

Structural Magnetostrictive Alloys:

From Flexible Sensors to Energy Harvesters and Magnetically Controlled Auxetics

Prof. Alison Flatau & MANY OTHERS!!!

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Outline



Intro to Magnetostriction

Actuators: the "direct" effect

Sensors: the "inverse" (or Villari) effect

Structural Magnetostrictive Alloys

Magnetostrictive, magnetic & mechanical properties

Thin, highly textured rolled sheet

Introduction to auxetic behavior

Applications that use Galfenol and/or Alfenol

Bending sensor applications

Implantable wireless bone fixity sensor (Tech Univ. Dresden)

Nanowire sensors (Univ. Minn. & UMD)

Applications for auxeticity?

Micro-motors (Kanazawa University, Japan)

Energy Harvesting (Kanazawa Univ, UMD & Techno Sciences, Inc., Oscilla Power)

Summary

What is Magnetostriction?



"A change in dimensions exhibited by ferromagnetic materials when subjected to a magnetic field." (Random House Dictionary)

- •1st documented by James Joule in 1842. – Attribute of iron, nickel, and other ferromagnetic metals
- Effect will not "de-pole"
 Above Curie temperature (~750°C for Fe-Ga alloys) domain ordering vanishes, but order returns when cooled below Curie temperature.



What Produces Magnetostriction?

Magnetostriction arises from magnetic-field induced changes in crystal structure

•Reorientation of electron spins to align fields with an externally applied magnetic field changes inter-atomic spacing while maintaining conservation of volume.

•Simple body centered cubic structure of Fe has maximum magnetostriction (λ_{sat}) along the <100> directions. ($\lambda_{sat < 100>} > \lambda_{sat < 110>} > \lambda_{sat < 111>}$)





Typical uses for Magnetostriction?

ACTUATION

- Moving a mass
- Producing a force



SENSING & ENERGY HARVESTING

- Detecting motion or strain
- Detecting a force or moment
- Voltage in a surrounding coil proportional to dB/dt



$$\varepsilon = \sigma / E_y^H + d_{33}H + \alpha \Delta T$$

Sensing along [100] axis modeled by the "inverse effect" (Villari effect):

$$B = d_{33}^* \sigma + \mu^\sigma H + P \Delta T$$

where *H=nI*

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The 33 subscript indicates the effect and response are in the same directions

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Magneto-mechanicalwoorpling/terms

The Direct Effect (Joule magnetostriction):

The change in the dimensions of a ferromagnetic body caused by a change in its state of magnetization.





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The Inverse Effect (Villari effect):

A change in magnetic state caused by a change in stress.



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A change in magnetic state caused by a change in stress.



19% Ga single crystal Galfenol

The Direct Effect:

The change in the dimensions of a ferromagnetic body caused by a change in its state of magnetization.

The Inverse Effect:

A change in magnetic state caused by a change in stress.





Production grade Terfenol-D

The Direct Effect:

The change in the dimensions of a ferromagnetic body caused by a change in its state of magnetization.



The Inverse Effect:

A change in magnetic state caused by a change in stress.



Comparison of direct effect in Galfenol (left) and Terfenol-D (right)



Galfenol data from Dr. J. "Atul" Atulasimha's dissertation (2006) Terfenol-D data from ETREMA Products, Inc. product sheet

MOKE images of quenched Fe₈₁Ga₁₉ under compressive stress/strain (0-1500 microstrain)

Images of Galfenol obtained using Evico Magnetics GMBH Magneto-Optic Kerr Effect (MOKE) microscope











MOKE images of quenched Fe₈₁Ga₁₉ under compressive stress/strain (0-1500 microstrain)

Images of Galfenol obtained using Evico Magnetics GMBH Magneto-Optic Kerr Effect (MOKE) microscope







[010]

100 *µ*m

MOKE images of quenched Fe₈₁Ga₁₉ under magnetic field (0-200+ Oe)

