



**LOCAL PROBING**



**OF FERROIC AND**



**MULTIFERROIC COMPOUNDS**



**INTC MEETING, CERN, CH, 2017**

# The collaboration

A.M.L. Lopes, J. Schell V.S. Amaral, C.O. Amorim, J.P. Araújo, A. Baghizadeh, M. Baptista, Hans-Werner Becker, M. Escobar Castillo, J.G. Correia, A. Fenta, J.N. Gonçalves, H. Haas, M. Kachlik, S. Kamba, A. L. Khoklin, A.A. Lourenço, D. C. Lupascu, K. Maca, G. Oliveira, S. Picozzi, A.L. Pires, E.C. Queirós, P. Rocha-Rodrigues, V. V. Shvarzman, M.R. Silva, A. Stroppa, P.B. Tavares, J.M. Vieira, Yulian Vysochanskii



**IFIMUP, Science Faculty**, Univ. Porto, Porto, Portugal



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**Centre for Nuclear Sciences and Technologies**, IST, Univ. de Lisboa, Portugal



**Ruhr-Universität Bochum**, RUBION, Bochum, Germany



**CEITEC , Brno Univ of Technology**, Purkyňova, Czech Republic



**Institute of Physics, Czech Academy of Sciences**, Prague, Czech Republic



**CNR-SPIN L'Aquila**, Italy



**Department Chemistry**, Vila Real, Portugal



**Institute of Solid State Physics and Chemistry**, Uzhgorod Univ, Ukraine

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**Centre for Nuclear Sciences and Technologies, IST, Univ. de Lisboa, Portugal**



Juliana Schell



Guilherme Correia



Vitor Amaral



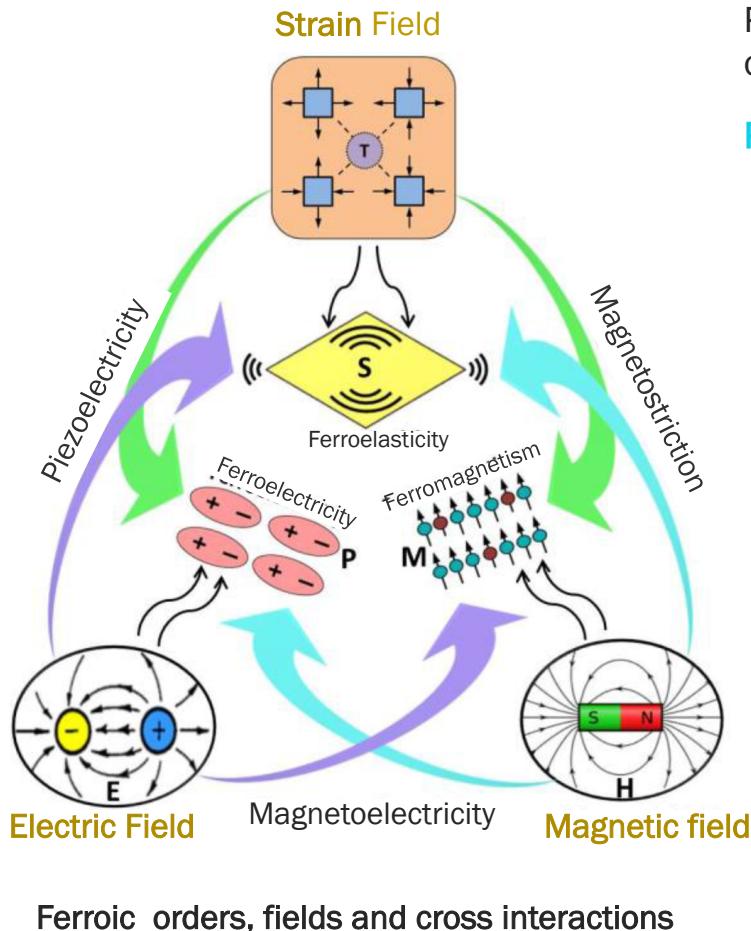
Armandina Lopes



# Ferroic & Multiferroic systems

# Multiferroic system

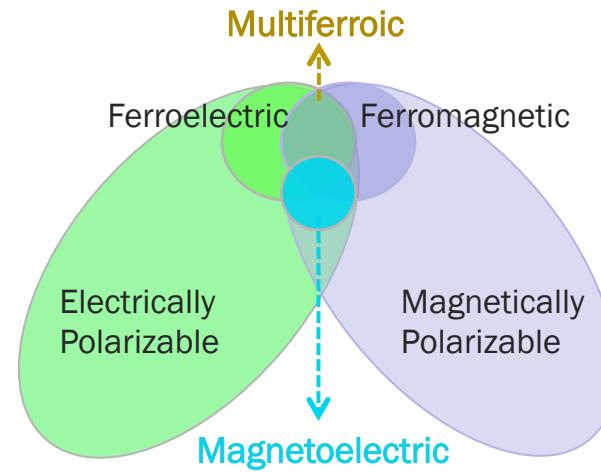
Systems exhibiting simultaneously ferroelastic, ferroelectric and ferromagnetic orders



Possibility to manipulate the magnetic degrees of freedom electrically or vice-versa.

**Path to faster, smaller, and more energy-efficient spintronic devices**

(e.g. memory elements, high-frequency magnetic devices, and systems for data-storage technologies)



But only a few multiferroic materials exists as a concomitant breaking of space-and time-inversion symmetries is necessary.

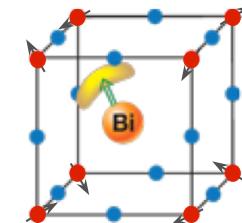
# Types of Multiferroics

## Proper Ferroelectrics:

$\text{BiFeO}_3$  and  $\text{BiMnO}_3$  - Ordering of the  $6s^2$  lone pairs of  $\text{Bi}^{3+}$   
(TFE  $\sim 1100\text{K}$ , TN=643 K, P  $\sim 90\mu\text{C}/\text{cm}^2$ )

Ferroelectricity and magnetism have different sources.

