







# Magnetic sensors: from ultrathin film growth to sensor integration in unexpected systems

### Susana Cardoso de Freitas

Group leader Spintronics & Magnetic Sensors

INESC – Microsystems and Nanotechnologies Lisbon, Portugal www.inesc-mn.pt



# Outline

Magnetoresistive sensors

- thin film materials
- thermal stability
- noise, SNR => detectivity for pT
- 3D detection on chip

Applications



# Magnetic sensors





#### WHY MAGNETORESISTIVE SENSORS ARE CANDIDATES FOR MANY APPLICATIONS ?

**INESC MN** Microsistemas & Nanotecnologias





### Magnetic tunnel junction - TMR

**INESC MN** 

Microsistemas & Nanotecnologias

Accurate control of the thin film thickness - impact on TMR - impact on R





**INESC MN** 

**Microsistemas &** 

**Nanotecnologias** 

1 Å => 10x R



**Microsistemas & Nanotecnologias** 



Film thickness: Controlled at the atomic scale 1 Å = 0.1 nm

### Multilevel device patterning





MgO target with Ar plasma



Microsistemas & Nanotecnologias



Wafer microfabrication in a Clean Room





# Challenges II

Low noise, high signal





### How to distinguish signal from noise ?









P.P.Freitas, R.Ferreira and S.Cardoso "Spintronic Sensors", Proceedings of the IEEE, 104, 1894 (2016)

E. Paz · <u>S. Serrano-Guisan</u> · <u>R. Ferreira</u> · <u>P. P. Freitas</u> – J. of Applied Physics; 115. 2014

### **Noise Spectrum of Magnetoresistive Sensors** Main noise contributions



Field detectivity (D)

INESC MN

**Microsistemas &** Nanotecnologias





Strategies to improve the minimum detectable field

### **Field modulation for high frequency**

Increase MR Increase V Reduce linear range ΔH Reduce Hooge value Sensors, *18*(3), 790; (2018) Micromachines, *7*(5), 88 (2016) IEEE Trans. Magn. 48 (11), pp. 4115 (2012) Journal of SPIN, Vol.1 (1), pp 71-91 (2011) J.Appl.Phys. 103, 07E924 (2008) Appl. Phys. Lett. 95, 023502 (2009)

### magnetic MEMS

Modulate dc magnetic field at high frequency using MEMS resonators with incorporated magnetic flux guides

#### goal

shift the sensor operating frequency to the kHz region where the noise can be 2 orders of magnitude lower then dc





Appl. Phys. Lett. 95, 023502 (2009) J.Appl.Phys. 103, 07E924 (2008)

geometry

#### INESC MN Microsistemas &

**Nanotecnologias** 



#### Strategies to improve the minimum detectable field

### Field modulation for high frequency

#### **Increase MR**

Increase A Reduce linear range ∆H Reduce Hooge value Nat Mater, 2004, 3:862–867 J PhysD-Appl Phys, 2007, 40: R337 J. Physics: Cond.Matter, 19 (2007) 165221 Ann Rev Mater Res, 2009, 39: 277–296 J Appl Phys, 2007, 101: 09B501 J. Appl. Phys, 99, 08A907 2006



*"Magnetoresistive Sensor Development Roadmap (Non-Recording Applications)" IEEE Trans.Magn. (2019)* 

## 200mm backend GMR / TMR technology

INESC MN

Microsistemas & Nanotecnologias







#### Strategies to improve the minimum detectable field

### Field modulation for high frequency

Increase TMR

### **Increase V** (area or thickness)

Reduce linear range ∆H Reduce Hooge value R.Chaves, et.al , Appl. Phys.Lett, 91, 102504 (2017) E. Paz et.al – J. Applied Physics; 115. 2014



Large Series 1102 TMR elements with  $A=100x100 \ \mu m^2$  each. **INESC MN** 

Microsistemas & Nanotecnologias

1 mm x 1 mm





E. Paz · <u>S. Serrano-Guisan</u> · <u>R. Ferreira</u> · <u>P. P. Freitas</u> - Journal of Applied Physics; 115. 2014

### Saving space: GMR sensors packed



AIP Advances 8(5):056644 (2018) Scientific Reports , 11, 215 (2021)