Images of Galfenol obtained using Evico Magnetics GMBH Magneto-Optic Kerr Effect (MOKE) microscope





[010]

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Summary

Common Magnetostrictive Materials

STRUCTURAL Magnetostricitve Alloys

| | Terfenol-D | Iron | Nickel | Fe _{1-x} Co _x | Fe _{1-x} Al _x | Fe _{1-x} Ga _x |
|--|---------------------------|---|---------|---|-----------------------------------|-----------------------------------|
| $3/2 \lambda_{s}$ (µstrain) | 1600-2400 | -24 | -66 | 140-220 | 185 | ~400 |
| Modulus (GPa) | 25-35 | 200 | 207 | 270 | 65 | 65 |
| Relative Permeability | 2-10 | 5000 | 100-600 | ~3000 | 100 | 70-100 |
| Saturation Magnetization | 1.0 T | 1.6-2.2 T | ~0.6 T | ~2T | 1.4 T | 1.6 T |
| Ultimate Tensile Strength (MPa) | 28 (& very brittle) | 400 | 500 | 300-870 | 500-800 | 500-600 |
| Hysteresis in λ- H and B-H curves | Moderate | Low | Low | Very Low | Very Low Good | Very Low |
| Poor magnetostriction Poor magnetic & mechanical properties | | magnetostriction Good mechanical properties | | Good magnetic and mechanical properties | | |

Comparison of binary Fe-Ga (λ =400 µ ϵ), Fe-Co (λ =150µ ϵ) and Fe-Al (λ =175µ ϵ)



Galfenol - $Fe_{1-x}Ga_x$ (0.13 < x < 0.35)

Gallium + Iron (Fe) + Naval Ordnance Laboratory Alfenol - Fe_{1-x}Al_x (0.13 < x < 0.35)

Alumium + Iron (Fe) + Naval Ordnance Laboratory

X% Fe replaced with Ga (or Al) Start with BCC Iron Lattice

Galfenol - $Fe_{1-x}Ga_x$ (0.13 < x < 0.35)

Gallium + Iron (Fe) + Naval Ordnance

Alfenol - $Fe_{1-x}Al_x$ (0.13 < x < 0.35)

Alumium + Iron (Fe) + Naval Ordnance

Plotting single crystal data at 15 kOe from [Restorff et al. JAP 111, 023905 (2012)]

Magnetostriction of Galfenol

•Sensitive to composition and to heat treatment ^[Clark et al. 2003]

Temperature Dependencies of λ(H) & M(H) in 19% Ga Fe-Ga Single Crystal

Fe₈₁Ga₁₉ Single crystal @ 45.3 MPa. -22°C (red) to +80°C (blue)

Note: hysteresis is induced by transducer bushing - is not in FeGa rod

The Direct Effect: The change in the dimensions of a ferromagnetic body caused by a change in its state of magnetization.

Rolling Steps to Lower Fabrication Costs

Warm Rolling

• Binary alloy experienced intergranular fracture

Cold Rolling

Hot Rolling

- Arc-melted buttons
- Roller set in 0.0031 inch
- 1000/800°C for 10 min. every 2 passes
- Seal in 321 Stainless

Intergranular Fracture Mode Changed to Intragranular with trace Additions of B or NbC

- Fracture surface of the B-free Galfenol clearly appeared to a typical intergranular mode by crack propagation along grain boundaries.
- Fracture surface of the B-added Galfenol was changed to a transgranular mode by crack propagation through the grains and the grain size was also small (grain refinement).
- Boron has a great effect on improvement of ductility due to suppressing grain boundaries fracture.