High FE order temperature and polarization / Low ME coupling

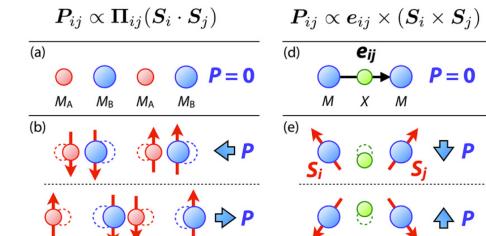


## Improper Ferroelectrics:

$\text{TbMnO}_3$  and  $\text{LuMnO}_3$  - Mn magnetic order  
(TFE =TN $\sim 28\text{ K}$ , P  $\sim 0.06\mu\text{C}/\text{cm}^2$ )

Ferroelectricity and magnetism have the same source.

Low FM and FE order temperature and polarization/ High ME coupling

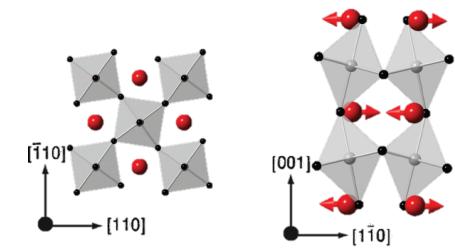


## Hybrid improper Ferroelectrics

$\text{Ca}_3\text{Ti}_2\text{O}_7$  and  $\text{Ca}_3\text{Mn}_2\text{O}_7$   
(TFE  $\sim 1100^\circ\text{K}$  and 280 K, P  $\sim 1\mu\text{C}/\text{cm}^2$ )

Ferroelectricity is driven by the coupling of two non polar modes.

High FE order temperature/High ME coupling





# Why Multiferroic materials

- 1 - In most cases the **Microscopic Mechanisms** controlling the magnetoelectric coupling are unknown
- 2 - The strong coupling among degrees of freedom offers a fertile playground of fundamental physics to be searched
- 3 - In many cases the systems are claimed to be multiferroic but experimental proof are inexistent since:
  - high electric leakage currents might lead to unrepresentative features of the intrinsic material properties
  - properties arising from local structural features that are not well described by a crystallographic approach based on long-range average models
- 4 - The correct symmetries of the ferroelectric phases are sometimes unknown

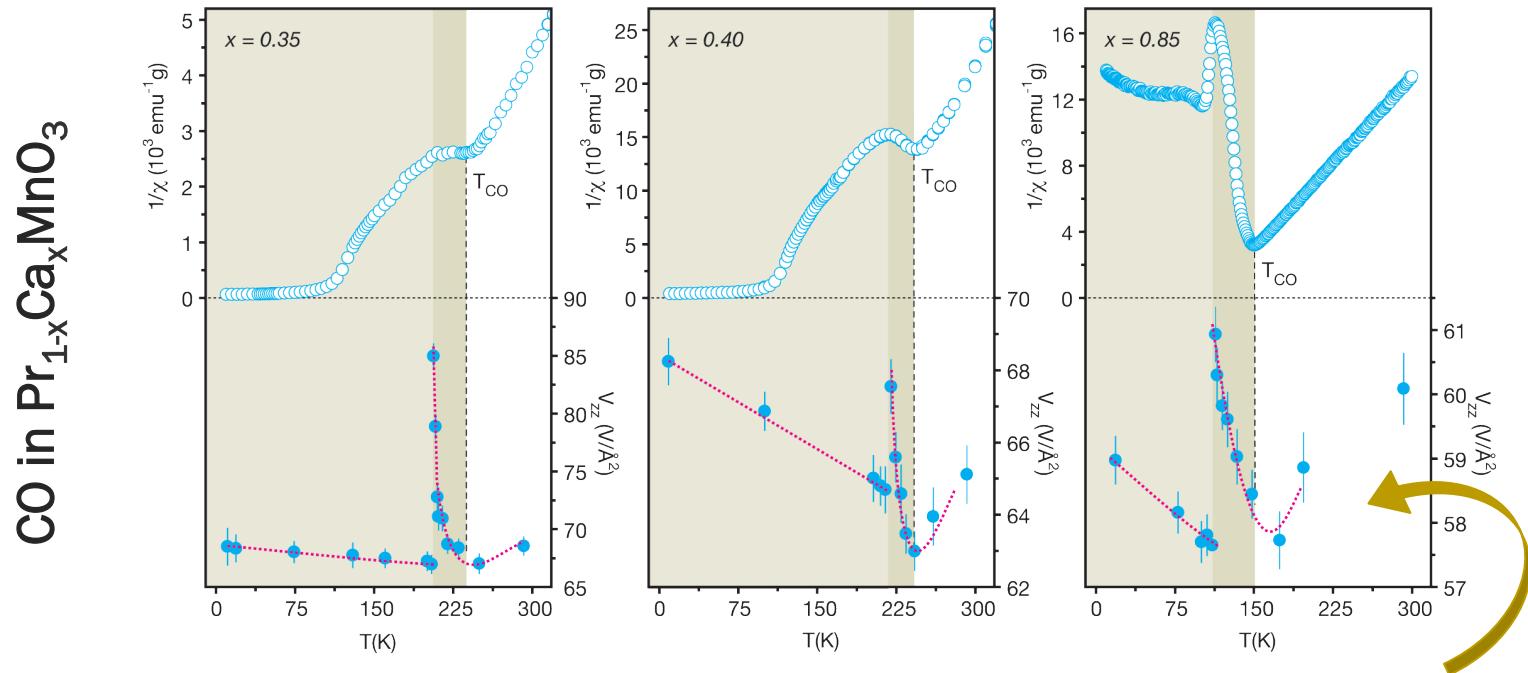
Thus, **complementary local scale information is necessary** to: Provide local and element selective information on the mechanisms that rule structural, charge, and orbital correlations, electronic and magnetic interactions.



# Our previous achievements in multiferroic using PAC

# Charge ordering induced Ferroelectricity

First experimental evidence of CO induced Ferroelectricity



EFG sensitive to atomic vibrations and critical fluctuations.

