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IRON-GALLIUM NANOWIRES FOR ACOUSTIC AND SEISMIC SENSOR APPLICATIONS

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ABSTRACT

This project details the experimental and analytical characterization of iron-gallium (Fe_{100-x} Ga_x) alloys in bending for use in sensor applications down to the nanometer scale. By incorporating significant magnetostriction with strong mechanical properties, this material is a strong candidate for use as a magneto-mechanical sensor to static and dynamic bending stresses. A series of experiments were conducted on the magnetic response of millimeter scale cantilevered beams to applied bending loads, with mechanical excitation at the beam tip and the magnetic response measured with a giant magnetoresistive (GMR) sensor and a pickup coil. Results show that this operating regime produces a magnetic induction change of 0.3 T at twice the frequency of excitation. These results were verified by an analytical model based on a free energy expression. Similar testing was performed on a hot-rolled iron-gallium sheet to determine if the material retained useful magnetostrictive properties after processing. With the pronounced sensing response characterized at the millimeter scale, efforts are underway to identify these same properties on electro-chemically deposited iron-gallium nanowires with diameters on the order of 100 nm and lengths of several microns. Arrays of cantilevered wires are visualized with an atomic force microscope (AFM) and quasi-static loads are applied with the AFM tip to experimentally determine their mechanical bending stiffness and self adhesion. These tests reveal purely elastic behavior over large deformations but also the presence of significant surface contamination. Magnetic force microscopy (MFM) scans appear to indicate a structure with multiple magnetic domains along the lengths of some wires.

Keywords: iron-gallium, galfenol, nanowire, magnetostriction, bending, sensor

INTRODUCTION

Iron-gallium, or Galfenol, alloys (Fe_{100-x} Ga_x $10 \leq x \leq 30$ at. %) have been shown in numerous studies to be a potential material for use in smart sensing and adaptive structure applications. The promising combination of moderate magnetostriction (up to 400 ppm strain, over 30 T/GPa sensing) and strong mechanical properties such as ductility, high tensile strengths, and machinability makes this material ideally suited for new devices that are not limited to uniaxial compressive operation as are conventional magnetostrictives. Research efforts have included quasi-static material characterization (Kellogg, 2003; Clark et al., 2000), stress annealing for tensile operation (Wun-Fogle et al., 2005), and dynamic actuator testing (Slaughter et al., 2005). Iron-gallium alloys have also been used in deformation processes such as hot rolling (Na and Flatau, 2005) to create thin films for both macro and MEMS sensors.

The focus of this research is to experimentally and analytically characterize the magnetomechanical sensing performance of the material in response to the antisymmetric bending stresses inherent in a vibrating beam. The primary goal is to utilize iron-gallium nanowires to mimic the cilia transduction

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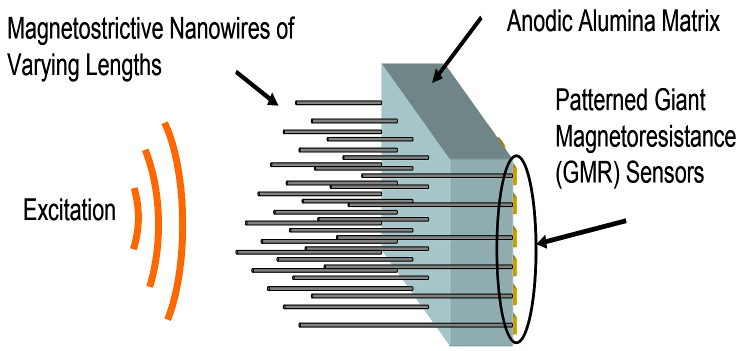
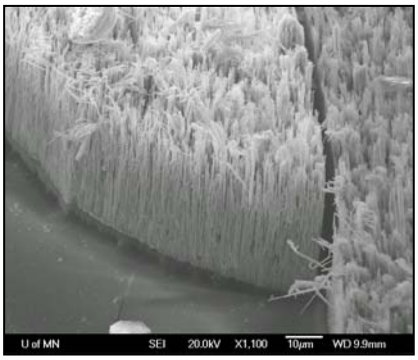
used in biological systems for acoustic, vibration, flow, and tactile sensing. Recent studies (Tucker et al., 2006) of similar technologies have shown that this detection mechanism has promise in a number of applications. In this work, the proposed sensor design calls for an array of cantilevered Galfenol nanowires that bend in response to acoustic, ultrasonic, or seismic excitation. These deflections will induce changes in the magnetization of each wire that can be measured remotely with conventional magnetic devices such as giant magnetoresistive (GMR) sensors near the base of the array. A schematic of this setup is shown in Figure 1(a). Due to the very high spatial density of the arrayed nanowires and the ability to tailor their mechanical response, this sensor should exhibit excellent sensitivity and a wide frequency bandwidth. There are a number of potential advantages to using artificial cilia for the detection of seismic activity specifically, which include shock and fatigue resistance, a reliable and well averaged transfer function, excellent output at low frequencies, and environmental insensitivity.