Microsistemas & Nanotecnologias

Packed arrays of sensors





AIP Advances 8(5):056644 (2018) Scientific Reports, 11, 215 (2021) [1] E. Paz et al., J. Appl. Phys., vol. 115,2014. [2] tdk.com, "TDK biomagnetic sensor", 2019 [3] S. H. Liou et al., Proc. IEEE Sensors, 2009 [1] P. Besse et al Appl. Phys. Lett. 80, 4199 (2002) [2] P. Maletinsky et al Nature Nanotechnology 7, 320-324 (2012) [3] F. Montaigne et al Sensors and Actuators A: Physical 81, 324-327 (2000) [4] L. Caruso et al Neuron 95, 1283-1291 (2017) [5] R. Jahns et al Sensors and Actuators A: Physical 183, 16-21 (2012) [6] F. Barbieri et al Scientific Reports 6, 39330 (2016) [7] J. Barry et al PNAS 113, 14133-14138 (2016) [8] Bartington Instruments, Mag-03 Three-axis [9] S. Yabukami et al JMMM 290, 1318-1321 (2005) [10] T. Sander et al Biomedical Optics Express 3, 981-990 (2012) [11] M. Pannetier et al Science 304, 1648-1650 (2004) [12] J. Gallop Supercond. Sci. Technol. 16, 1575 (2003) [13] I. Kominis et al Nature 422, 596-599 (2003) E. Paz et al., J. Appl. Phys., vol. 115,2014. [14] [15] tdk.com, "TDK biomagnetic sensor", 2019 [16] S. H. Liou et al., Proc. IEEE Sensors, 2009



Microsistemas & Nanotecnologias

# Applications

- Neurosciences
- Robotics
- Pattern readout
- Biochips







If no time: move to end



Microsistemas & Nanotecnologias

# Applications

# Neuronal probes with MR sensors



**Microsistemas &** Individual **Nanotecnologias** Cell assembly pyramidal cells Estimation of the magnetic field: L1 MagnetoEncephaloGraphy: • SQUID - signal distance = 3 cm current Field = 1 fT(Field decay :  $1/(r^2)$ flow Magnetrodes: ٠ 2/3 MR - signal distance =  $10-100 \mu m$ Field  $\approx 100 \text{ pT} - 1 \text{nT}$ L4 Estimated fields ~pT - nT 5 L5 100 µm 100 µm Helmchen and Denk. 2005. **FET-EU project** 

Methods 2 : 932-940.

Magnetrodes

AGNETRODES











SEM image: sharp tip (in-vivo)



SEM image: flat tip (in-vitro)

### When penetrating the tissues:





Valadeiro et al IEEE Trans Mag 51 (2015) 4401104



### In Vivo validation of MR sensor probes





L.Caruso, S.Cardoso, P.P Freitas, P.Fries, M. Lecoeur, et.al

*"In vivo magnetic recording of neuronal activity", Neuron,* 95, 1–9 (2017)



**IEEE Trans Magn**. vol. 51 (11) 4401104 (2015) J.Gaspar et.al, IEEE Trans Magn. Vol.53 (4), 5300204 (2017

### Applications in robotics

**INESC MN** Microsistemas & Nanotecnologias



If no time:move to



Magnetic cilia bending induces magnetic profile variation

MR sensor transduces variation into an electrical signal











### Geometry A







**INESC MN** 

Surface roughness ( $\mu$ m)

P.Ribeiro et.al., IROS 2020 : International Conference on Intelligent Robots and Systems 2020





# Proof of concept Fruit quality classifier

#### **Braeburn apples**

- 12 ripe fruits
- 12 senescent fruits

#### Sabrina strawberries

- 12 ripe fruits
- 12 senescent fruits

#### **Data acquisition**

- Data rate: 1 kSPS
- Scan speed: 1 mm/s
- 10 consecutive scans in each area
- 2 areas per fruit



### **FRUIT QUALITY SENSING - RESULTS**







Microsistemas e Nanotecnologias

NESC



#### 3 features were extracted from the signal

	FEATURE	WHAT IS MEASURED	PHYSICAL CHARACTERISTIC
	Stiffness (E)	Sensor signal with achieved contact	Fruit hardness
*	Waviness (S)	Std. deviation of 100 point moving average	Deformation over fruit surface
•	Roughness (R)	Std. deviation of high- pass filtered (f > 150 Hz) signal	Fruit surface texture
	Fruit can be cla into two classes	ssified	Ripe Senescent

**FRUIT QUALITY SENSING - RESULTS** 









Figure 6.4: Computed *E* parameter histogram from all scans of apples scanned using a geometry A ciliary sensor.

