Tuning magnetic anisotropy in nanostructures for biomedical applications

IEEE
MAGNETIC:

bizka

::taler

... the story of anisotropy...

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NIVERSITY OF SOUTH FLORIDA

 University of South Florida – 12<sup>th</sup> largest in the country – 47000 students

 USF Physics offers the <u>only Applied Physics Ph.D. program</u> in the State of Florida....PhD near the beach! (www.physics.usf.edu) 3





# **Functional Materials Lab@ USF Students**

Dr. Hari Srikanth Professor

Dr. Manh-Huong Phan **Research Professor** 

# Postdocs

Dr. Raja Das (Asst. Prof. Phenikaa Univ, Vietnam) Dr. Javier Alonso (Asst. Prof. U. Cantabria, Spain) Dr. Hafsa Khurshid (Asst. Prof. American Univ of Sharjah, UAE)

Dr. Zohreh Nemati Dr. Vijaysankar Kalappattil Joshua Robles

#### Dr. Eleanor Clements

Richa Madhogaria Valery Ortiz Hana Nazari Yen Pham Jason Cardarelli (UG)

#### Our group's current focus areas....

- Magnetic Nanostructures
- Nanomedicine
- •Tunable Microwave Materials
- Spin Seebeck Effect
- Multicaloric oxides
- Helical magnets
- •Magnetic Refrigeration
- Giant Magnetoimpedance Magnetic Sensors

DC, AC Magnetization Transport measurements RF transverse susceptibility MCE **GMI** MOKE SAR for hyperthermia Spin Seebeck Effect Spin Hall Magnetoresistance





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# Outline



Magnetic anisotropy



Anisotropic nanoparticles for biomedical applications

Dancing with the SARs





# Magnetic Anisotropy -basic idea



# $U_{A} = K_{1}(\alpha_{1}^{2}\alpha_{2}^{2} + \alpha_{1}^{2}\alpha_{3}^{2} + \alpha_{2}^{2}\alpha_{3}^{2}) + K_{2}\alpha_{1}^{2}\alpha_{2}^{2}\alpha_{3}^{2}$ Cubic

Configuration

...And if you thought that's it.....think again!

Surface

Shape

Strain/Striction

Unidirectional/Exchange





#### Anisotropic nanoparticles are omnipresent!



#### Magnetotactic bacteria

Magnetite crystals in Allen Meteorite from Mars Ferrite crystals produced in plasma chamber at CMU







Experimental observations and nucleation and growth theory of polyhedral magnetic ferrite nanoparticles synthesized using an RF plasma torch

R. Swaminathan a,\*, M.A. Willard b, M.E. McHenry a,\*

<sup>a</sup> Department of Materials Science and Engineering. Carnegic Mellon University, Patroburgh, PA 13213, United States <sup>b</sup> US Naral Research Laboratory, Physical Metallurgy Branch, Code 6320, Washington, DC 20375, United States Received 18 July 2005; received in revised form 9 October 2005; accepted 12 October 2005





# Tuning the anisotropyNANORODSOCTOPODS

The high aspect ratio of the nanorods gives rise to an enhanced **shape anisotropy**.

By deforming the surface of the nanoparticles, the **surface anisotropy** can be increased.









## Transverse susceptibility using a resonant RF TDO method

- Ultrastable Tunnel Diode Oscillator
- LC Tank circuit self-resonant at ~ 10 – 25 MHz
- Operates in a PPMS
- Sensitivity 1-10Hz in 25 MHz
- Temperature range: 2K < T < 300K
- Variable DC field: 0 < H < 7T



P. Poddar, G. T. Woods, S. Srinath and H. Srikanth, IEEE Trans. Nanotech. 4, 59 (2005)
P. Poddar, J. L. Wilson, H. Srikanth, D. F. Farrell, S. A. Majetich, PRB 68, 214409 (2003)

REVIEW OF SCIENTIFIC INSTRUMENTS

VOLUME 70, NUMBER 7

JULY 1999

Radio-frequency impedance measurements using a tunnel-diode oscillator technique

H. Srikanth,<sup>a)</sup> J. Wiggins, and H. Rees Advanced Materials Research Institute, University of New Orleans, New Orleans, Louisiana 70148



 $\chi_T = \left(\frac{dM_x}{dH_z}\right)$ 

JOURNAL OF APPLIED PHYSICS 104, 063901 (2008)

Transverse susceptibility study of the effect of varying dipolar interactions on anisotropy peaks in a three-dimensional assembly of soft ferrite nanoparticles

Pankaj Poddar,<sup>1,2,a)</sup> Marienette B. Morales,<sup>1</sup> Natalie A. Frey,<sup>1</sup> Shannon A. Morrison,<sup>3</sup> Everett E. Carpenter, and Hariharan Srikanth<sup>1,b)</sup>





Nanoparticles for biomedical applications



Targeted Drug Delivery



Magnetic Heating (Hyperthermia)



**Cell Separation** 



MRI Image Enhancement





#### Magnetic hyperthermia for cancer treatment:

Using magnetic nanoparticles under an external AC magnetic field to target,

heat and destroy cancer cells.