Expanding  $V_{zz}$  in powers of ionic charge displacements

$$V_{zz} = V_{zz}^0 + \alpha P_s^2 + \beta T \chi_{el}$$

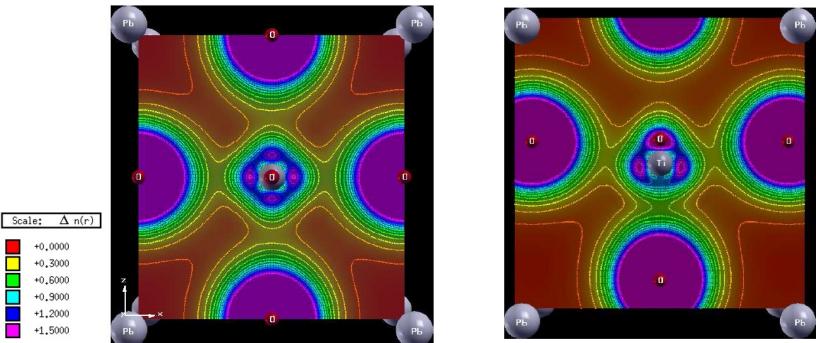
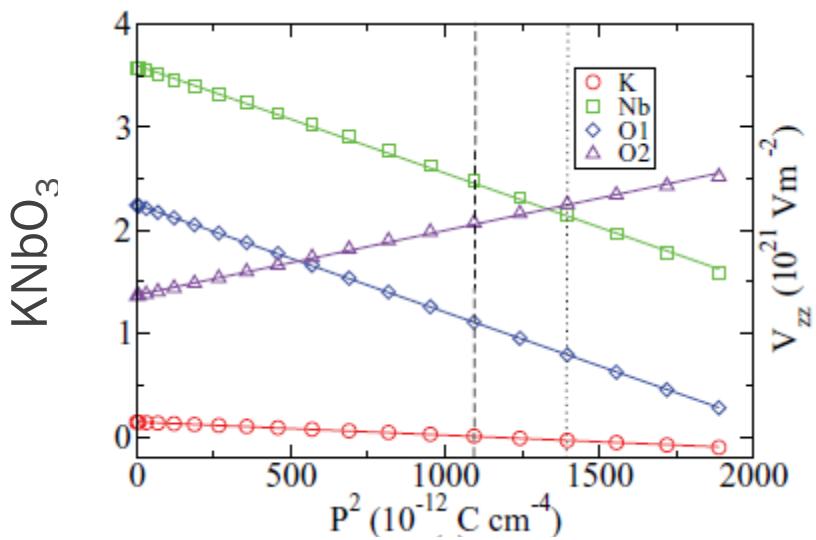
A M L Lopes et al. PRL 100, 155702 (2008)

Each PAC point  $\sim 4$  h  
measurement and  
1 h machine

# ab-initio studies



**ab-initio studies:** EFG and spontaneous electric polarization quadratically correlated in most simple ferroelectrics



Electronic density of  $\text{PbTiO}_3$  in the  $xz$   $\text{TiO}_2$  plane, for the paraelectric and ferroelectric structures.

The sensitivity of the EFG to the polarization and the atomic number of the A site in the perovskite structure is seen.



# Proposed multiferroics to be studied using PAC

# Selected systems



For a consolidated overview we will target:

Proper Ferroelectrics



Vladimir Shvartsman

Improper Ferroelectrics



João Gonçalves

Hybrid Improper Ferroelectrics

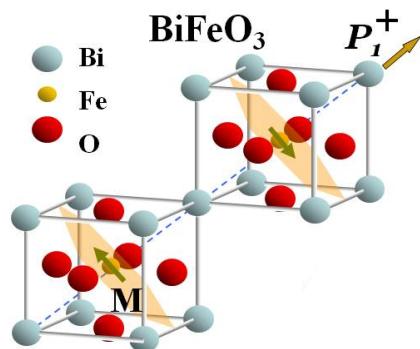


Gonçalo Oliveira

# Proper ferroelectricity: $\text{BiFeO}_3$

## Ferroelectricity and antiferromagnetism at room temperature

$T_N \sim 370^\circ\text{C}$  and  $T_C \sim 830^\circ\text{C}$   
Varying as a function of the particle size



Magnetic properties change considerably when approaching the cycloidal period length

NANO  
LETTERS

Letter  
[pubs.acs.org/NanoLett](https://pubs.acs.org/NanoLett)

### Mössbauer Study of Temperature-Dependent Cycloidal Ordering in $\text{BiFeO}_3$ Nanoparticles

J. Landers,<sup>†</sup> S. Salamon,<sup>\*,†</sup> M. Escobar Castillo,<sup>‡</sup> D. C. Lupascu,<sup>‡</sup> and H. Wende<sup>†</sup>

<sup>†</sup>Faculty of Physics and Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg-Essen, 47048 Duisburg, Germany

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OPEN

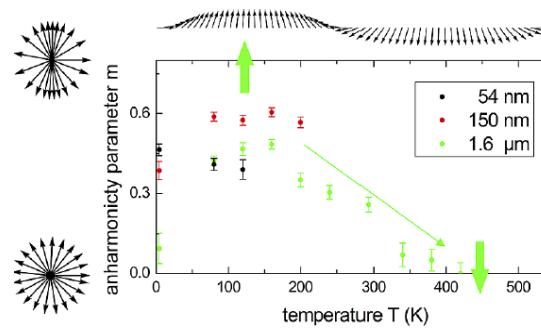
SUBJECT AREAS:  
MAGNETIC PROPERTIES  
AND MATERIALS  
APPLIED PHYSICS  
CHEMICAL PHYSICS  
FERROELECTRICS AND  
MULTIFERROICS

Received  
12 June 2013

### Peculiar magnetism of $\text{BiFeO}_3$ nanoparticles with size approaching the period of the spiral spin structure

Fengzhen Huang<sup>1</sup>, Zhijun Wang<sup>1</sup>, Xiaomei Lu<sup>1</sup>, Junting Zhang<sup>1</sup>, Kangli Min<sup>1</sup>, Weiwei Lin<sup>2</sup>, Ruixia Ti<sup>1</sup>, TingTing Xu<sup>1</sup>, Ju He<sup>1</sup>, Chen Yue<sup>1</sup> & Jinsong Zhu<sup>1</sup>

<sup>1</sup>National Laboratory of Solid State Microstructures, Physics School, Nanjing University, Nanjing 210093, People's Republic of China, <sup>2</sup>Institut d'Électronique Fondamentale, Université Paris-Sud, Orsay 91405, France.