The nanowire array is fabricated by our collaborators at the University of Minnesota, and consists of an anodic alumina oxide matrix in which the wires are self-assembled in the pores by electrochemical deposition (McGary et al., 2006). The alumina oxide is then etched away to leave cantilevered wires protruding from the base electrode, and the entire structure is bonded to a silicon substrate for handling. Figure 1(b) is an SEM image of an example batch of nickel wires produced by the same technique. Work is proceeding to mount these arrays onto commercially available GMR sensors where a proper magnetic circuit allows measurement of an entire nanowire array and improves the signal to noise ratio and target recognition of the sensor. This device can then be packaged for underwater or structural operation.



(a)



(b)

Figure 1. Schematic of nanowire sensor design (a), where excitation can be provided by acoustics, seismic vibrations, etc. An SEM image of early nickel nanowire array (b).

EXPERIMENT

Cantilevered Beam Bending

The foremost experiments were conducted at the macroscale to identify the nature of any magnetostrictive sensing response of iron-gallium alloys to applied bending loads, where the top and bottom of the beam experience equal yet opposite stresses. The primary material sample was a cantilevered beam of single crystal $Fe_{84}Ga_{16}$ (1.58 mm diameter by 32.74 mm long) extracted from a [100] ingot grown using the modified Bridgman technique. After testing, the composition of this specimen was verified with wavelength dispersive x-ray spectroscopy (WDS) measurements to have an average gallium content of 15.72 atomic percent. Mechanical excitation was applied at the beam tip manually and via a dynamic shaker, while the corresponding change in magnetic induction was measured with both a pickup coil of AWG34 magnet wire wound directly on the sample and a GMR sensor located at the fixed end. Large cylindrical permanent magnets surrounded the Galfenol to provide dc bias fields up to approximately 120 Oe.

Rolled Sample

A test was performed on a sheet of polycrystalline Galfenol ($Fe_{81.3}Ga_{18.7}$) that was hot rolled successively at 900 ˚C, 700 ˚C, and 600 ˚C while being doped with +2% molybdenum to improve ductility during the rolling process (Na and Flatau, 2005). The finished sample is 216 mm long by 29.2 mm wide with a thickness of 0.18 mm. The material was cantilevered in a vise with a GMR sensor at its base and an accelerometer affixed at mid-length, and the experiment consisted of simply comparing these signals in response to impulse loading on the beam tip. This was intended to serve two purposes, to measure the magnetic response from a vibrating sample with a large aspect ratio closer to that expected from the nanowires, and to verify the continued presence of sensing magnetostrictive behavior following the rolling procedure.

Nanowire Testing

Advancing the experiments to the nanowire scale, a number of tests were conducted to visualize, manipulate, and measure fundamental properties of iron-gallium nanowires using a Nanoman II atomic force microscope (AFM) from Veeco Instruments, Inc. Topographical scans were performed to observe the nanowires in both cantilevered arrays and as small clusters placed on a silicon substrate. The FESP model cantilever tip of the AFM was also used as a mechanical testing probe for ramp procedures on the cross-section of the array, where the tip was moved rapidly downward into contact with the surface causing the AFM cantilever to deflect upwards. By calibrating the mechanical stiffness of this cantilever and measuring the amount of deflection caused by the material surface, the results can reveal contact forces between tip and sample and approximate stiffness values of the nanowire array (Downey et al., 2006). These ramp tests were repeated in the presence of an external magnetic bias field in order to quantify changes in elasticity and adhesion. Manipulation paths traced by the probe tip allow for deflections and stresses to be applied to structures as small as individual nanowires, with implications for complete mechanical characterization. Magnetic force microscopy (MFM) was performed with a MESP-HM model tip to visualize the magnetic domain structure of these nanowires.

