on a characteristic length scale that can be compared to the physical dimensions of microelectronics, micro-devices, or nano-devices. Elastic properties are dependent on the bonding nature of the material and only exhibit size effects at the atomic scale. In contrast, plastic, fatigue, and fracture properties all exhibit size effects in the micrometer and sub-

micrometer regime. These properties all depend on defect generation and evolution, which are mechanisms that operate on characteristic length scales [40].

Several recent investigations of fatigue strength of LIGA Ni [41–43], Al foils [44], crack growth behavior of NiP amorphous alloys films [45–46] as well as fatigue damage behaviors of 304 SS thin films have been conducted by using either micro-tension [41–42] or micro-bending [47–48] methods. These studies have shown the potential size effects and the importance for the failure analysis of the MEMS structures. Thus, to deepen the understanding of fatigue damage process and to obtain a wide range of fatigue strength data of various micrometer scale materials would be rather necessary for the MEMS product design. Fig. 14 presents an SEM image of a group of the micro-beams with dimensions of $25\mu\text{m}\times 30\mu\text{m}\times 60\mu\text{m}$ (thickness \times width \times length).



Fig. (14): An SEM image of a group of micrometer-scale cantilever beam-type samples, which were fabricated by FIB, for fatigue testing.

Other MEMS devices such as those made of polysilicon MEMS thin films also have been investigated for their fatigue initiation and short fatigue crack growth behavior [49–51]. Study by Kahn et al. [49] used monotonic electrostatic loading to initiate fracture from an atomically sharp crack in polysilicon film samples induced by nanoindentation, and determined the fracture toughness for the polysilicon MEMS devices. Cho et al. [50] not only experimentally measured the mode I fracture toughness for polysilicon MEMS films, but also investigated the effect of mixed mode loading on fracture toughness. Most recently, Bomidi et al. [51] developed a 3D finite element model to study the fatigue behavior of MEMS devices in general, which featured a novel 3D micrograin debonding procedure enabling the investigation of intergranular crack initiation, propagation and coalescence during fatigue failure. However, none of these studies considered the potential role of the native oxide layers (silicon oxide or nickel oxide) residing on the thin films' surfaces, which seems to play an important role in fatigue crack initiation [52] (see figure 15).

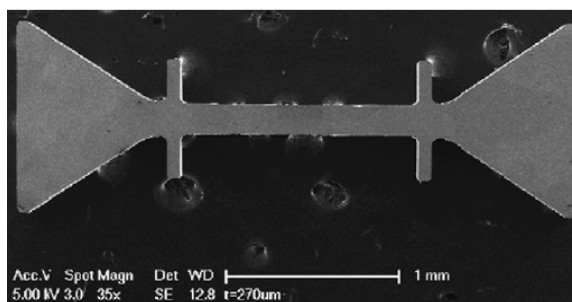


Fig. (15): Scanning electron microscopy (SEM) image of LIGA Ni MEMS thin films (top view, 270 nm thick).

Fig. 16(a) compares stiffness curves measured dynamically for films with a thickness of 0.2, 0.4, 0.6, 0.8, 1.0, and 1.5 μm fatigued with stress amplitudes σ_a that were approximately the same in all tests. In the following, stated mean stresses and stress amplitudes correspond to the largest stresses occurring in the film, i.e. the stress component in

the direction of the beam axis at the surface of the Ag film close to the beam support as determined by FEA. Note that the initial stiffness of the beams depends on the thickness of the Ag film as well as on the lever length that was not constant in these tests. The mean stress σ_m , and a number of 3.8×10^6 force cycles was applied. The stiffness decrease suggests the onset of damage formation; the number N_d of cycles to stiffness decrease was defined according to the following procedure: Constant slope regions of the stiffness curves were fitted by straight lines, the intersection of which then defined N_d . This is illustrated in Fig. 3.5(b) for the case of the 1.5 μm thick film. Micro-scopical damage analyses confirmed that this decrease in stiffness is associated with the formation of fatigue damage such as extrusions, cracks, and voids [53].