Anharmonicity parameter



Ana Pires

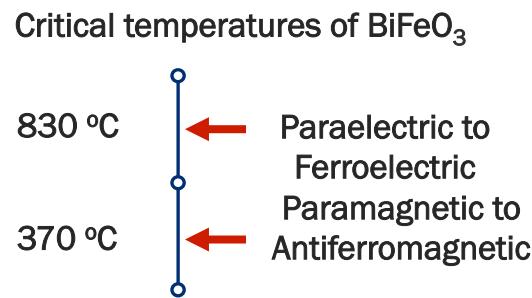
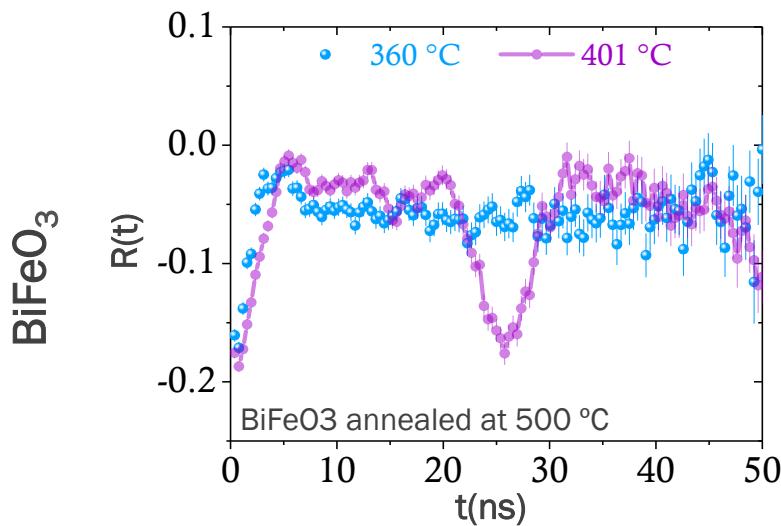


Danilo Olzon

# Proper ferroelectricity: BiFeO<sub>3</sub>

## Local magnetic structure and its evolution with nanoparticles size

PAC measurements above and below the magnetic phase transition at ISOLDE using <sup>181</sup>Ta γ-γ decay from <sup>181</sup>Hf

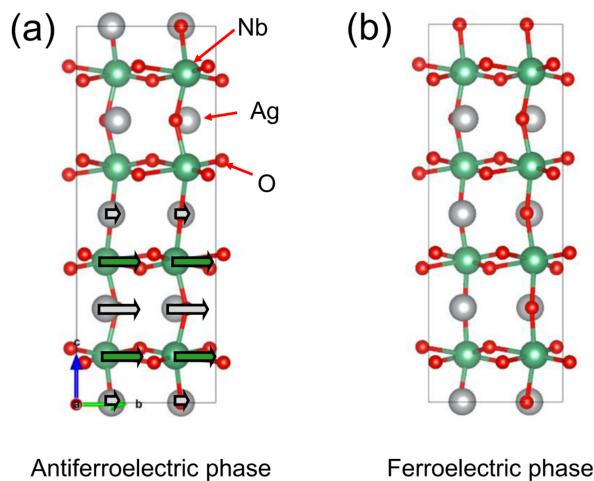


Aim: Bi site probing of local magnetic structure and magneto-electric coupling  
Combination of <sup>204</sup>Bi/Pb, <sup>111</sup>In/Cd, <sup>111m</sup>Cd/Cd and <sup>181</sup>Hf/Ta  
Information source: EFG(T) + B(T)

# Proper ferroelectricity: $\text{AgNbO}_3$

Large polarization at room temperature

Clarification of the anti/ferroelectric phases symmetries



AIP Journal of Applied Physics

HOME BROWSE INFO FOR AUTHORS

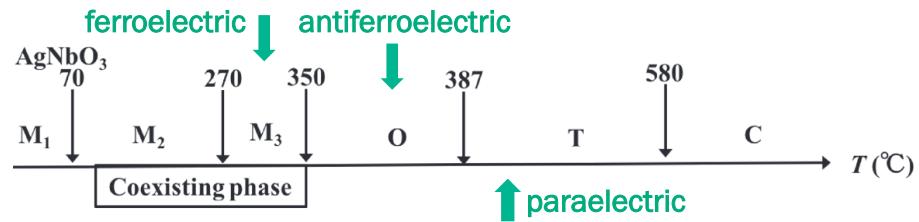
Home > Journal of Applied Physics > Volume 119, Issue 6 > 10.1063/1.4941319

Published Online: February 2016 Accepted: January 2016

## The electric field induced ferroelectric phase transition of $\text{AgNbO}_3$

Hiroki Moriwake<sup>1</sup>, Ayako Konishi<sup>1</sup>, Takafumi Ogawa<sup>1</sup>, Craig A. J. Fisher<sup>1</sup>, Akihide Kuwabara<sup>1</sup>, and Desheng Fu<sup>2</sup>

JAP, 119, 064102 (2016)



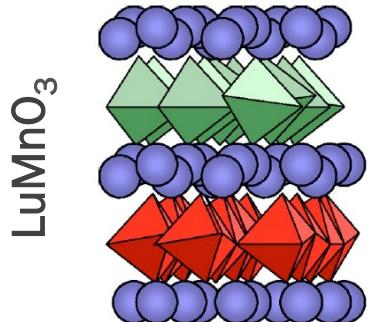
Aim: Ag site probing of structural paraelectric, antiferroelectric and ferroelectric transitions  
 $^{111}\text{Ag}/\text{Cd}$ , crosscheck for electronic relaxation effects upon parent decay  $^{111m}\text{Cd}/\text{Cd}$  and  
 $^{111}\text{In}/\text{Cd}$ . Information source: EFG (T) + B (T)

# Improper ferroelectricity: Lu-Fe-O systems and $\text{LuMnO}_3$

## Ferroelectricity at room temperature

$\text{LuFeO}_3$  -hexagonal multiferroic with large Lu plane rumpling, with net ferromagnetic moment, and orthorrombic non-polar phases

$\text{LuFe}_2\text{O}_4$  - spin and charge frustration in the Fe lattice due to frustration geometry



LETTER

J. A. Mundy et al., Nature 537, 523  
(2016)  
doi:10.1038/nature19343

Atomically engineered ferroic layers yield a room-temperature magnetoelectric multiferroic

