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Research paper

A micro test platform for in-situ mechanical and electrical characterization of nanostructured multiferroic materials

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ABSTRACT

This paper presents a novel approach to simultaneously fabricate a Si micro test platform and a nanostructured test specimen of a multiferroic material with critical dimension of 200 nm and lateral aspect ratio of 33 for in-situ characterization in a scanning electron microscope (SEM). The design is adjusted to allow for electrical resistance measurements and tensile tests. We report on a fabrication process comprising e-beam lithography and dry etching technology and, as a proof of concept, on the measurement of the force-displacement characteristics of Ni nanobeams.

1. Introduction

Multiferroic materials exhibit unique properties including ferromagnetic and ferroelastic ordering as well as ferroic phase transformations with large changes in lattice parameters, which is of special interest for actuation at the nanoscale. Typical examples are thermal and ferromagnetic shape memory alloys (SMAs) that exhibit a martensitic phase transformation which can be either induced by temperature or by a magnetic field, respectively [1]. The energy density of SMAs can be as high as 10 MJ/m^3, allowing for distinct actuation properties [1]. Downscaling of SMA actuators to the nanoscale has the potential to overcome size limitations of conventional actuators such as electromagnetic motors [2]. Using the multifunctional properties of SMAs enables a new way of exploiting material properties, as “the material is the machine” [3]. Materials of interest include ferroelastic alloys such as Ni-Ti and the ferromagnetic alloy Ni-Mn-Ga. Recently, beam cantilever devices have been fabricated from epitaxial Ni-Mn-Ga films having beam width and thickness of 100 nm and 125 nm, respectively, showing thermoelastic behavior [4]. First nanostructured FSMA actuators have been developed for optical switching of nanophotonic waveguides [5].

In order to understand fundamental size effects as well as to utilize these effects for nanoactuation and sensing, in-situ characterization of mechanical and electrical properties is needed. A number of methods for in-situ nano-mechanical testing have been developed, e.g. for transmission electron microscopy or for specimens that are fabricated separately from the measurement device [6,7]. In this investigation, a test platform is developed for use in a SEM to allow for simultaneous characterization of stress-strain and electrical resistance measurements. The technological challenges will be discussed and a viable process flow will be shown for the case of a Ni nanobeam on a Si test platform.

2. Design

The layout consists of various functional units for operation at different length scales as sketched in Fig. 1a. On the micro scale, a microelectro-mechanical system (MEMS) platform is designed comprising a fully suspended frame, folded springs for guiding and resetting as well as anchoring structures. Openings are provided to allow for mechanical displacement by a nanomanipulator. On the nanometer scale, a fully suspended Ni nano-bridge is designed with critical dimensions of 200 nm and lateral aspect ratios up to 33 for quantitative tensile analysis as well as for electrical resistance measurements. In order to avoid alignment issues, the layout is designed such that all functional units can be fabricated in a common lithography step. The fully suspended Ni nano-bridge is connected on one side to the movable frame. On the other side, it is connected to a fixed anchoring structure. Upon deflection of the movable frame by a nanomanipulator, the nano-bridge is subject to tensile force. The information on deflection from real-time

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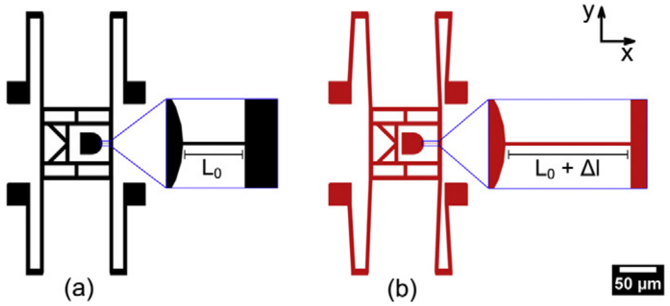

Fig. 1. Schematic of the micro test platform and nanostructured test specimen. (a) in initial state and (b) in deflected state. The strain induced in the nano-bridge is highlighted.

SEM images and force are used to determine the stress-strain characteristics of the material.

3. Fabrication technology

Simultaneous micro-nano fabrication of this device is attractive, as ferroic SMA materials can be deposited by physical vapor deposition (PVD) methods and alignment issues of sample transfer are avoided. However, various challenges in fabrication arise due to the high aspect ratios involved and the high deposition temperature during DC magnetron sputtering of about 500 °C. High deposition temperatures are required to obtain functional crystalline films [5], rendering polymer-based lift-off processes impossible. Furthermore, ferroic SMAs belong to the difficult-to-etch materials that do not allow for nanomachining by reactive ion etching (RIE). Recently, two different approaches for nanomachining the SMA film have been reported [5]. Accordingly, one way to achieve nanosized structures is the use of ion beam etching (IBE) after deposition of the SMA film. However, this method leads to redeposition and thereby to hard-to-remove sidewalls at the border of the structures. The second approach is based on nanomachining the Si prior to the deposition of the SMA film. This way, common Si-based technology can be used. Therefore, we pursue a process flow based on micro- and nanomachining of a silicon on insulator (SOI) substrate prior to deposition of the SMA film onto the prestructured device as illustrated in Fig. 2.