- protein denaturation
- cell membrane restructuring
- cell deactivation
- (driven to apoptosis) around 40-45 °C



NanoTherm<sup>™</sup> Therapy



MagForce AG (Germany)



The standard measure of heating efficiency is the **Specific Absorption Rate (SAR)**:

SAR = Area  $\cdot$  f

Higher SAR → fewer nanoparticles

[5] D. Ortega and Q. Pankhurst, "Magnetic hyperthermia," Cambridge, Royal Society of Chemistry, 60-88 (2013).[6] R. Ivkov et al. Int. J. Hyperthermia, 29(8), 703-851 (2013).





#### Introduction: magnetic fluid hyperthermia



#### **SLP: Specific Loss Power**

SLP=f(f, H<sub>0</sub>, Relaxation Mechanism)



#### Linear Response Theory (LRT)

- non-interacting single domain particles
- Linear susceptibility (H<<H<sub>K</sub>)

$$P = \pi \mu_0 \chi_0 H_0^2 f \frac{2\pi f \tau}{1 + (2\pi f \tau)^2}$$
$$\frac{1}{\tau} = \frac{1}{\tau_B} + \frac{1}{\tau_N} \qquad \tau_B = \frac{3\eta V_H}{k_B T}$$
$$\tau_N = \tau_0 e^{K_e f f V/k_B T}$$

R. Rosensweig, J Magn Magn Mater, 252, 370-374 (2002).C. Dennis, R. Ivkov, Int J Hypertherm, 29(8), 715-729 (2013).













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# Magnetic Hyperthermia

### I. Calorimetric method

### II. AC Magnetometry method



$$SAR = rac{m_s}{m_n} C_p rac{\Delta T}{\Delta t}$$

 $C_p$ : specific heat of the solution  $m_s$ : mass of the solvent  $m_n$ : mass of the nanoparticles  $\Delta T/\Delta t$ : initial slope of the heating curves 30 20 20 20 0 10 0 -10 -20 -30 -15 0 $H_{app}$  (kA/m)

Eneko Garaio, Irati Rodrigo, Jose Angel Garcia  $SAR = Area \times frequency$ 

> AC magnetometry is more accurate and reproducible than other methods.

#### Atkinson Brezovich limit: $H \ge f = 4.85 \ge 10^8 \text{ Am}^{-1}\text{Hz}$



Hea



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# **Motivation**







## Anisotropy



Size





*How to increase M<sub>s</sub> and retain biocompatibility?* 



> Bulk saturation magnetization:

✓ Iron-oxide ----- 92 emu/g
 ✓ Iron ---- 220 emu/g



By creating core/shell nanostructures with a metallic core and an Fe oxide shell, it is possible to achieve nanoparticles with good biocompatibility and high saturation magnetization.





## Synthesis of Core/Shell and Hollow Nanoparticles



### Kirkendall Effect







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### Magnetic Hyperthermia



Z. Nemati,<sup>a</sup> J. Alonso,<sup>ab</sup> H. Khurshid,<sup>a</sup> M. H. Phan<sup>a</sup> and H. Srikanth<sup>\*a</sup>





**Exchange coupled nanoparticles for hyperthermia** 



nature nanotechnology

#### Exchange-coupled magnetic nanoparticles for efficient heat induction

Jae-Hyun Lee<sup>1</sup>, Jung-tak Jang<sup>1</sup>, Jin-sil Choi<sup>1</sup>, Seung Ho Moon<sup>1</sup>, Seung-hyun Noh<sup>1</sup>, Ji-wook Kim<sup>1</sup>, Jin-Gyu Kim<sup>2</sup>, II-Sun Kim<sup>3</sup>, Kook In Park<sup>3</sup> and Jinwoo Cheon<sup>1\*</sup>

- A significant increase in the efficiency of magnetic thermal induction by nanoparticles taking the advantage of the exchange coupling between a magnetically hard core and magnetically soft shell to tune the magnetic properties of the nanoparticle and maximize the specific loss power.
- The optimized core-shell magnetic nanoparticles have specific loss power values that are an order of magnitude larger than conventional iron-oxide nanoparticles.