Julia A. Mundy<sup>1\*</sup>, Charles M. Brooks<sup>2\*</sup>, Megan E. Holtz<sup>1\*</sup>, Jarrett A. Moyer<sup>3</sup>, Hena Das<sup>1</sup>, Alejandro F. Rébola<sup>1</sup>, John T. Heron<sup>2,4</sup>, James D. Clarkson<sup>5</sup>, Steven M. Disseler<sup>6</sup>, Zhiqi Liu<sup>5</sup>, Alan Farhan<sup>7</sup>, Rainer Held<sup>2</sup>, Robert Howden<sup>1</sup>, Elliot Padgett<sup>1</sup>, Qingyun Mao<sup>1</sup>, Hanjong Park<sup>2</sup>, Rajiv Misra<sup>8</sup>, Lena F. Kourkoutis<sup>1,9</sup>, Elke Arenholz<sup>7</sup>, Andreas Scholl<sup>7</sup>, Julie A. Borchers<sup>6</sup>, William D. Ratcliff<sup>6</sup>, Ramamoorthy Ramesh<sup>5,10,11</sup>, Craig J. Fennie<sup>1</sup>, Peter Schiffer<sup>3</sup>, David A. Muller<sup>1,9</sup> & Darrell G. Schlom<sup>2,9</sup>

RT magnetoelectric multiferroic in  $(\text{LuFeO}_3)_m/(\text{LuFe}_2\text{O}_4)_n$

Giant magneto-elastic coupling results in large atomic displacements as  $f(T)$

Exploration of self-doped  $\text{LuMn}_x\text{O}_{3+\delta}$  with weak ferromagnetic moments.

$\text{Lu}_3\text{Fe}_5\text{O}_{12}$  – magnetodielectric response and  $\text{Lu}_2\text{Fe}_3\text{O}_7$  – 2D antiferromagnet, charge order



Carlos Amorin



Nuno Fortunato

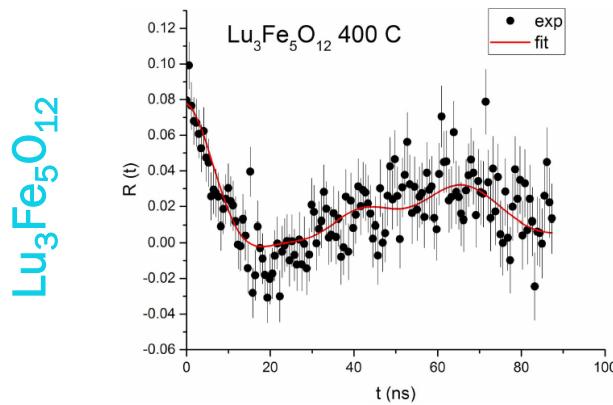
# Improper ferroelectricity

www.rotadaluiz.pt

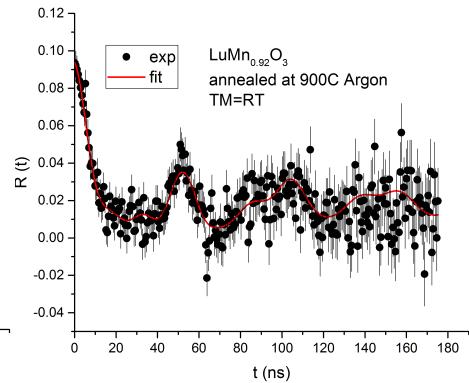
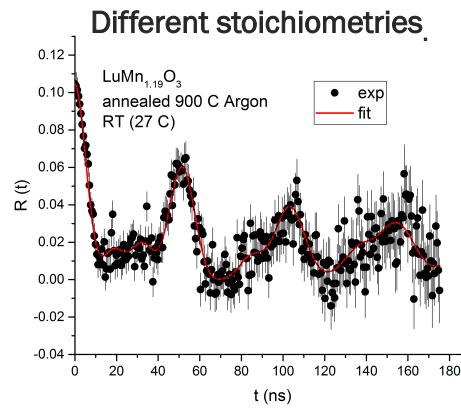
rota da luz

## Magnetoelectric coupling through the hyperfine parameters

PAC measurement with implanted  $^{111m}\text{Cd}$



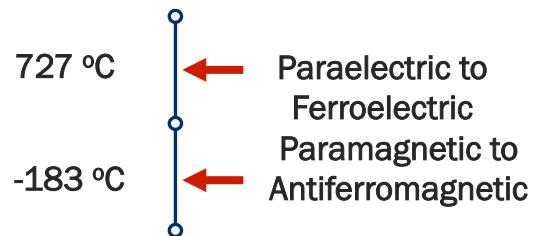
$\text{LuMnO}_3$



One local environment by the preliminary fit, which corresponds to a  $V_{zz}=5.4 \times 10^{-21} \text{ V m}^{-2}$ , and  $\eta=0.4$ .

Aim: Lu site probing of the phase transitions using  $^{172}\text{Lu}$ ,  $^{111m}\text{Cd}$  and  $^{149}\text{Gd}$ . Information source: EFG(T) + B(T)

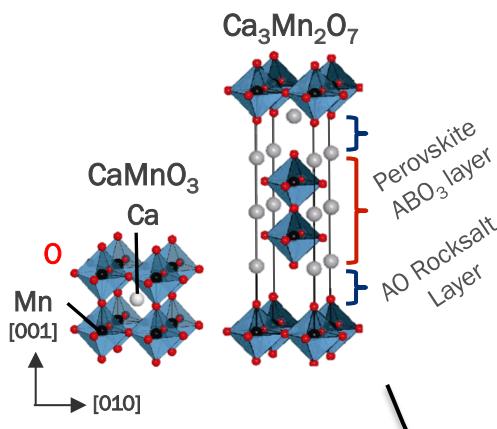
Critical temperatures of  $\text{LuMnO}_3$