ANALYTICAL MODEL

The change in magnetic induction B due to the bending stresses in a vibrating beam has been modeled using a free energy expression (Wun-Fogle et al., 2005; Atulasimha and Flatau, 2005) summing magnetic, stress, and cubic crystalline anisotropy terms,

$$E = -\mu_0 M_s H \alpha_1 - \frac{3}{2}\lambda_{100}\sigma\alpha_1^2 + E_A \tag{1}$$

where E is the total energy, μ_0 is the permeability of free space, M_s is the saturation magnetization, H is the magnetic field, λ_{100} is the magnetostriction coefficient, σ is the axial stress, and E_A is the crystalline anisotropy. The α term represents the direction cosine of the magnetization, where the subscript '1' denotes the axial coordinate of the beam parallel to the stress and field. The iron-gallium material is considered to have a cubic structure with a crystalline anisotropy (Rafique et al., 2004; Carr, 1958) that reduces to $E_A = k_1\ \alpha_1^2\ \alpha_3^2$ in the case of two dimensional bending. Taking an energy weighted average (Wun-Fogle et al., 2005) and converting the magnetization results via $B = \mu_0 (M + H)$ yields the expected induction value at every stress and field state throughout the beam.

RESULTS

The paramount result from the single crystal beam testing is that under an appropriate magnetic bias field, a sinusoidal force of +/- 4 N applied to the tip results in a strong 0.3 T change in the magnetic induction of the material that appears as a rectified version of the input force signal, as demonstrated in Figure 2. This behavior is due to the tensile stresses on one side of the beam trivially acting in the same axial direction as the bias, while the opposing compressive loads tend to orient the magnetic moments perpendicular to this field direction and have a much larger rotation range. Thus, as the sample vibrates the compressive surface of the beam during each half-cycle dominates the magnetization change and the resultant induction signal appears at twice the frequency of the beam motion. Figure 2 also displays the magnetic induction result of the analytical model to compare with the experimental data. These results were generated at 67 Oe of dc magnetic bias to match the tests and the imperfect force signal was used as an input. As this figure shows the model matches well with the amplitude of the data and captures the rectified magnetic behavior seen in the experiment due to the implicit coupling of stress and magnetization effects.

Measurements taken from the rolled polycrystalline beam when subjected to an impulse input are plotted in Figure 3. The top graph shows beam displacement and magnetic induction change measured from the accelerometer and the GMR sensor, respectively. Note that as both signals damp out the induction remains at twice the frequency of vibration, in agreement with the bending experiments and model predictions from previous work. The bottom graph depicts the auto power spectrum of each signal, where the GMR data is primarily at 6.2 Hz but does contain some portion of the first natural frequency of 3.1 Hz. This is most likely due to the fact that the GMR sensor will inherently include some component attributed solely to the motion of the magnetic material in front of it. The magnetic results of the single crystal sample did not exhibit this phenomenon primarily because the amplitude of beam motion was much lower due to greater stiffness. The result of this test is promising if only because it confirms that the hot-rolling procedure has not overly degraded the magnetostrictive performance of the material and remains a viable source of thin films. These films have applications in a number of structures, as they provide a malleable yet durable medium for vibration, strain, or crack detection.