# Exchange coupled FeO/Fe<sub>3</sub>O<sub>4</sub> nanoparticles

JOURNAL OF APPLIED PHYSICS 117, 17A337 (2015)



# Anisotropy effects in magnetic hyperthermia: A comparison between spherical and cubic exchange-coupled FeO/Fe<sub>3</sub>O<sub>4</sub> nanoparticles

H. Khurshid,<sup>1,a)</sup> J. Alonso,<sup>1,2</sup> Z. Nemati,<sup>1</sup> M. H. Phan,<sup>1</sup> P. Mukherjee,<sup>1</sup> M. L. Fdez-Gubieda,<sup>2,3</sup> J. M. Barandiarán,<sup>2,3</sup> and H. Srikanth<sup>1,a)</sup> <sup>1</sup>Department of Physics, University of South Florida, Tampa, Florida 33620, USA <sup>2</sup>BCMaterials Edificio No. 500, Parque Tecnológico de Vizcaya, Derio 48160, Spain <sup>3</sup>Depto. Electricidad y Electrónica, Universidad del País Vasco, Leioa 48940, Spain







# Nanoparticle legos: Playing with shapes



The Fe<sub>3</sub>O<sub>4</sub> nanoparticles were obtained by further annealing the asprepared exchange coupled FeO/ Fe<sub>3</sub>O<sub>4</sub>.

H. Khurshid et al. Nanoscale, 5, 7942 (2013).





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### Shape dependence: Dancing with the Stars...SARs!



spheres



cubes



octopods





Increased shape or surface anisotropy gives rise to higher heating rates: SAR (octopods) > SAR (cubes) > SAR (spheres)





'Chains' of improving SAR is higher in high aspect ratio nanostructures!



Chains of magnetosomes (magnetic nanoparticles produced by bacteria) show higher heating efficiency than isolated magnetosomes.

High aspect ratio nanostructures (chains, wires, rods...) can give rise to a notable increase of the SAR.

C. Martinez-Boubeta et al., *Scientific Reports* **3**, 1652 (2012); E. Alphandery et al., ACS. Nano. **3**, 1539–1547 (2009).





OMHDA

Fe<sub>3</sub>O<sub>4</sub> nanorods with tunable aspect ratio

OA/HDA = 6.6











 $Fe(CO)_5$ 





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### Structural Characterization



The aspect ratio and volume of the nanorods were tuned.

511 '

60

422

50

20 (degree)

S1

S2

Nanoscale

Check for updates

Cite this: Nanoscale, 2017, 9, 7858

PAPER

311

222

40

400

С

Intensity (arb. unit)

200

30

	Length (nm)	Width (nm)	Aspect ratio
<b>S1</b>	41.0	7.0	5.8
<b>S</b> 2	65.0	57	11.0



View Article Online View Journal | View Issue

# Epitaxial magnetite nanorods with enhanced room temperature magnetic anisotropy†

Sayan Chandra,‡<sup>a</sup> Raja Das,‡<sup>b</sup> Vijaysankar Kalappattil,<sup>b</sup> Tatiana Eggers,<sup>b</sup> Catalin Harnagea,<sup>a</sup> Riad Nechache,<sup>c</sup> Manh-Huong Phan,\*<sup>b</sup> Federico Rosei <sup>b</sup>\*<sup>a</sup> and Hariharan Srikanth <sup>b</sup>\*<sup>b</sup>



The

M(7



Inductive heating properties of Fe<sub>3</sub>O<sub>4</sub> nanorods



# Tunable High Aspect Ratio Iron Oxide Nanorods for Enhanced Hyperthermia

Raja Das,<sup>\*,†</sup> Javier Alonso,<sup>†,‡</sup> Zohreh Nemati Porshokouh,<sup>†</sup> Vijaysankar Kalappattil,<sup>†</sup> David Torres,<sup>†</sup> Manh-Huong Phan,<sup>\*,†</sup> Eneko Garaio,<sup>§</sup> José Ángel García,<sup>‡,||</sup> Jose Luis Sanchez Llamazares,<sup>⊥</sup> and Hariharan Srikanth<sup>\*,†</sup> ions

rystalline.

