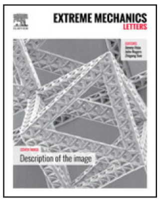
Extreme Mechanics Letters 18 (2018) 79–85

Contents lists available at ScienceDirect

Extreme Mechanics Letters

journal homepage: www.elsevier.com/locate/eml



Digital micromirror device (DMD)-based high-cycle tensile fatigue testing of 1D nanomaterials

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ARTICLE INFO

Article history:
Received 9 August 2017
Received in revised form 20 November 2017
Accepted 29 November 2017
Available online 6 December 2017

Keywords:
Microelectromechanical systems (MEMS)
Nanomechanics
Nanowire
High cycle fatigue testing
Tensile testing
Digital micromirror device (DMD)

ABSTRACT

Fatigue behavior of nanomaterials could be critical for numerous nanomechanical applications involving dynamic deformation processes, such as in flexible electronics devices. Despite that substantial research efforts have been made on mechanical characterization of various one-dimensional (1-D) nanomaterials under quasi-static loading, very few works have been done so far on the challenging fatigue testing of individual 1-D nanostructures, in particular for their high-cycle fatigue behavior. Here, instead of designing a new device, commercially available digital micromirror device (DMD) has been adopted to develop a cost-effective platform for investigating the high-cycle fatigue responses of individual nanowires/nanotubes, due to its ultra-high actuation frequency (up to 32,000 Hz), under cyclic tensile straining. We further demonstrated that, due to the small footprint of this MEMS-type device and its remote controlling mechanism, desired *in situ* tensile fatigue testing of individual nanowire can be achieved inside a scanning electron microscope (SEM). In addition, the millions of independent movable micromirrors on a single DMD chip make our platform particularly suitable for high-throughput testing of various 1-D nanomaterial samples for the statistical analysis of their fatigue characteristics.

1. Introduction

1D nanomaterials, such as metallic, semiconductor, polymer nanowires (NWs) and carbon nanotubes (CNTs), with diameters ranging from tens to hundreds of nanometers, have recently stimulated great interest due to their importance as building blocks for nanoscale electronics and electro-mechanical systems in numerous emerging engineering applications, such as flexible/wearable electronics [1–3], bio-integrated electronics [4–6], sensors/detectors [7–9]. Such as, a random network of silver (Ag) nanowires could provide a conductive path for electrical circuits in flexible display, polymer/MnO_2-coaxial nanowires could make the energy storage sources become more deformable [10]. However, the ability to achieve the full potential of aforementioned new technologies in these fascinating applications is ultimately limited by how these one-dimensional building blocks will behave at relevant length scales, in particular, their mechanical performance and reliability. For example, in order to maintain stable electrical characteristics under the repetitive and extreme bending strain conditions for display and flexible battery, the mechanical properties of Ag nanowire or the coaxial metal/polymer nanowire should remain stable during long-time services respectively [11]. However, fatigue, the progressive and localized structural damage that occurs when a material is subjected to cyclic loading, is a commonly encountered mode of failure in numerous mechanical and electro/thermo-mechanical devices and mechatronic systems, which ultimately limits their stability and lifetime [12,13]. Whether fatigue also exists at the nanoscale and whether one can properly characterize the corresponding fatigue behavior of individual nanomaterial structures at the relevant length scales become an interesting and important problem.

Driven by the purposes of dynamical mechanical characterization of individual 1-D nanostructures, several methods and testing devices have been developed in the past two decades to study the cyclic/fatigue behavior of nanowires/nanotubes, such as atomic force microscopy (AFM)-based mechanical testing platforms [14,15]. Recent years, microelectromechanical system (MEMS)-based testing platforms [16–19] have emerged as powerful and versatile tools to study low-dimensional micro- and nanostructures. However, typical MEMS-based testing devices have some limitations in terms of stable load and displacement outputs due to its complicated electro-mechanical coupling actuation mechanisms, not suitable for long-time cyclic testing and fatigue behavior characterization. Therefore, pure micromechanical devices have been developed for both *in situ* SEM and TEM

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https://doi.org/10.1016/j.eml.2017.11.005
2352-4316/

mechanical testing, including those actuated by external nanoindenter based on push-to-pull mechanism [20–24], allowing us to characterize individual 1-D nanostructures with relatively low loading frequency but robust load outputs, thus useful for low-cycle fatigue behavior study [25]. However, the intriguing high-cycle fatigue behavior and the associated cyclic tensile loading of individual nanowires remains extremely difficult to perform but desperately needed, largely due to the challenges of applying well controlled high cyclic loading on such exceedingly small samples as well as to reduce the total testing time for high cycle fatigue responses (as well as avoid other issues caused upon prolonged *in situ* SEM/TEM testing, such as sample contamination and carbon deposition inside electron microscope chambers). Some pioneers have tried the resonance cyclic loading method to investigate ZnO nanowires in TEM [26,27], but those tests were less controllable and hard to be quantified. Recently in our lab, instead of designing and fabricating new MEMS-type device dedicated for high cyclic tensile testing, we proposed to adopt a commercially available MEMS product “Digital Micromirror Device” (DMD, invented by Texas Instruments^{TM}, TX, USA), for the high frequency loading of individual 1-D nanostructures. We previously demonstrated its application for monotonic and cyclic torsional loading of an individual nanowires as a micromachine [28]. In this work, we further extended this DMD-based platform to tackle the challenge of high-cycle tensile fatigue testing of individual nanowires/nanotubes.

