High-Throughput Determination of Mechanical Properties using Microcantilever Arrays

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Abstract:
We are developing a platform for measuring the composition-dependent mechanical properties of materials using an array of microcantilevers. The goal is to be able to investigate the properties of nearly all compositions in a ternary phase diagram with 1 atomic percent (at.%) resolution in a single experiment, and to use this capability to determine trends and identify unusual and/or useful materials in a wide range of ternary systems [1]. Our approach is to prepare a large array of prefabricated cantilevers on a single wafer, deposit a composition-spread thin film on the array, and interrogate each cantilever as a function of some probe stimulus (temperature change, magnetic field, etc.) and thereby infer the composition-property relationship. Our initial experiments are aimed at measuring magnetostrictive materials, for example in the Fe-Ni-Al ternary system. The composition-spread films are deposited using three spatially-separated sputter guns, yielding a typical composition gradient on the order of 1 at.% per mm. In order to determine the properties with ~ 1 at.% resolution, we therefore require microcantilevers located on ~ 1 mm centers. This approach eliminates the run-to-run variations that can confound one-off experiments. With an automated measurement system that is capable of characterizing in one day the response of a 3,000-cantilever array representing an entire ternary phase diagram, this technique enables investigation of magnetostriction in thin films with unprecedented composition resolution and speed. Our “combinatorial” platform will also enable studies of other mechanical properties of advanced materials such as conventional and ferromagnetic shape-memory alloys.

Summary:
All of our device preparation is done at the Cornell NanoScale Science & Technology Facility (CNF). We choose Si₃N₄ as cantilever material because it is straightforward to deposit and requires only a single lithographic step. We use the nitride tube furnace to deposit ~ 1 µm of low-stress LPCVD Si₃N₄ on a <100> silicon wafer. Patterns are transferred by standard photolithography using the GCA 5X stepper. After exposure, MIF 300 base developer is used to develop the patterns and PlasmaTherm72 reactive-ion etcher is used to etch the nitride. After stripping photo resist, KOH is used to wet etch the Si to undercut cantilevers—the Si <111> surface acts as etch stop. A CO₂ critical-point dryer is used to dry and release the cantilevers to avoid sticking. With our current design, cantilevers are designed to have dimensions of 500 µm x 50 µm and are spaced 1 mm apart in a square array. These dimensions ensure that each cantilever is small enough that the composition of the film on the cantilever is essentially constant, and that composition-dependent properties can be interrogated with a resolution of ~ 1 at.%. The composition-spread thin films are deposited in a custom-designed sputtering system in our lab.

At the center of our measurement design, the cantilever acts as a base substrate for a deposited thin film whose properties (for example, intrinsic stress, shape memory, or magnetostriction) are to be measured [2]. For magnetostriction measurements, the curvature of the deposited bilayer film-substrate cantilevers are measured as a function-applied magnetic field. The cantilever base has essentially no magnetic response, so the Stoney formula for bending of a cantilever under differential stress can be adapted to the present magnetostriction measurements to give:

\[ \lambda = D \frac{E_s I_s^2}{E_f I_f} \left( \frac{l + v_f}{l - v_s} \right) \]

where \( \lambda \) is the magnetostrictive response, \( D \) is the vertical deflection at the end of the cantilever, \( E_s \) is Young’s modulus of the cantilever, \( I_s \) is the thickness of the substrate, \( I_f \) is the thickness of the film, \( l \) is the length of a cantilever and \( v_s \) and \( v_f \) are the values for Poisson’s ratio for the substrate and film [3], respectively. The deflection of the cantilever depends only on the differential strain between the film and cantilever base material. Thus this approach can be used for measurement of any source of differential strain, such as differential thermal expansion and, importantly, shape memory materials.

References:
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Figure 1, top left: Fabrication process flow of a Si₃N₄ cantilever.

Figure 2, middle left: Cantilever bending measurement setup. Changes in cantilever curvature due to magnetostriction under an applied magnetic field will modify the optical path of the laser, which is detected by a position sensitive detector, analyzed by software, and reported in terms of the differential strain.

Figure 3, bottom left: (Left) Intrinsic stress in thin films of Ni-Fe-Al, as inferred from cantilever deflections as a function in position in a composition-spread sample. Labels indicate the approximate position of the elemental sources creating the composition spread. (Right) Photograph of the sample.