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Fe<sub>3</sub>O<sub>4</sub>/CoFe<sub>2</sub>O<sub>4</sub> Core/Shell Nanoparticles







SAR in different viscous environments

# SAR Data: 10nm CoFe<sub>2</sub>O<sub>4</sub> @ Fe<sub>3</sub>O<sub>4</sub>



H (Oe)	Hexane	Water	Agar
400	61.990	83.837	47.125
600	121.01	148.447	103.63
800	158.97	246.18	144.51





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### Internalization of Fe<sub>3</sub>O<sub>4</sub> nanorods in the macrophages

#### Cells were kept at 37 °C, 5% CO<sub>2</sub> Total volume: 3.5 ml/well $5 \times 10^5$ cells/ml 30 µg nanorods /ml

#### After 2 h

After 24 h



In vitro experiments were carried out by Rosa Martínez, David Muñoz and Eneko Garaio at the University of Basque Country (UPV/EHU)

### Magneto-mechanical cancer cells destruction





#### U87 Glioma cells with gold-plated vortex Au/NiFe/Au



1 image / 10 min – total 48h

UNIVERSITÉ Grenoble



This video was taken during a toxicity test for the particles, without applied field. Here, the amount of particles is maximum. Viability of the cells is assessed after 48h incubation with the particles. Cells death would be achieved by applying a 20 Hz rotating field.

#### Credits for the video:

Cécile Naud, Thèse de Doctorat (Univ. Grenoble Alpes, 2019)

#### If one paper cited:

"Triggering the apoptosis of targeted human renal cancer cells by the vibration of anisotropic magnetic particles attached to the cell membrane"

Selma Leulmi, Xavier Chauchet, Mélissa Morcrette, Guillermo Ortiz, Hélène Joisten, Philippe Sabon, Thierry Livache, Yanxia Hou, Marie Carrière, Stéphane Lequien, and Bernard Dieny,

Nanoscale 7, 15904 (2015). doi 10.1039/c5nr03518j







### Effect of Hyperthermia



- The incorporation of nanorods into cells slows down their growth.
- Application of AMF generates a 40% decrease in their population after 24 h.
- These results are very promising for magnetic hyperthermia treatment of cancer.





## Magneto-mechanical destruction of cancer cells

# Kim et al. Nature Materials 9, 165 (2010) Argonne group (Ryzkhova, Novosad)

Mansell et al. Scientific Reports 7, 4257 (2017) Vemulkar et al. APL 110, 042402 (2017) Cambridge group (Russell Cowburn)

- Microdiscs with vortex states
- Synthetic antiferromagnetic discs
- Low field/frequency actuation induced torque





### FLASH NEWS!

MagForce AG Receives FDA Investigational Device **Exemption Approval to Conduct a Clinical Trial with** NanoTherm Therapy as Focal Ablation Treatment for **Intermediate Risk Prostate Cancer** February 10, 2018 05:55 AM Eastern Standard Time BERLIN & CARSON CITY, Nev.--(BUSINESS WIRE)--MagForce AG (Frankfurt, Scale, XETRA: MF6, ISIN: DE000A0HGQF5), a leading medical device company in the field of nanomedicine focused on oncology, together with its subsidiary MagForce USA, Inc., announces that it has received U.S. Food and Drug Administration (FDA) Investigational Device Exemption (IDE) approval to conduct a clinical trial with NanoTherm therapy as focal ablation treatment for intermediate risk prostate cancer.





#### OPEN ACCESS

J. Phys. D: Appl. Phys. 50 (2017) 363001 (33pp)

Journal of Physics D: Applied Physics

https://doi.org/10.1088/1361-6463/aa81a1

#### **Topical Review**

### The 2017 Magnetism Roadmap

D Sander<sup>1</sup>, S O Valenzuela<sup>2,3</sup>, D Makarov<sup>4</sup>, C H Marrows<sup>5</sup>, E E Fullerton<sup>6</sup>, P Fischer<sup>7,8</sup>, J McCord<sup>9</sup>, P Vavassori<sup>10,11</sup>, S Mangin<sup>12</sup>, P Pirro<sup>13</sup>, B Hillebrands<sup>13</sup>, A D Kent<sup>14</sup>, T Jungwirth<sup>15,16</sup>, O Gutfleisch<sup>17</sup>, C G Kim<sup>18</sup> and A Berger<sup>10</sup>

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# Summary

Functionalized magnetic nanoparticles with variable size and shapes for nanomedicine applications The importance of anisotropy and its influence on functional response in hyperthermia and tunable RF device applications



# Nano size that is....and shapes, surfaces and interfaces





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Some sample fabrication and characterization facilities at USF Functional Materials Laboratory









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Advanced DC, AC, RF magnetization, transport and thermal measurements @USF Functional Materials Laboratory





# The USF FML group and IEEE Magnetics Society thanks you for your attention...





FML group seen here doing what we love the most...discussing research in bars and coffee shops...