Force and deflection data collected from a ramp test on the nanowire array is plotted in Figure 4(a). A number of key features can be extracted from this graph, including that the adhesive force between the AFM tip and sample is only 30 nN compared with the 140 nN measured from a silicon test grid. The equal slope of the approach and retract lines signifies purely elastic deformations, but the delayed return to the original state is a symptom of contamination on the nanowire surface. To determine the effect of applied magnetic field to this data, the array was also subjected to ramp tests with a small permanent magnet affixed to the specimen holder, which provides approximately 150 Oe of external field along the axial coordinate of the nanowires. The result of this biased experiment is plotted in Figure 4(b). The first observation is that the sample has stiffened by 6.5%, a phenomenon that is not

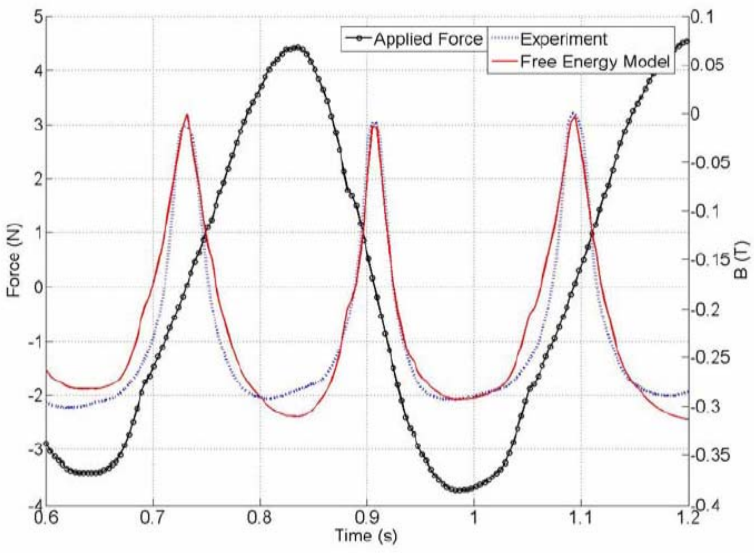


Figure 2. Experimental magnetic induction signal measured with the pickup coil of the single crystal iron-gallium beam due to the applied manual loading of +/- 4 N, compared with prediction from the free energy model.

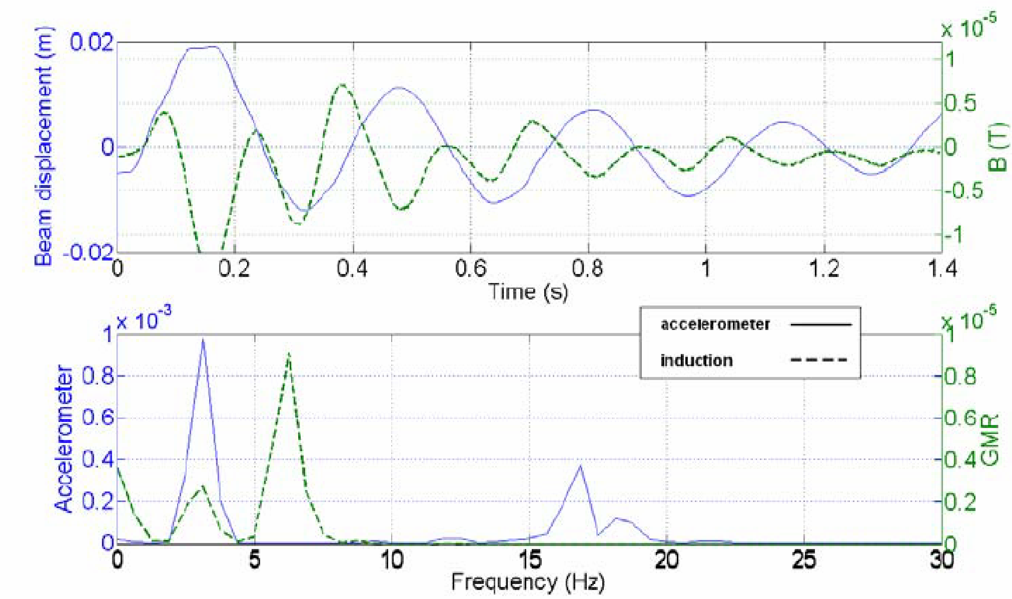
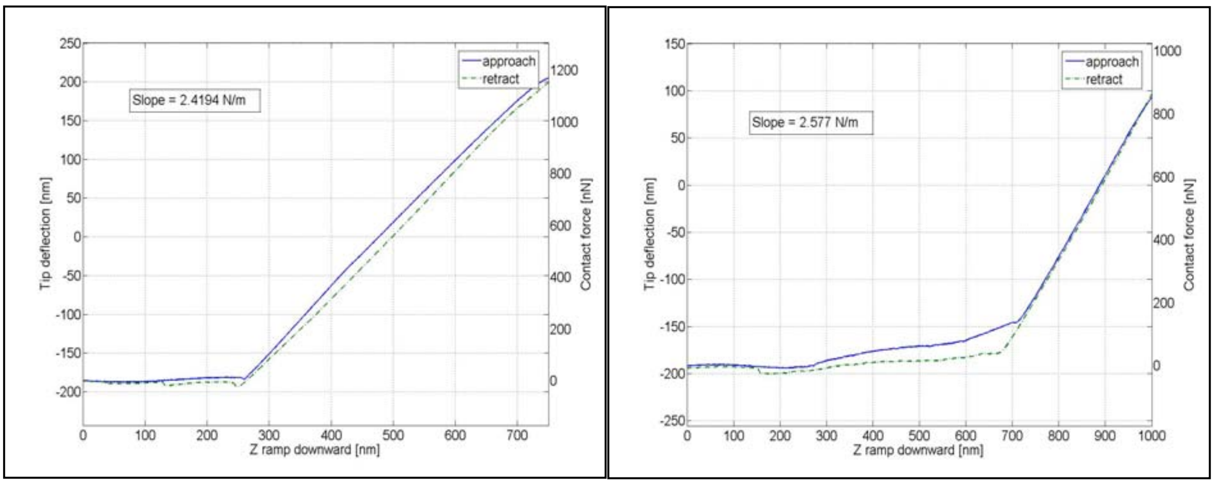