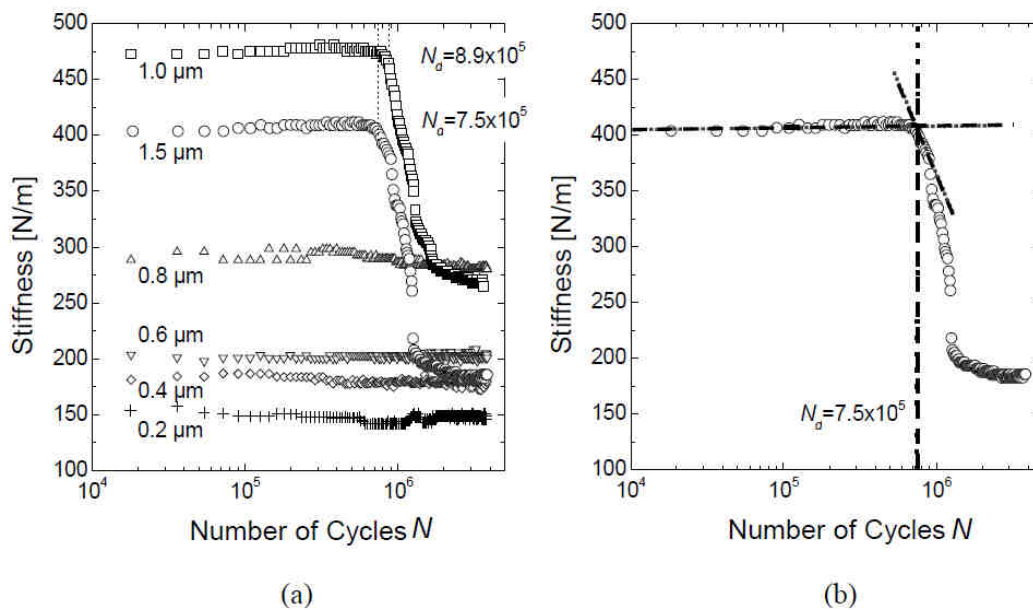


Fig. 16: (a) Stiffness at a stress amplitude $\sigma_a \sim 54$ MPa and a mean stress σ_m . Bilayer beams with 1.5, 1.0, 0.8, 0.6, 0.4, and 0.2 μm thick films were tested; the lever lengths were 46, 44.5, 42.5, 43.3, 41, and 40 μm , respectively. (b) Determination of the number N_d of cycles to stiffness decrease.

Polymers and Fatigue Testing

Experimental fatigue investigation of engineering plastics or polymers is a newer subject of researches compared to metals. Many of the established test methods for metals were adopted in plastics fatigue testing. They are to be distinguished from that of other categories of materials, especially metals, due to the differences in microstructure, proximity of test temperature and melting point, temperature increases associated with testing, craze development, and large differences between tensile and compressive strengths. Several factors such as temperature susceptibility even at room temperature, large inelastic deformation and cyclic softening are among factors to be considered for plastics fatigue testing [54]. Two main mechanisms have been observed in fatigue failure of many plastics, depending on force (stress) level and frequency; thermal fatigue failure that involves thermal softening (yielding) and mechanical fatigue failure that is due to the conventional fatigue crack initiation and propagation under cyclic loads [55]. The experimental fatigue study of plastics should consist of a multi-step procedure; first, a consistent laboratory condition should be maintained for all of the tests that a comparison is targeted. Second, a proper test control method should be selected, i.e. force, displacement or strain. Third, the effects of significant parameters that influence fatigue behavior of plastics should be assessed. These may include (but not limited to) mean stress, stress concentration, waveform, frequency, environment, and thermal effects due to cycling. If a part or component test is aimed, a set of material tests are to be completed by an analysis and life prediction, as well as a (limited) number of full scale part or component test. These full-scale tests, though in many cases complicated and costly, provide significant confidence to the engineer with regard to the combined material testing and analysis he already performed.