Yep...we put the 'fun' in dysfunctional





# Schemes for functional material structures for tunable EM applications (Magnetoelectric, multiferroic, meta-materials...)



systems can range from ceramics to polymers





Fig. 2. Influence of the particle size on the microwave absorption: (1) 5  $\mu$ m, (2) 65 nm.







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'Suck-cessful' synthesis of nano popsicles





IOP PUBLISHING

Nanotechnology 20 (2009) 485604 (7pp)

doi:10.1088/0957-4484/20/48/485604

#### **Carbon nanostraws: nanotubes filled with** superparamagnetic nanoparticles

Susmita Pal, Sayan Chandra, Manh-Huong Phan, Pritish Mukherjee and Hariharan Srikanth<sup>1</sup>

Integrated Functional Materials Group, Department of Physics, University of South Florida, Tampa, FL 33620, USA





## **Two-Step Anodization Process**

Ester Palmero and Manuel Vazquez (ICM-CSIC, Madrid)







XRD and TEM for NFO and NFO-Filled CNTs from custom templates



~80 nm diameter mulit-walled CNTs



7 nm NiFe<sub>2</sub>O<sub>4</sub>–Filled CNTs









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### The aggravation of agglomeration....





Controlled dispersion and assembly of nanoparticles within a polymer matrix is a challenge in materials processing and manufacturing

2.35 2. 2.25 2.2 T 0. 0. 0.5

Soft Materials Structure and Dynamics, ed J. Dutcher and A. Marangoni <u>(Marcel Decker, New York NY, 2005)</u>

- Clustering is a common problem in polymer composites
- Molecular dynamics simulations predict interaction conditions favoring uniform dispersion or formation of larger clusters Can we control this process?

A. Heilmann, Polymer Films with Embedded Metal Nanoparticles, Springer, New York 2003.

T. Desai, P. Keblinski, S. K. Kumar, J. Chem. Phys. 2005, 122, 134 910.





#### Yes! Countering steric forces with surface charges on nanoparticles

### Cross-sectional TEM of Bilayer







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#### ZFC and FC curves of Fe<sub>3</sub>O<sub>4</sub> nanoparticles



#### ZFC and FC curves of particles in PMMA





# First demonstration of superparamagnetic polymer nanocomposite films

Superparamagnetic Polymer Nanocomposites with Uniform Fe<sub>3</sub>O<sub>4</sub> Nanoparticle Dispersions\*\*

By James Gass, Pankaj Poddar, James Almand, Sanyadanam Srinath, and Hariharan Srikanth\*

© 2006 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim

Adv. Funct. Mater. 2006, 16, 71–75





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## **Microwave Tunability**

In collaboration with microwave gurus Jing Wang and Tom Weller (EE, USF)



#### **Resonator in Electromagnet**





#### **Cavity Resonator**





### **Q-factor, Power loss and Resonance Frequency**



Morales, C., et al., Tunable Magneto-Dielectric Polymer Nanocomposites for Microwave Applications. leee Transactions on Microwave Theory and Techniques, 2011. **59**(2): p. 302-310.





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### Microstrip patch antenna fabrication and performance











Antenna Design	Resonance Frequency (GHz)	Bandwidth (MHz)	Maximum Gain (dBi)	Efficiency	Area (mm²)	Miniaturization % /Factor
PDMS	3.931	185 (4.7%)	5.095	50.74%	594.05 (27.25x21.8)	
PDMS- Fe <sub>3</sub> O <sub>4</sub> 80% PNC	3.298	245 (7.45%)	2.12	31.28%	312.05 (19.75x15.8)	» 57% / 2.3
PDMS- Fe <sub>3</sub> O <sub>4</sub> 50% PNC	3.986	244 (6.12%)	4.063	44.10%	312.05 (19.75x15.8)	» 47.5% / 1.9
PDMS- Fe <sub>3</sub> O <sub>4</sub> 30% PNC	4.585	230 (5.02%)	5.085	40.49%	312.05 (19.75x15.8)	» 39.5 / 1.65