NESC

Microsistemas e Nanotecnologias



#### Advanced Robotics @ Queen Mary ARQ **TÉCNICO** LISBOA NESC MI **FRUIT QUALITY SENSING - RESULTS** Institute for Systems and Robotics IJî Microsistemas e Nanotecnologias 1 Apple Strawberry Conf. A True positive True positive True negative True negative Accuracy Accuracy 11/12 12/120.96 7/12 10/12 0.71 h = 3 mmNaïve Bayes Naïve Bayes $\phi = 400 \,\mu m$ Random Forest 10/12 12/120.92 Random Forest 8/12 11/120.79

m			True positive	True negative	Accuracy	-	True positive	True negative	Accuracy
	h = 1.6  mm	Naïve Bayes	10/12	11/12	0.88	Naïve Bayes	10/12	7/12	0.71
uc	$\phi = 320 \ \mu m$	Random Forest	10/12	11/12	0.88	Random Forest	10/12	10/12	0.83

Conf. C

		8	True positive	True negative	Accuracy		True positive	True negative	Accuracy
	h = 3 mm	Naïve Bayes	9/12	8/12	0.71	Naïve Bayes	10/12	10/12	0.83
1112	φ = 400 μm	Random Forest	9/12	11/12	0.83	Random Forest	10/12	10/12	0.83
				2					$\searrow$

### "From farm to fork"

**INESC MN** Microsistemas & Nanotecnologias





### Magnetic Biosensors and biomedical interfaces





Needle sensors Flexible MR Sensors 40 μm

gold pad

Spin Valve sensors







If no time: move to

Microsistemas & Nanotecnologias

**INESC MN** 

# Magnetic trap



# Microfabrication





Silvério et.al., Micromachines 10(9):607 (2019);

Silverio et. Al., IEEE Trans Magn. 53 (11) 5100806 (2017)

- I. PECVD 500 nm SiO<sub>2</sub>: 6" wafer
- 2. magnetron sputtering 15 nm TiW(N)/600 nm AlSiCu/15 nm TiW(N) : bottom electrode (M1 layer)
- 3. optical lithography
- 4. etching
  - This MI film also connects the center of the coils to the contact pad
- 5. RF sputtering SiO<sub>2</sub> 500 nm /Al<sub>2</sub>O<sub>3</sub> 25 nm
  - passivation layer to insulate the coil from the bottom electrode
- 6. optical lithography
- 7. electroplating 40  $\mu$ m of Cu (M2 layer)
- 8. spin coating 40 µm polyimide
  - passivation
- 9. sputtering I μm AlSiCu
  - ensures an optimum optical contrast for the detection of the fluorescence in the MNP
- 10. sputtering 2 µm SiO<sub>2</sub>
  - guarantees the planar surface required for the magnetic trapping and in the future, to promote probe linking
- II. RIE to open 250 μm wide vias for contact pads

# **Magnetic Field Mapping**



Magnetic Tunnel Junction (MTJ), sensitivity 0.04 V.mT<sup>-1</sup>, dimension 91  $\times$  14.5  $\mu m^2$ 

The vertical magnetic field over the **central coil** is **opposite** to the vertical field in the **4 outer coils** 

• favorable for particle trapping and cell concentration

The field generated by 100 mA was measured to range between -70 to +90  $\mu$ T

1000 mA actuation + PM field

- larger magnetic fields: -1500 to +3000 μT
- sufficient to generate a magnetic force to deflect the MNP trajectories and trap them

# Thermal response

• Joule heating  $\rightarrow$  flow cooling



Silvério et.al., Micromachines 10(9):607 (2019); Silverio et. Al., IEEE Trans Magn. 53 (11) 5100806 (2017)