Figure 3. Beam vibration and magnetic induction signal from impulse loading on the rolled polycrystalline sample (top), and auto power spectrum vs. frequency revealing the dominant component of induction at twice the natural frequency (bottom).

uncommon in magnetostrictive materials subjected to near-saturating external fields. Unfortunately, due to the lack of precise geometric knowledge of the surface feature this magnitude cannot be directly compared with the macroscale material behavior. It is also noted that the spacing between the approach and retract curves grows near the point of contact, which correlates to an increased adhesion of 75 nN and suggests a higher risk of permanent attachment between adjacent wires. The last result of interest is the presence of a small upward slope across the region prior to solid contact between AFM tip and sample. Typically this appears when there is some distant part of the AFM cantilever interacting with the specimen before the tip reaches the surface. It is possible that in this case the permanent magnet has induced sufficient deflection or strain in the overall sample holder to cause this minor interference.



(a) (b)

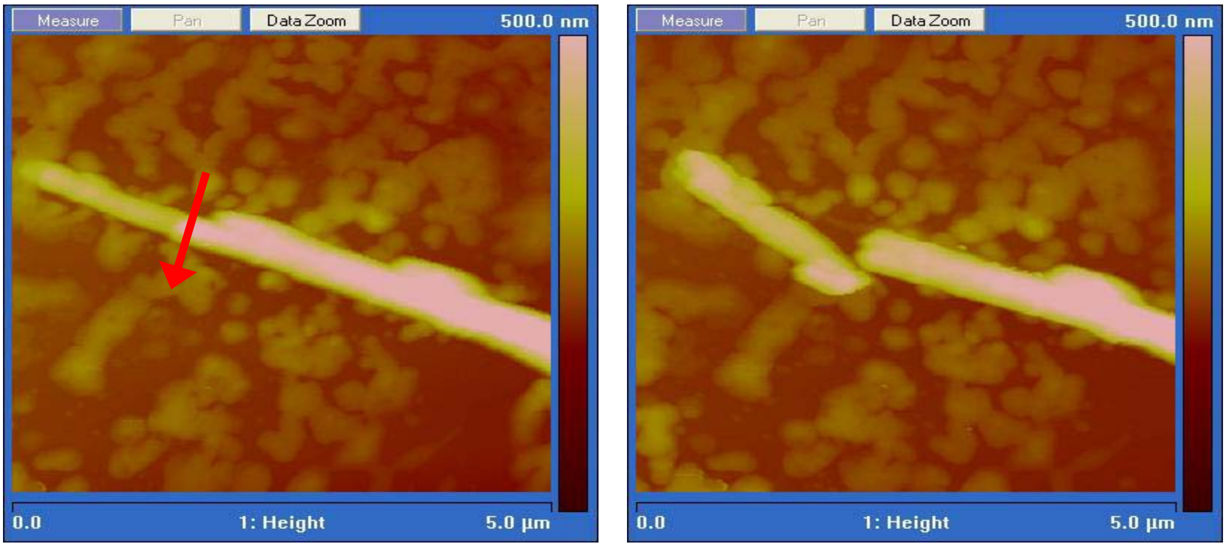
Figure 4. Ramp tests performed along the cross-section of the nanowire array without (a) and with (b) 150 Oe permanent magnet. The slope increased by 6.5% and the contact force grew from 30 nN to 75 nN.