# Hybrid improper ferroelectricity

Tri-linear coupling of two non-polar lattice distortion modes

Octahedral rotation/tilting modes with a polar displacement mode



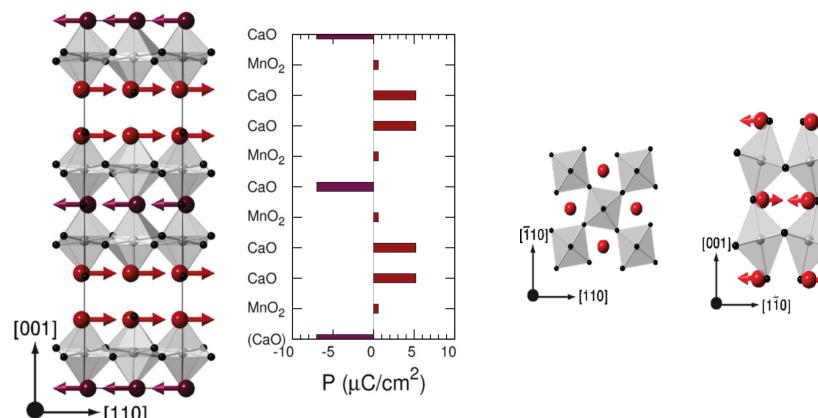
nature  
materials

ARTICLES

PUBLISHED ONLINE: 12 JANUARY 2015 | DOI: 10.1038/NMAT4168

## Experimental demonstration of hybrid improper ferroelectricity and the presence of abundant charged walls in $(Ca,Sr)_3Ti_2O_7$ crystals

Yoon Seok Oh<sup>1,2†</sup>, Xuan Luo<sup>3</sup>, Fei-Ting Huang<sup>1,2</sup>, Yazhong Wang<sup>1,2</sup> and Sang-Wook Cheong<sup>1,2,3\*</sup>



Pedro Rodrigues



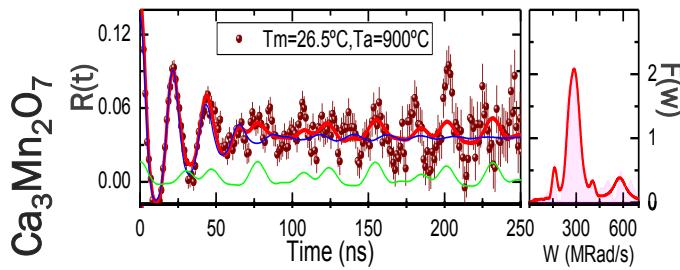
Ricardo Teixeira

Breaking of inversion center at the B-site by the AO layers

# Hybrid improper ferroelectricity

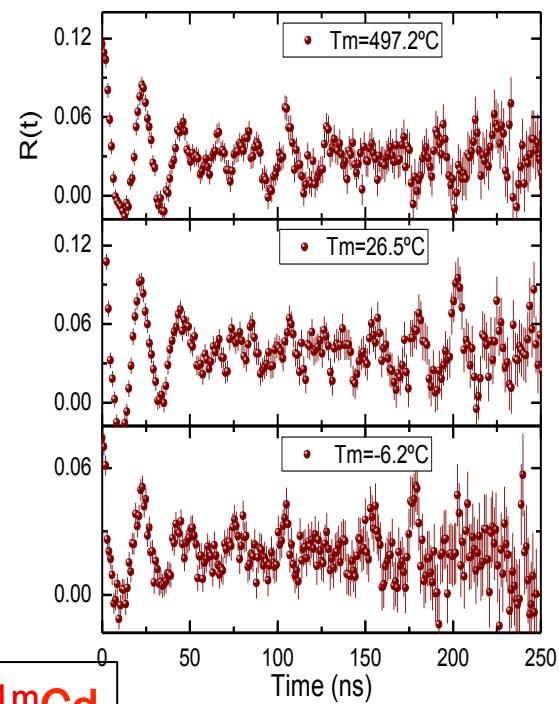
No direct evidence for spontaneous polarization in  $\text{Ca}_3\text{Mn}_2\text{O}_7$   
Symmetry details unknown

PAC measurements at ISOLDE using  $^{111m}\text{Cd}$



Critical temperatures of  $\text{Ca}_3\text{Mn}_2\text{O}_7$

- 386.9°C ← Tetragonal to orthorhombic
- 6.9°C ← Structural & HIF
- 148.2°C ← Paramagnetic to Antiferromagnetic



Aim: Ca site probing of the phase transition using  $^{111m}\text{Cd}$  and  $^{149}\text{Gd}$ . Information source: EFG(T) + B(T)



# Feasibility tests with PAC PROBES

Other systems to be studied by PAC:

## **CuInP<sub>2</sub>S<sub>6</sub>**

In site probing of paraelectric to ferrielectric phase transition with <sup>111m</sup>Cd(48m)/Cd (crosscheck for electronic relaxation effects upon parent decay of <sup>111</sup>In/Cd @ In site)

Information source: EFG(T)

## **ATiO<sub>3</sub>, AZrO<sub>3</sub> A = Ba, Pb**

Sensitive probing of spontaneous polarization variations at A site with <sup>111m</sup>Cd(48m)/Cd and <sup>204m</sup>Pb(67m)/Pb.

Information source: EFG(A, T) + B(A, T)

## **ANiMnO<sub>6</sub>, A = In, Y, Bi, Gd**

Probing A site role on polarization values/switching and short-range magnetic correlations with <sup>111m</sup>Cd(48m)/Cd and <sup>204m</sup>Pb(67m)/Pb (crosscheck for electronic relaxation effects upon parent decay of <sup>111</sup>In/Cd and <sup>204</sup>Bi/Pb @ A site) and <sup>149</sup>Gd(9.3d)/Eu

Information source: EFG(A,T) + B(A,T)

# Beam Time Request

ISOLDE Beam	Approximate intensity (ion/ $\mu$ C of p-beam)	Target	Ion source	SHIFTS 1 + 1 years	Machine days per year
$^{111m}\text{Cd}$ (49m)	$1.\text{10}^8$	Molten Sn	Vadis MK5	$12+12$	4
$^{111}\text{Ag}$ (7.45d)	$10^9$	UC2	RILIS	$2 + 2$	$2/3$
$^{149}\text{Gd}$ (9.3d)	$3.\text{10}^9$	Ta foil	Surface ioniser	$1 + 1$	$1/3$
$^{172}\text{Lu}$ (6.7d)	$2.\text{10}^7$	Ta foil	Surface ioniser	$2 + 2$	$2/3$
$^{204m}\text{Pb}$ (67m)	$5.\text{10}^7$	UC2	RILIS	$6 + 6$	2
$^{204}\text{Bi}$ (11.2h)	$1.\text{10}^7$	UC2	RILIS	$2 + 2$	$2/3$
TOTAL (two years)=				$25 + 25$	