Rolling Steps

Hot Rolling

- Arc-melted buttons
- Roller set in 0.0031 inch
- 1000/800°C for 10 min. every 2 passes
- Seal in 321 Stainless

Warm Rolling

- Roller set in 0.002 inch
- 600/400°C for 10 min. every 1 pass
- Intermediate annealing @ 800°C for 2 hrs in Ar

Cold Rolling

- Roller set in 0.0005 inch
- Subsequent annealing @ 1000 ~ 1200°C under flowing argon, sulfur atmoshere

As-rolled sheet: $\lambda_{sat} \sim 60 \text{ ppm}$ Goss or Cube texture $\sim 7X$ more magnetostrictive

Electron BackScatter Diffraction (EBSD) images show as-rolled grains are tilted at random orientations λ_{sat} ~60 ppm

EBSD legend for grain orientation

cube textures)

Once rolling achieved, need to develop a preferred texture in rolled sheet

Texturing of Polycrystalline Galfenol

ARGON ANNEAL

Inexpensive production of rolled sheet Galfenol (Alfenol) relies on the abnormal grain growth of surface textures Goss texture {110}

 $\lambda_{100} \sim 200 \text{ ppm}$ Easily reproduced Cube texture {100}

> $\lambda_{100} \sim 350 \text{ ppm}$ Difficult to produce

Texturing of Rolled Sheet Alfenol

- NbC-added 20% Alfenol rolled sheets annealed under a sulfur atmosphere.
- Single (011) grain is abnormally grown, with λ = ~177 ppm.
- Large single (011) grains were fully grown in the samples, covering 80-99% of sample surface for sample thicknesses of 0.2, 0.35, and 0.5 mm.

Seeking additional insights lead to an unexpected result



(3) Tensile Test

- 1) Test specimen thickness: 0.1, 0.2, 0.35, 0.5 mm
- 2) The test specimens were prepared according to ASTM A370-12a (Standard Test Methods and Definitions for Mechanical Testing).
- 3) Machine: MTS 858 Mini Bionix 25kN servo-hydraulic load frame at a rate of 0.0025 mm/s. The strain was monitored with a gauge length of 25 mm.



Tensile Loading to Study Slip Planes Two Test Configurations: [100] & [110] Single Crystal Samples

- Emphasize mechanical anisotropy
- High symmetry crystal orientations
- Use axial loading (tension)







Single Crystal Galfenol Samples

- Single crystal **Fe_{100-x}Ga_x** x = 15.8, 17.9, 20.4, 25.3, 31.0 & 33.0
- Composition verification: Laue X-ray back reflection
- Dogbones Cut: EDM along (100) face in [100] & [110] directions
- Strain measurement: Bi-directional **strain gage** rosettes **[100]** & **[110]**

RUS samples (notches) cut from dogbones after testing



Monotonic Tension Tests

Setup & Procedure:

Longitudinal and transverse strain gages Room temperature MTS machine pull - constant rate mode @ 0.5 μm/s Elastic regime ~ 3 με/s Plastic regime ~ 18 με/s Pulled specimens ~ 2% strain

Testing under magnetic fields also conducted

from Dr. R. Kellogg's Dissertation



[100] Tension Test Experimental Results

σ

[100]

- Load applied along [100] direction, measure strain responses in the longitudinal & transverse directions
- Ratio of stress & strain give elastic modulus, E
- Negative ratio of longitudinal and transverse strains gives the <u>Poison ratio</u>, v, (nu)



The test speed is 0.5 μ m/sec in each test with constant load limits.

[110] Tension Test Experimental Results

σ

1100.

[110

- Load applied along [110] directions
- Samples get longer and wider (and really thin!)
 - Both stress-strain curves have a positive slope!!
- The alloys have a negative Poisson ratio
 Alloys are AUXETIC in [110] directions



Auxeticity in response to applied mechanical stress



σ

1200.

Magnetically induced auxeticity in Galfenol?



....is there a Delta Poisson Ratio Effect?

±10mT/s

Yes! Poisson Ratio (negative ratio of longitudinal to transverse strain) changes when a DC magnetic field is applied along the sample length



Typical results from [110] Galfenol Samples (31% Ga shown) :



For compositions of <17% Ga, sample width increases more than the length!