The nanomanipulation capabilities of the AFM have been explored with a few basic exercises. A small cluster of free nanowires were deposited onto a silicon substrate and the end of a protruding wire was pushed with the AFM tip in a move represented by the arrow in Figure 5(a). As can be seen from the adjacent image, this maneuver actually broke the wire in two at the point of contact. In the case of a wire cantilevered from the array, this move would be expected to bend the wire rather than fracture it, but by laying on the rough silicon substrate there are clearly numerous attractive and frictional forces acting to pin this wire at various points along its length. The observation of this type of brittle failure is rather unexpected considering that all previous evidence pointed to rugged wires exhibiting large elastic and plastic deformations, but it presents an interesting course for further study. Figure 6 demonstrates another operation, this time on a wire lying across several others. Here the desired result is obtained as the wire is displaced into its new position following the AFM tip move.

An intriguing result can be seen in the MFM images of Figure 7. Here the topography is subtracted out of the phase data, which yields a qualitative representation of the magnetic moment orientation relative to the plane. Thus, the dark regions represent moments pointing into the page, with lighter colors sticking out. The most notable feature is that the diagonally laying wire has a distinct change in direction, with a very dark region at its tip jumping to a lighter zone when it intersects the vertical nanowire. This distribution suggests the presence of multiple magnetic domains in a single wire. A complete investigation of the size and effect of stress and field on this domain structure is underway.