# Thermal response

#### pulsed current: 500 mA 8 s ON / 15 s OFF





Silvério et.al., Micromachines 10(9):607 (2019); Silverio et. Al., IEEE Trans Magn. 53 (11) 5100806 (2017)

Tombelli, et.al., Analytical and Bioanalytical Chemistry 414 (10), 3243 (2022)

# **MNP** Trapping and concentration





direction of the current in the coils

**I 6 pulses @ \_750 mA** I s ON + 2 s OFF + I s ON + ...



$$T_{surf,max} = 3 \circ C$$







MNP concentration 0.058 mg.mL<sup>-1</sup>

Silvério et.al., Micromachines 10(9):607 (2019);

Silverio et. Al., IEEE Trans Magn. 53 (11) 5100806 (2017)

Tombelli, et.al., Analytical and Bioanalytical Chemistry 414 (10), 3243 (2022)





# **Magnetic biosensors**









# Challenges – quantification

- **100nm magnetic particles** coated with protein A;
- Rabbit α-goat Ab → More affinity to protein A;
- Goat α-E.coli Ab → Already tested by IF;
- Special SEM processing without organic solvents;



Adapted from Fernandes et.al. (2014)





UB010 15 0kV 7 0mm x12 0kV0 600 (FCUL)





#### **INESC MN** Microsistemas & Nanotecnologias

ELSEVIER

Contents lists available at ScienceDirect

#### **Biosensors and Bioelectronics: X**

journal homepage: www.journals.elsevier.com/biosensors-and-bioelectronics-x

### INESC MN

Microsistemas & Nanotecnologias

If no time: move to

### On-site magnetic screening tool for rapid detection of hospital bacterial infections: Clinical study with *Klebsiella pneumoniae* cells

Ana R. Soares <sup>a,b,\*</sup>, R. Afonso <sup>b,c</sup>, V.C. Martins <sup>a</sup>, C. Palos <sup>d</sup>, P. Pereira <sup>d</sup>, Diogo M. Caetano <sup>b,c</sup>, Davide Carta <sup>a,b</sup>, S. Cardoso <sup>a,b</sup>

<sup>a</sup> Instituto de Engenharia de Sistemas E Computadores – Microsistemas e Nanotecnologias (INESC MN), Rua Alves Redol 9, 1000-029, Lisbon, Portugal
<sup>b</sup> Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais 1, 1049-001, Lisbon, Portugal

<sup>c</sup> Instituto de Engenharia de Sistemas E Computadores - Investigação e Desenvolvimento, Rua Alves Redol 9, 1000-029, Lisbon, Portugal

<sup>d</sup> HBA – Hospital Beatriz Ângelo, Av. Carlos Teixeira 3, 2674-514, Loures, Portugal







Microsistemas & Nanotecnologias

# Applications in scanners

If no time: move to





### Swipe reader: printed barcode



Regular laserjet toners contain ferromagnetic (Fe<sub>3</sub>O<sub>4</sub>/Fe<sub>2</sub>O<sub>3</sub>) nanoparticles



Maglb INESC MN Microsistemas & Nanotecnologias

### Alignment

Disc magnet provides a limited region where B < 1 mT (±170  $\mu$ m)  $\rightarrow$  requires good accuracy from the alignment procedure



### Swipe reader: assembly





#### **Purpose:**

- Hold reader components
- Protect sensor chip
- Limit sensor tilt
- Minimise sensor-barcode distance





MaglD



S.Abrunhosa. S.Cardoso, et.al IEEE Trans. Magn. 58 (8), 4002304 (2022) **INESC MN** 

Microsistemas & Nanotecnologias

### Handheld magnetic code reader

September 2020

4 mm









July 2020

February 2021





#### February 2020



October 2020



December 2020

May 2020



13/20

### Handheld magnetic code reader









NEXT CHALLENGE: Decode QR codes

### **Acknowledgments**

To my team at INESC-MN (past and present)





**INESC MN Microsistemas & Nanotecnologias** 

#### Contact for PhD, Post-Doc or internships: scardoso@inesc-mn.pt



a Tecnologia







IAPMEI







Mag-ID H2020-EIC-FTI-870017 MASMA H2020-EIC-SMEInst n. 858934 MagScopy4IHC LISBOA-01-0145-FEDER-031200