Η

12001

[110]

Poisson ratio is less than -1.0!

Results from the 16% Ga [110] Galfenol Sample:



Comparison of models and experimental Poisson ratio data.

Poison ratio values under a saturating magnetic field (400 mT) for different compositions of FeGa from an energy-based analytical model, density functional theory simulations, and ratios of measured strain values





Poisson ratio data for Galfenol of different compositions





GAS MODEL FOR AUXETIC EFFECT IN BCC CRYSTALS





Atoms that move away from one another



Atoms that approach one another

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Summary

Early Bending Tests



- Tensile stresses act trivially in same direction as bias field
- Compressive side of
 beam controls response
- Bias shifts where max coupling occurs
- GMR measures volumeaveraged change in induction (NOTE: v



(NOTE: will show later these assumptions were only partly correct!)

Bio-inspired thin "whisker-like" bending sensors







Bio-inspired thin "whisker-like" bending sensors







Magnetostrictive biased magnetic field sensor Apicella, Caponero, D. Davino & C. Visone (2018); Univ. Sannio-Benevento, ITALY



Integrated Laboratory for Research on New magnetic devices and Innovative Technologies

UNIVERSITY of SANNIO - Department of Engineering

Galfenol Resonant Sensor for Indirect Wireless Osteosynthesis Plate Bending Measurements *Fischer et al. (2009); Wentzel et al. (2009); Tech. Univ. Dresden*



Planar coil (22 turns)



Stress induces a change in permeability. Changes resonance of planar coil

Excitation/pickup coil 1 cm above bone plate and 6 cm radially from center of 10 x 2 x 100 mm³ sample

Loading of 700 grams produces a 70kHz shift in resonance

Fischer et al. (2009); Wentzel et al. (2009); Tech. Univ. Dresden



Micro spherical motor Prof Toshiyuki Ueno Kanazawa Univeristy, Japan



Galfenol –> drive element and magnetic core

used at endoscope for e.g. minimally invasive surgery

Joint of micro robot (amusement)

Movie of movement



Cilia: Nano-scale transducers

- Human cochlea: ~98mm³
 ~ 35 mm long with varied width & thickness.
- Basilar membrane increases in width and thickness with distance
- Cochlea acts as a low pass spatial filter, with high frequency response near stapes/oval window
- Location of maximum motion correlates to the detected frequency



/www.vimm.it/cochlea/cochleapages/theory/hydro/hydro.htm

Schematic of the cross-section of the human-ear cochlea



Ref: http://hyperphysics.phy-astr.gsu.edu/hbase/sound/corti.html

Cochlea/Cilia-based Sensors

- ~3500 inner hair cells with
 ~50 cilia/cell
- ~12,000 outer hair cells with
 ~150 cilia/cell
- •Cilia dimensions: ~2-6 μm long ~100 nm at their base ~250 nm at their tip (ohc)
- Cilia tips are tethered to neighboring cilia

Cilia from outer hair cell





Cilia & Whiskers as sensors





Microsystems Workshop, June 2006. Hilton Head, SC, USA

Whisker deflection creates chemical potential change that fires neuron



Magnetostrictive whisker

Whisker deflection creates chemical potential change that fires neuron Wire deflection creates magnetization change that changes GMR resistance



Fe-Ga NW: DC Electrodeposition



□ FeGa nanowires are grown using a single bath containing Fe⁺² and Ga⁺³ ions with Sodium Citrate as complexing agent.