Finite Element Based Fatigue Life Analysis

Fatigue analysis has traditionally been performed at a later stage of the design cycle. This is due to the fact that the loading information could only be derived from the direct measurement, which requires a prototype [56]. Multi body dynamics is capable of predicting the component loads which enable designer to undertake a fatigue assessment even before the prototype is fabricated [57]. The purpose of analyzing a structure early in the design cycle is to reduce the development time and cost. This is achieved to determine the critical region of the structure and improving the design before prototype are built and tested. The finite element (FE) based on fatigue analysis can be considered as a complete engineering analysis for the component. The fatigue life can be estimated for every element in the finite element model, and the contour plots of life damage can be obtained. The geometry information is provided by the E result for each load case applied independently. Appropriate material properties are also provided for the desired fatigue analysis method. An integrated approach to fatigue life analysis combines the multi body dynamic analysis, finite element analysis, and the fatigue analysis into a consistent entity for the prediction of the fatigue life of a component [57]. The flowchart of the finite element based fatigue analysis is shown in Figure 17.

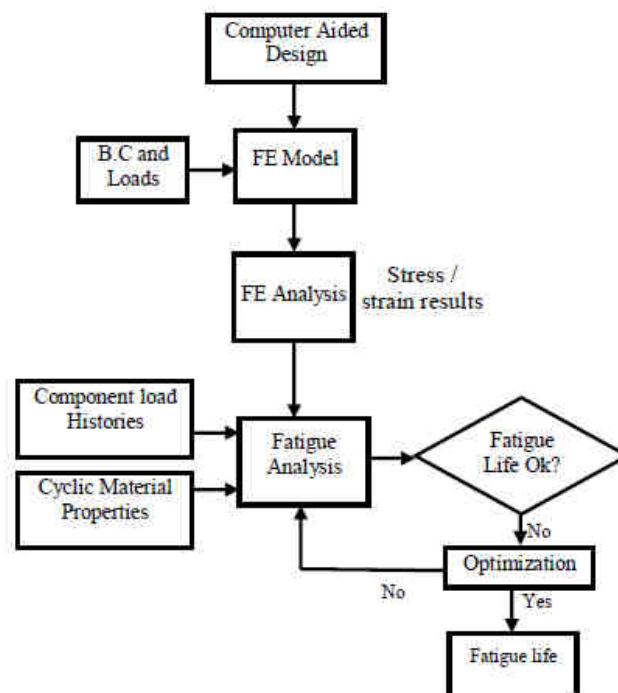


Fig. (17): The finite element based fatigue analysis.

Conclusion:

From the present review, the most important testing techniques were briefly described. So, the main findings mentioned are summarized as:

1. Many solutions have been presented over time, but still today new methodologies are required and emerging to overcome limits of the existing ones.
2. New testing methodologies are also needed to support the continuous advance of micro and nano-electronics, as well as biology, for a deeper understanding of the behavior of materials like nano-fibers, nanotubes, nanostructured, and biological samples.
3. The increase in yield strength with decreasing length scales including both microstructural and geometric dimensions.
4. Fatigue properties of small-scale materials and reveal size effects of the fatigue behavior of small-scale materials can obtain by fatigue testing techniques.
5. Convincible experimental evidence shows that when either the film thickness or the grain size is decreased below roughly $1\ \mu\text{m}$, the typical dislocation wall and cell structures found in fatigued coarse-grained bulk materials no longer develop and are replaced by individual dislocations for thin metal films confined by a substrate.
6. The transition from damage of bulk to damage localized at interfaces is attributed to constraints on dislocation activity at submicron length scales, where the typical surface damage of fatigued bulk metals, such as extrusions and cracks near extrusions, is gradually suppressed and replaced by damage that is localized at interfaces, such as cracks, grooves, and voids along grain and twin boundaries.

Despite the great work carried out for developing more accurate, reliable and simple methodologies, at present, there are still neither standard methods nor universally recognized fixtures for mechanical testing of materials at micro and nano-scales.

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