

## METHOD AND APPARATUS FOR ADJUSTABLY INDUCING BIAXIAL STRAIN

### CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority from U.S. provisional application Ser. No. 60/467,163 filed on Apr. 30, 2003, incorporated herein by reference in its entirety.

### STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

[0002] This invention was made with Government support under Grant No. DE-AC03-76SF00098, awarded by the Department of Energy. The Government has certain rights in this invention.

### INCORPORATION-BY-REFERENCE OF MATERIAL SUBMITTED ON A COMPACT DISC

[0003] Not Applicable

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### BACKGROUND OF THE INVENTION

[0005] 1. Field of the Invention

[0006] This invention pertains generally to fixtures for inducing strain on a specimen in a transmission electron microscope, and more particularly to an apparatus and methods for inducing biaxial and radial strain on a thin film as a function of change in temperature.

[0007] 2. Description of Related Art

[0008] Measurement of mechanical properties and the identification of deformation mechanisms from direct micro-structural observations of thin films has been done using various experimental techniques. Such techniques present challenging problems because bulk methods such as uniaxial tension testing, are very difficult to apply directly to films. Difficulties include generating forces and strains required in a small space, gripping the film, and prevention of bending force components in the film. While micro-electromechanical uniaxial strain fixtures have been fabricated, the approach does not generate the state of strains and stresses seen in chemical and biological micro-sensors, micro-actuators, passivation layers, micro-electronics, data storage and other film-based devices. The stress states that develop in these devices, whether intrinsic or extrinsic, are typically biaxial, plane-stress in nature. Bulge testing has been used to evaluate the biaxial modulus of thin films, but this method requires elaborate hardware to induce a state of

stress that is generally biaxial but still varies across the bulged sample. In addition, it is infeasible to install the complex bulge test apparatus inside the five cubic millimeter volume and vacuum environment of a Transmission Electron Microscope (TEM) objective lens.

[0009] Inducing radial strain through direct thermal expansion for observation of a thin film by a TEM is infeasible. For example a stainless steel ring would need to be heated to a temperature of nearly 3,000 deg. C to achieve a desired expansion, which is a temperature in excess of the melting point of stainless steel.

[0010] What is needed is an apparatus that applies uniform, variable biaxial strain on a thin film and is configured to fit within the dimensional restraints and environment of a TEM. An apparatus that applies and releases biaxial strain to thin films in cycles to simulate fatigue for failure analysis is further desirable. An apparatus that allows observation of strain on a freestanding film without a substrate support is also desirable.

### BRIEF SUMMARY OF THE INVENTION

[0011] The present invention generally comprises an apparatus and method for making and using a sample holder that will induce biaxial strain on a thin film in response to temperature changes. In one embodiment, the sample holder, or fixture, is made from a Shape Memory Alloy (SMA) such as Nickel Titanium (NiTi) alloy. It will be appreciated that one property of an SMA is that it will return to a previously memorized configuration through a phase transformation in response to a change in temperature.

[0012] An apparatus, such as a die and a press, is used to reversibly deform a tube of SMA of a first shape to a second shape. A sample holder made from the tube of SMA will retain the second deformed shape until a change of temperature is applied, then the sample holder will return to the previous memorized shape. The range of temperature change necessary to cause a phase transformation depends on the composition of the SMA. This change in shape imparts a biaxial strain on a thin film attached to the sample holder.

[0013] In one mode of use, the sample holder is a ring and the change in shape changes the inner perimeter of the ring. In the example of a circular ring, a change in the inner perimeter is also a corresponding change in the inner diameter. In another mode of use, the sample holder is sized to use within the environment of a TEM and, in an exemplary embodiment, can be configured to impart reversible and repeated cycles of biaxial strain in response to temperature changes to the sample holder.

[0014] Accordingly, the invention will enable dynamic microstructural characterization of a thin film sample as a function of biaxial strain. Applications for microstructural study include biological microsensors, microactuators, passivation layers, micro-electronics, data storage and other film based devices. In one embodiment, a ring assembly can be expanded and contracted repeatedly. The invention can also be configured as a thermally activated prime mover for a reversible radial actuator, a reversible radial brake, a reversible coupling, or for tuning resonant RF cavities. One application for a reversible radial actuator is in a micro electromechanical system (MEMS).