2. Methodology and platform development

The key component of our platform is the commercially available DMD chip [29], which was invented by Dr. Larry Hornbeck in 1987. Originally, it was developed as an optical MEMS device widely used in the fields of DLP (digital light processing) projector and television. The DMD chip is essentially a digitally controlled micro opto-electro-mechanical system (MOEMS) spatial light modulator (SLM). Depending on the model and resolution, there are about 1–2 million independent aluminum micromirrors inside each DMD chip. Based on the working principle of the DMD, the tilting movement of individual micromirror could be naturally used to apply mechanical loading on the nanowire attached on mirrors with controllable high loading frequency. The flipping rates of the micromirror could be up to 32,000 Hz, well enough for high cycle fatigue testing. The high frequency of the DMD could also greatly reduce the electron deposition effect under SEM, which was a common issue of long-time *in situ* SEM experiments. Despite no dedicated force sensor, the load and strain distribution on the tested nanowire can be obtained from separated monotonic tensile tests [20,21] and/or estimated and verified by finite element method (FEM), as detailed below.

2.1. The concept of DMD (digital micromirror device)-based cyclic tensile loading

In the present work, 0.7-inch 1024 × 768 Extended Graphics Array (XGA) TI DMD chip module was used for our research. After removing the cover protective glass lid on the DMD chip as shown in Fig. 1(A), we can observe the chip surface under microscopes: arranged in a rectangular array, almost one million microscopic aluminum mirrors (13.5 μm × 13.5 μm) were contained in the DMD and each of them corresponded to the pixel of the image to be displayed. The Fig. 1(B) shows that the micromirrors can be individually removed without influencing their neighboring mirrors. The actuation of the tilting movement of the micromirror was induced by the electrostatic force between the micromirror and the electrode structure under it as shown in the inset of Fig. 1(B) which was caused by the voltage difference. Then, we use a Micromanipulator^{TM} (Nevada, USA) probe station with a sharp tungsten STM (scanning tunneling microscope) probe to pick up individual nanowire samples and place them onto the designated location at DMD. For nanowire clamping, we applied conductive silver epoxy glue (Chemtronics^{TM} CircuitWork$^{®}$ Conductive Epoxy) to fix the both nanowire ends onto the micro mirror corners. Alternatively one can also use FIB or EBID for the clamping. To ensure the solid bonding between the nanowire and DMD stages, multilayer coating of the silver glue was applied [24] to protect the nanowire during fatigue testing.

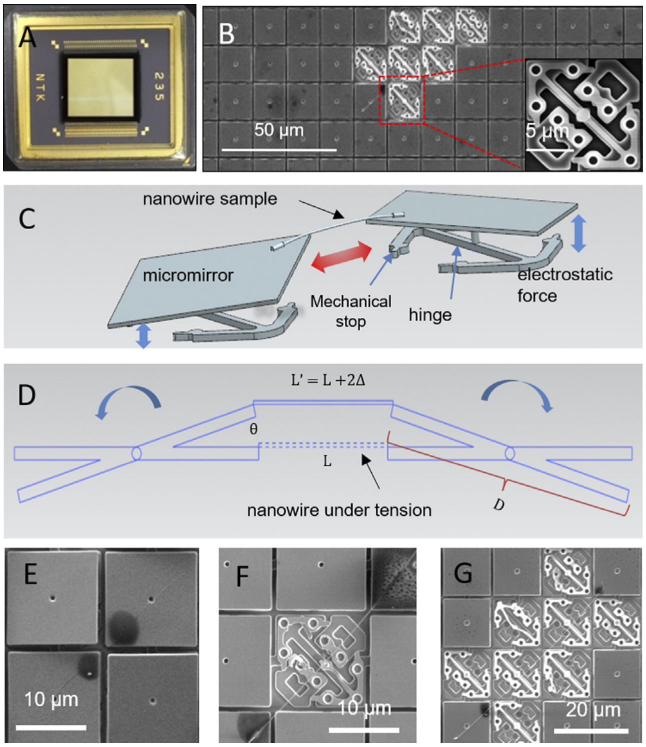

Fig. 1. Basic concept for nanowire tensile fatigue testing based on DMD: (A) shows a DMD (digital micromirror device) chip after removing the protection glass cover. (B) Micromirror arrays on the DMD chip with some of them removed. The inset image shows the micro-fabricated structure under a single micromirror, whose main components were torsion hinge and electrodes. (C) is the schematic image shows how to exert tensile force on nanowire specimen by the micromirrors movement due to electrostatic force underneath from different view. (D) shows how the tensile strain was calculated during experiment. (E), (F), (G) shows that the strain of the nanowires can be changed by adjusting the effective gauge length.

Fig. 1(C) shows the concept of our methodology in detail. The compliant torsion hinge, whose axle was fixed at both ends, was responsible for the support of the micromirror by a short cylinder. The spring tips were used as mechanical stop for the micro mirror, therefore the rotation angle was limited to +12° and −12° [30] (or +17° and −17° in other DMD devices). The two electrodes under the micromirror were used to control the “on” or “off” position of the mirror by electrostatic attraction, therefore reflecting the light to project the black and white pixel. According to TI’s official specifications, the electrode switching rates could be up to 32,000 Hz which means the micromirror can also be tilted at such frequency. What’ more, with the commercial DLP development kit, the movement of individual micromirror was remotely controllable through customer input program. Due to these advantages of DMD [31], we discovered that the tilting movement of DMD micromirrors could be indeed used in applying high cyclic mechanical loading on the individual 1D nanostructures that are attached on the activated micromirrors [28].

Here, for tensile loading on the nanowire attached on the micromirrors, the tensile strain could be determined according to