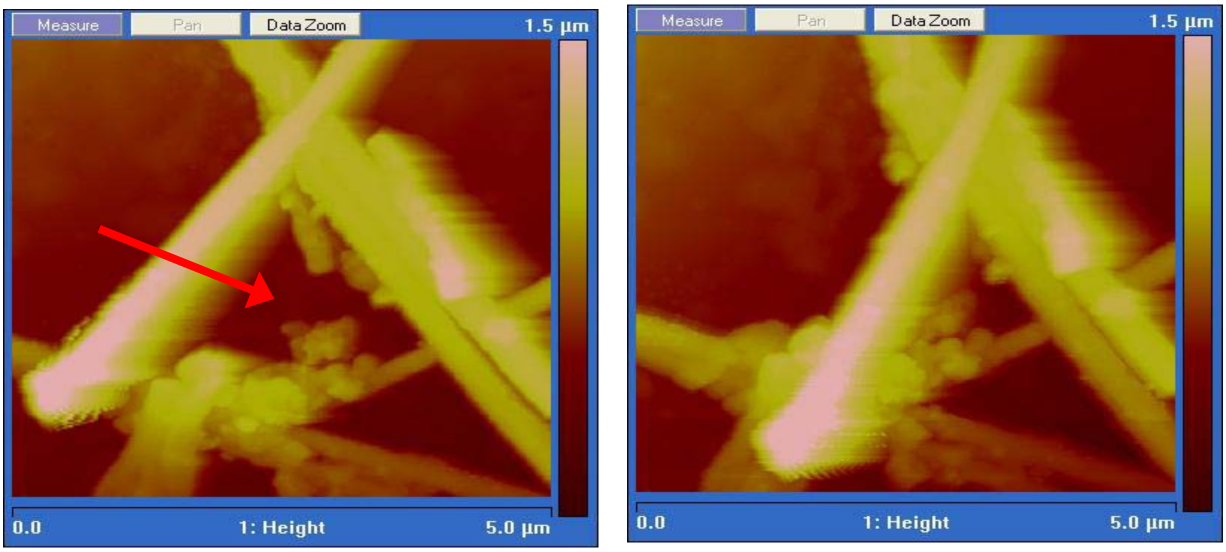
CONCLUSIONS

Iron-gallium alloys were investigated as magnetostrictive sensors operating in pure bending. Vibrations in cantilevered beams of both single crystal and hot rolled samples resulted in a change of up to 0.3 T in the material, with the compressive stresses dominating the output and yielding an induction signal occurring at twice the frequency of motion. A free energy model of the system accurately described this process and can be used to predict the material response to arbitrary loads. A cantilevered array of Galfenol nanowires has been visualized and mechanically tested with an AFM. The wires were found to vary in length and show some external contamination, but improvements in the fabrication and handling processes should enhance their uniformity. When subjected to ramp tests, the wires exhibited purely elastic deflections with a bulk stiffness of 2.4 N/m and adhesive forces of only 30 nN. The application of 150 Oe of magnetic field yielded a slight increase in stiffness but a larger adhesion that needs to be further studied for detrimental effects to the sensing behavior. The capability of the AFM to be a useful nanomanipulation tool has been demonstrated. Initial MFM images reveal a unique magnetic domain structure that differs between individual nanowires.



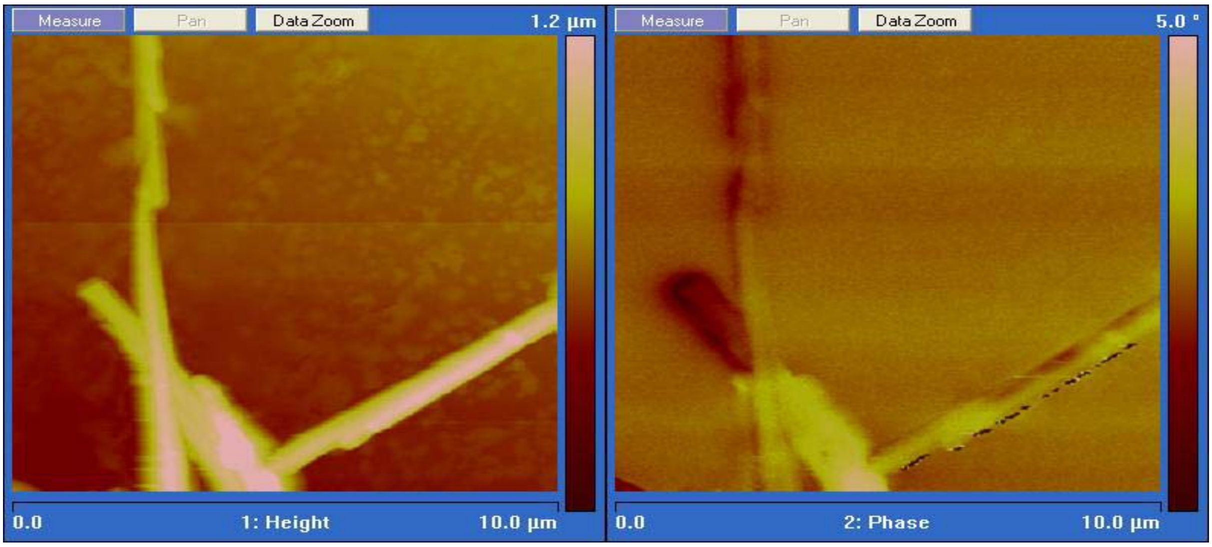
(a) (b)

Figure 5. AFM height image of protruding nanowire (a), with arrow indicating direction of the manipulation path, and resultant fractured wire (b).



(a) (b)

Figure 6. AFM height image of nanowire cluster (a), with arrow indicating direction of the manipulation path, and resultant displaced wire (b).



(a) (b)

Figure 7. Height (a) and MFM phase (b) scans of a group of nanowires, showing a wire with distinct domains oriented both into and out of the page.

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