Mechanical properties of Fe-Ga NWs



McGary et al. J. Appl. Phys 99 (2006) Downey et al. J. Appl. Phys 103 (2008)



During testing the soft AFM cantilever deflects upward

Nano-manipulator

UNIVERSITY OF MINNESOTA







Tensile Tests



| Wire # | Diameter (nm) | Length (µm) | Ultimate Strength (MPa) | Young's Modulus (GPa) |
|--|---------------|---------------|----------------------------|--------------------------|
| 1 | 105 +/- 5 | 9.80 +/- 0.01 | 1232 +/- 147 | 58.3 +/- 6.0 |
| 2 | 135 +/- 5 | 13.8 +/- 0.01 | 1202 +/- 119 | 59.4 +/- 6.3 |
| 3 | 225 +/- 5 | 36.8 +/- 0.01 | 1084 +/- 60 | 54.9 +/- 7.1 |
| 4 | 130 +/- 5 | 7.55 +/- 0.01 | 1050 +/- 91 | 58.9 +/- 8.6 |
| Bulk Fe ₈₃ Ga ₁₇ [100] (Kellogg et. al, <i>Acta Mater.</i> 52, 2004) | | | 500 | 65 |

Resonance Method Results

Wire attachment assumed fixed Frequencies swept with piezo Modulus related to resonance:

 $f_0 = \beta_0^2 / 2\pi \sqrt{\left(EI / mL^4\right)}$

Results are lower bounds Two distinct values suggest a compositional variation



Q - 170

| Wire # | Diameter (nm) | Length (µm) | Resonance Frequency (kHz) | Young's Modulus (GPa) |
|--------|---------------|---------------|------------------------------|--------------------------|
| 5 | 100 +/- 5 | 19.4 +/- 0.01 | 86.6 +/- 0.05 | 44.2 +/- 8.2 |
| 6 | 120 +/- 5 | 11.6 +/- 0.01 | 304.3 +/- 0.05 | 45.1 +/- 7.9 |
| 7 | 225 +/- 5 | 7.88 +/- 0.01 | 1781.1 +/- 0.05 | 93.1 +/- 8.3 |
| 8 | 125 +/- 5 | 4.43 +/- 0.01 | 3143.0 +/- 0.05 | 93.8 +/- 15 |

EBSD study on uniform FeGa NW



• Uniform FeGa NW (ø ~ 150 nm)





<110> orientation along NW length

EBSD study on FeGa nanowire



 Uniform FeGa NW
 <110> orientation along NW length

011













Scale bar: 0.5 µm

| Energy term | Maximum cost [erg/ cm ³] |
|-------------------------------|---|
| Exchange coupling | 3 x 10 ⁴ |
| Magnetocrystalline anisotropy | 3 x 10 ⁵ |
| Magnetoelastic | 2.5 x 10 ³ / MPa |
| Shape anisotropy | 7 x 10 ⁶ |
Magnetic studies of Multilayer





Scale bar: 0.5 µm



Fabrication of Multilayer Fe-Ga/Cu NW

Electrodeposition

- Fe/Ga/Cu plating bath
- Deposition potential of -1.12V and -0.8V
- Grown in Anodized Aluminum Oxide (AAO) templates
- 150nm diameter, 200nm spacing

Nanowires separated and Isolated



Cathode





FeGa/Cu NW: Structural characterization





 Use of citrate baths for electrodeposition; <u>highly textured growth</u> even for FeGa-Cu multilayered nanowires. => high magnetostriction

 \checkmark Extensive structural characterization by XRD and TEM reveals a strong < 110 > textured Fe_{1-x}Ga_x

Magnetic studies of Multilayer



FEM (Magpar) of Fe-Ga segment





FEM predicts that at remanence, a nanowire with aspect ratio of two actually forms two opposite vortex domains at each end separated by a domain wall near the center.

Moment rotation in Fe-Ga NW and in Fe-Ga/Cu NW



Magnetic force microscopy (MFM)



Multilayered, low aspect ratio nanowires



Multilayered, low aspect ratio nanowire has multiple domain structures / magnetization states

(H ~ 550 Oe) ~ 5 °

~ 105 °



Park et al. J. applied physics (2010)



The expected length changes of 10 μ m long multilayered (50:50) Fe-Ga:Cu NW with a magnetostriction of <~50 ppm along <110> are estimated to be in **range between 0.1 nm and 0.25 nm =** below the vertical resolution of our AFM system.