# Outputs

## Deliverables with radioactive nuclear methods (2012-2016)

### IS487 - Study of Local Correlations of Magnetic and Multiferroic Compounds

**2** Ph. D. in Physics concluded

**4** Ph. D. in Physics ongoing

**11** Publications (3 Proceedings)

**11** Oral Communications in international meetings

**13** Posters in international meetings

## Collaboration publications with NON - radioactive nuclear methods

**+ 60** publications in international scientific journals with referee



# Sample production



## Laboratory

Family of samples	Type of samples	CENIDE UDEMAT Essen	CICECO Aveiro	IFIMUP Porto	Uzhgorod Univ.; Vilnius University	Acad. Sciences Czech Republic; Commercial
BiFeO <sub>3</sub>	Pellets	Solid State Reaction				
AgNbO <sub>3</sub> , AgTaO <sub>3</sub>	Pellets S. Crystals	Czochralski				
CuInP <sub>2</sub> S <sub>6</sub>	Pellets S. Crystals				Czochralski	
Lu(Mn/Fe)O <sub>3</sub> , LuFe <sub>2</sub> O <sub>4</sub> , Lu <sub>3</sub> Fe <sub>5</sub> O <sub>12</sub>	Pellets		Solid State Reaction			
ATiO <sub>3</sub> , AZrO <sub>3</sub> A = Ba, Pb	Pellets S. Crystals					Czochralski
Ca <sub>3</sub> (Mn/Ti)2O <sub>7</sub> Ca <sub>2-x</sub> Gdx(Mn/Ti)2O <sub>7</sub>	Pellets			Solid State Reaction		
A NiMnO <sub>6</sub> A = In, Y, Bi, Gd	Pellets			Hydrothermal synthesis		



# Sample characterization

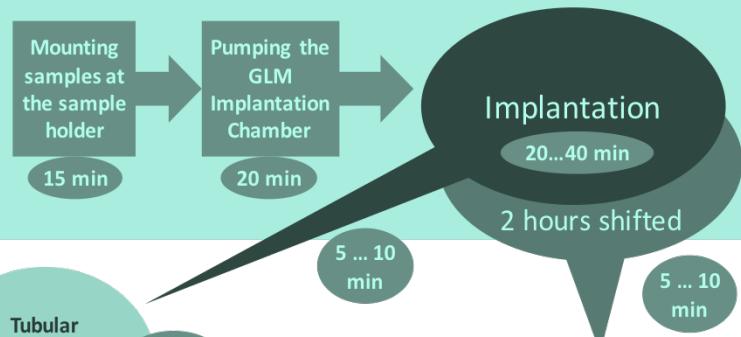
Samples are methodically characterized before and after PAC experiments  
At our home institutes we have at our dispose:

- X-ray powder, high resolution and single crystal diffraction: Aveiro, Essen, Porto
- Scanning Electron Microscopy (SEM) with (EDX): Aveiro, Essen, Porto
- Magneto-electro characterization Magnetic (SQUID, VSM, ac Susceptibility), Dielectric and Electric resistivity measurements (with magnetoresistance) : Aveiro, Essen, Porto
- Scanning Probe Microscope (SPM), Atomic-force microscopy (AFM) and Piezoresponse Force Microscopy (PFM) techniques.
- Transmission Electron Microscopy (HRTEM) with temperature variation: Aveiro and Essen
- Rutherford Back Scattering/Channeling (RBS/C) ion beam analysis characterizing surface composition and defects on implanted regions : Lisboa/Sacavém
- Mossbauer spectroscopy: Essen and Porto

# Practical information

## CPM diagram

170-ISOLDE



508 R-004

Tubular Furnace Annealing  
Two samples

10 ... 20 min

508 R-008

3 hours measurement and CPM restart

TYPE A Sample

10K  
...  
300K  
T1

6Det  
PAC 1

TYPE B Sample

300K  
...  
1173K  
T2

6Det  
PAC 2

300K  
...  
1173K  
T2

6Det  
PAC 4

TYPE A Sample

LN2 or  
300K

6Det  
PAC 3

TYPE C Sample



Images taken From:

Yoshinori Tokura et al, Rep. Prog. Phys. 77, 076501, 2014

N. A. Hill. The Journal of Physical Chemistry B, 104, 6694, 2000

S. Lee et al, Nature 451, 805, 2008

J. Hemberger et al, Nature 434, 364, 2005

S. Seki et al, Physical Review Letters 101, 2008

<http://www.ipem.espci.fr/ocg/>

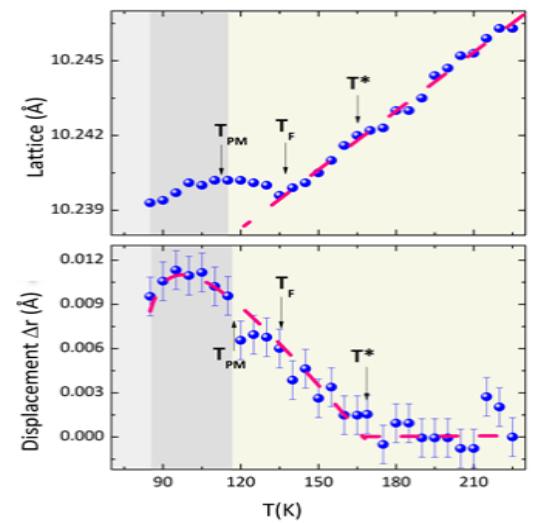
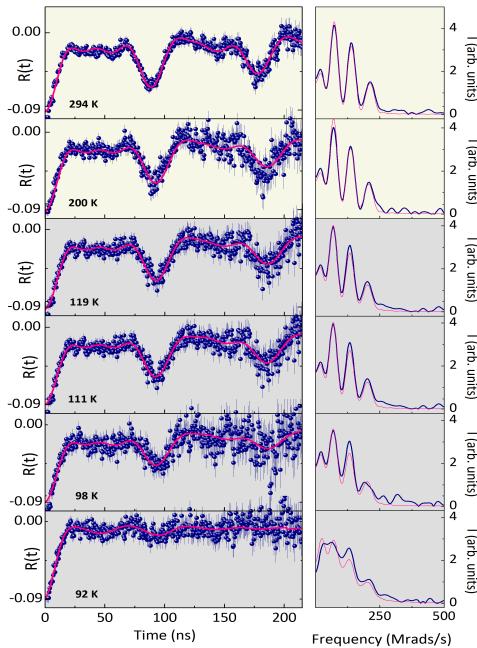
# Practical information