The starting SOI substrate consists of a 12 μm thick device layer and a 1 μm thick buried oxide layer as well as a 1.4 μm thick surface oxide layer (Fig. 2a). The fabrication process comprises electron beam lithography (EBL), PVD of Cr and lift-off (Fig. 2b), anisotropic CHF_3 etching of the surface oxide layer (Fig. 2c) and cryogenic silicon SF_6 etching of the device layer (Fig. 2d). Nano-sized silicon bridges are formed at thin mask structures. The buried oxide layer as well as the surface oxide layer with residual Cr are removed by wet-etching with hydrofluoric acid, leading to a fully suspended test platform (Fig. 2e). Sticking effects are avoided by replacing the hydrofluoric acid with isopropyl alcohol and ensuing critical point drying. Subsequently, a 200 nm thick SMA-film is deposited (Fig. 2f). In order to obtain a freestanding bridge of the deposited material, the silicon supporting structure underneath the bridge is removed by means of a short isotropic dry etching step using SF_6 (Fig. 3b). The influence of this process step to the Si-frame is negligible due to the size ratio of the Si-frame with respect to the bridge. However, after this process some residual material remains underneath the Ni test specimen. This could be due to sidewall deposition of the

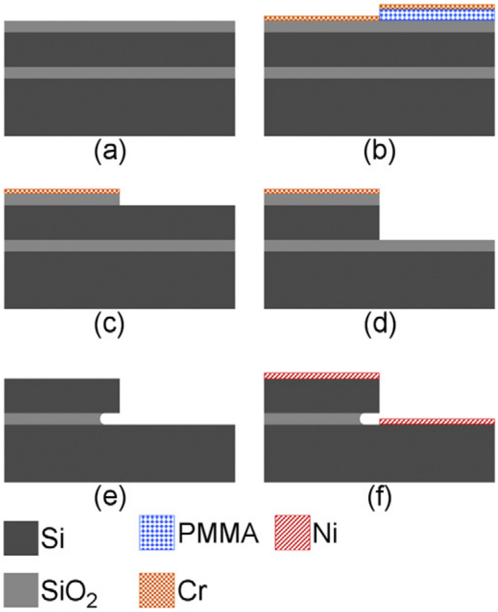

Fig. 2. Schematic illustration of the process flow for the simultaneous fabrication of the Si micro test platform and nanostructured test specimen. a) SOI wafer with surface oxide b) EBL, Cr-PVD and lift-off c) Anisotropic etching of surface oxide layer d) Dry etching of Si device layer e) Wet etching of sacrificial layer f) Deposition of ferroic Ni layer

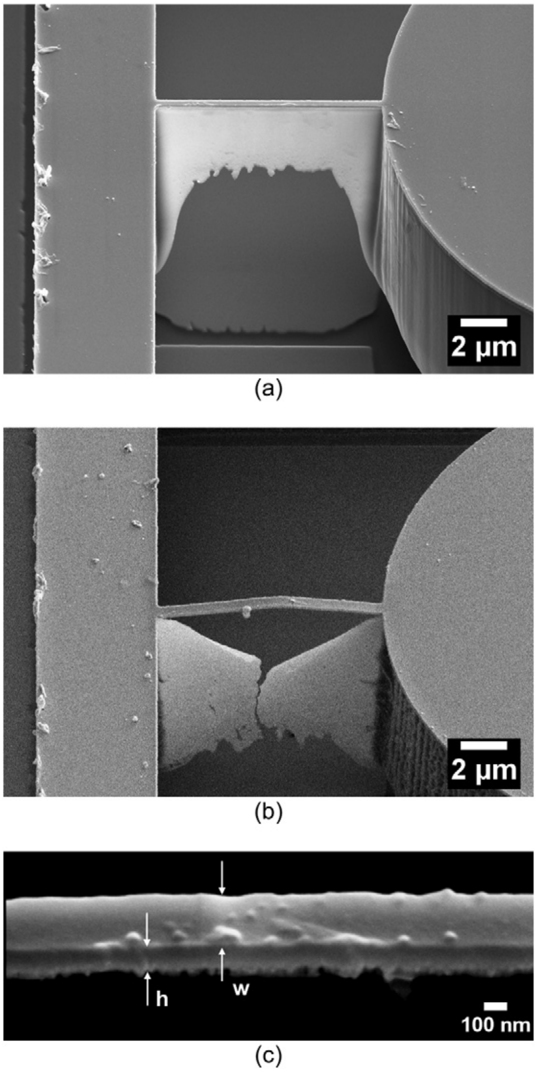

Fig. 3. a) SEM image of the silicon structure below the nanostructured test specimen after process step (f) (Fig. 2). b) After isotropic Si etching, compressive loading shows breaking of residual material. c) Close-up of Ni test specimen. The ratio of length l: width w: height h is 9600:290:110, corresponding to a lateral aspect ratio of 33.