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Sample preparation and AFM measurement









Direct Effect λ-H along <100> and <110>

<100>, 19 at. % Ga

<110>, 18 at. % Ga



18-19 at. % Ga, single crystal

Pressure sensing: GMR sensing of magnetostrictive NW array





Partially etched AAO allows free standing NW array which is attached on a top substrate.

We would like to observe contact between NWs to GMR surface in SEM, using a nano-manipulator.

- Minimize stress on GMR sensor
- Appropriate NWs contact on GMR surface
- NW deformation

=>Controlled experiment

Magnetostrictive Fe-Ga/Cu Nanowires



for Sensing Applied Pressure

| | SEM: nanowire array compression | | | | | | |
|-----|---------------------------------|------------|----------------|-------------|----------|------|-----------|
| (a) | NM | /s | Si wafer | (b) | | | |
| | | | - | | 1 | In . | 1 |
| | ALC: N | | | a to a to a | | | |
| 1 | | 01 | | | | | |
| UMD | SCI | 5.0KV X250 | 10µm WD 23.1mm | UMD | SEI 50KV | 2050 | 10µm WD 2 |

- No resistance change was observed by Cu NW array sample.
- The coarse step (~ 1 μm) causes 2 times larger GMR % value change (0.5 0.65%) than the value change (0.2 0.3%) caused by the fine step (~ 100 nm).
- Estimated values of sensor sensitivity are 1 ~ 4 mΩ/kPa.



 $[GMR\% = ((R(H) - R(H_{max})))/R(H_{max})\%]$

Applications for Auxeticity include:

- Impede crack propagation
- Increased indentation resistance
- •Reduced fiber pull-out
- •Press fit fastening devices control actively with Galfenol?





Nano indenter experiment to visualize effect of "magnetically tunable" auxeticity

Hardness is proportional to $1/(1-v^2)$





NANO INDENTATION DATA: HARDNESS AT 18% Ga Galfenol

Vicker's Hardness ~600MPa in Iron

Without magnetic field: Hardness ~ 400-700 MPa Average Hardness <mark>~550 MPa</mark>

Applied force of 0-980 microNewton Displacement scale is 0-300 nm

With a DC magnetic field ~150mT (>H_{sat}): Hardness ~ 0.95- 2GPa Average Hardness <mark>~ 1.5GPa</mark>

Applied force of 0-980 microNewton Displacement scale is 0-125 nm

Measured Hardness Ratio 2.72 Predicted Estimate of 2.51!!





Benchtop Wireless Torque Sensor Setup





Close up of Galfenol patch on Aluminum shaft and flux piece with attached permanent magnet, coil and Hall effect sensor



Black: COTS torque sensor Blue: averaged Hall effect sensor



Galfenol and Alfenol patch output for clockwise and counterclockwise torque loading



Spinning shaft at 360 and 1800 rpm Upper: COTS Torque sensor Lower: Hall effect sensor output (mean of 4)



Oscilla Power is developing cost-effective magnetostrictive devices to harvest wave



https://vimeo.com/154632694?swipeboxvideo=1

energy

Oscilla Power's Magnetostrictive Wave Energy Harvesters



Oscilla Power - Transforming Wave Energy to Electricity



Surface Float Arrival in Seattle







rictive Wave Upper end rigidly attached to float.

Lower end is tethered by cables to a subsurface heave plate.

Waves cause oscillatory axial compression in Alfenol rods





Thank you

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Examples of <u>Structural</u> Magnetostrictive Alloys



Torque Sensor



Large-scale Wave Energy Harvesting



Nanowires



Micro-scale motion



Courtesy Prof. Ueno, U. Kanazaga Courtesy of TUDresden

Implanted Bone-Plate Sensor



Courtesy of Profs. Fischer & Marschner TI IDresden

Rolled Sheet