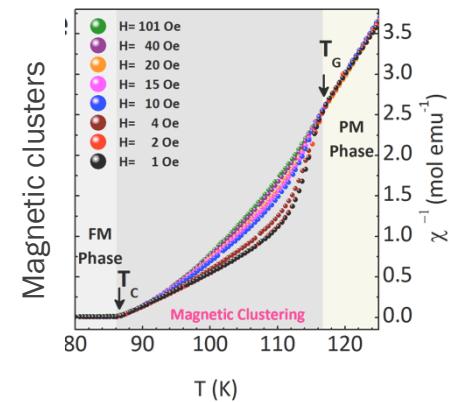
Isotope	Samples prepared at	Experiments at ISOLDE	Atoms per collection, per sample ( $10^{11}$ )	Activity (MBq)	Nº of spectra per collection, per sample	Acq. time per spectrum
$^{111m}\text{Cd}$ (49 m)	ISOLDE	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 85\text{ns}, I=5/2$	0.3	8	1	3h
$\downarrow$						
$^{111}\text{Cd}$ (stable)						
$^{111}\text{In}$ (2.8d)	BONIS RUBION	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 85\text{ns}, I=5/2$	1	0.28	15 - 25	3h $\square$ 12h (*)
$\downarrow$						
$^{111}\text{Cd}$ (stable)						
$^{111}\text{Ag}$ (7.45 d)	ISOLDE	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 85\text{ns}, I=5/2$	3 - 5	0.2 - 0.5	5 - 10	1d $\square$ 1w (*)
$\downarrow$						
$^{111}\text{Cd}$ (stable)						
$^{149}\text{Gd}$ (9.3d)	ISOLDE	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 2450\text{ns},$ $I=11/2$	1.5 - 2.5(***)	0.1 - 0.2	~5	2d $\square$ 1w (*)(**)
$\downarrow$						
$^{149}\text{Eu}$ (93.1d)						
$^{172}\text{Lu}$ (6.7d)	ISOLDE	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 8.33\text{ns}, I=5/2$	2 - 5	0.2 - 0.6	5 - 10	1d $\square$ 1w (*)
$\downarrow$						
$^{172}\text{Yb}$ (stable)						
$^{181}\text{Hf}$ (42d)	BONIS RUBION	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 10.8\text{ns}, I=5/2$	5	0.1	20 - 30	12h - 5d (*)
$\downarrow$						
$^{181}\text{Ta}$ (stable)						
$^{204m}\text{Pb}$ (67m)	ISOLDE	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 265\text{ns}, I=4$	0.15(***)	2.6	1	4h
$\downarrow$						
$^{204}\text{Pb}$ (stable)						
$^{204}\text{Bi}$ (11.2h)	ISOLDE	1 <sup>st</sup> annealing  $\gamma$ - $\gamma$ PAC $T_{1/2} = 265\text{ns}, I=4$	1(***)	1.7	2 - 5	3-6
$\downarrow$						
$^{204}\text{Pb}$ (stable)						

# Magneto-Electric Clusters

$\text{Cr}^{3+}$  dynamic off-centering leading to Magneto-electric Clusters



Cr Local distortion



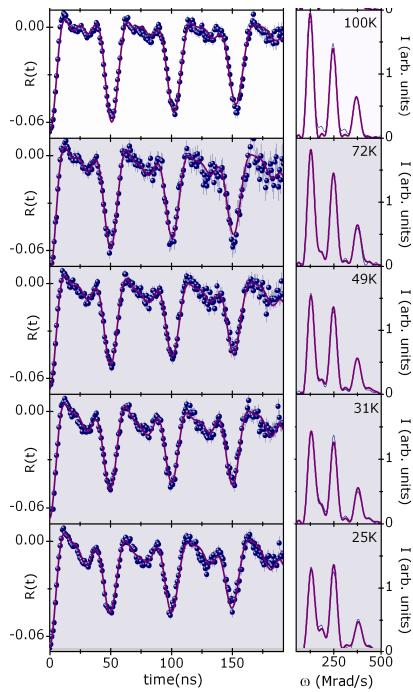
Magnetic clusters

Combined PAC, PDF and M( $T$ ) analysis → dynamic state caused by the presence of simultaneous polar and magnetic nanoclusters

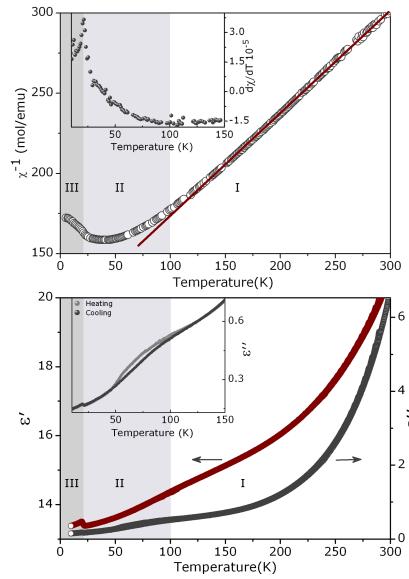
# Triangular spin lattice

## Local distortions in multiferroic $\text{AgCrO}_2$ triangular spin lattice

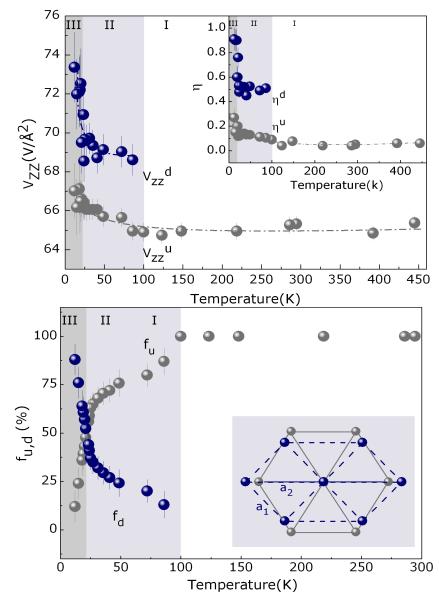
$\text{AgCrO}_2$



Still above TN, part of the system loses the local rhombohedral symmetry



Short-range magnetic correlations



Cr sounding local distortion

Coupling between the elastic and magnetic degrees of freedom provides a channel for magnetic frustration release through a lattice distortion