



NIS colloquium

X-ray induced modifications in materials: applications and challenges, 6-7 April, 2017, Torino

**National Institute
of Materials Physics**

Bucharest-Magurele,
Romania

<http://www.infim.ro>

Materials Research at National Institute of Materials Physics

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Romania: East of Europe, North of the Balkan Peninsula



Located: ~17 Km south from the Bucharest city center and ~150 Km from Dracula Castle



**Dracula
Castle**

Magurele Science Hub

1. 'Horia Hulubei' National Institute for Physics and Nuclear Engineering (includes Extreme Light Infrastructure – Nuclear Physics)
2. National Institute for Laser, Plasma and Radiation Physics (includes Institute of Space Science)
3. National Institute for Earth Physics
4. National Institute for Optoelectronics – INOE 2000

History: Institute of Physics of the Romanian Academy of Science 1949

Labs:

10. Multifunctional Materials and Structures Laboratory

20. Laboratory of Magnetism and Superconductivity

30. Nanoscale Condensed Matter Laboratory

40. Optical Processes in Nanostructured Materials

50. Laboratory of Atomic Structures and Defects in Advanced Materials

60*. *Certified Laboratory for chemical analysis of advanced materials (XPS)*

*** - *it is not an administrative entity***

Fields of interest:

1. Cond matter – phenomena and processes in nano-dimensional, surface and interface systems
2. Synthesis and characterization of nanomaterials and nanostructures
3. Functional materials and structures with technological impact

A: Fundamental studies cond. matter

1. Dimensional effects in nano-objects and cuantic layers
2. Surfaces and interfaces in structured materials
3. Electronic correlations and magnetic interactions
4. Computational modeling and simulation of the dynamics microstructures
5. Interaction between matter and radiation at micro and nano scales

B: Multifunctional nanostructures and materials

- 1 – Materials for energy (generation, transport, conversion, storage)
- 2 – Materials for applications from the high tech industry (materials for high frequency electronics MW, THz, materials for optoelectronics, transparent electronics, materials for non-volatile memories, sensors for automatization and control)
- 3 – Materials for applications in biomedicine and environment protection (biocompatible and biofunctional materials, bio and chemical sensors and foto-catalysts)

Laboratory of Magnetism and Superconductivity

Processing:

- arc and induction furnaces
- melt spinning,
- r.f. sputtering, PLD
- Planetary mills
- spark plasma sintering
- microwave sintering
- hot press sintering
- high temp furnaces
- rolling/drawing machines
- high purity glove box

Characterization:

- SQUID magnetometry,
- PPMS including transport and magneto-conduction, VSM
- Nuclear gamma resonance (Mossbauer)
- DSC, heat capacity and flash calorimetry
- Gas reaction controller with gas absorption/desorption



Nanostructures responding to *magnetic* excitations. Fundamentals and applications.

1. *Magnetic nanostructures for permanent magnets*

- Exchange-spring magnets (nano-composites and multilayers).
- Nanostructures with shape anisotropy
- Molecular magnets, spin frustrated systems

2. *Magneto-functional nanostructures*

- Magneto-conductive nanostructures (spin valves, exchange bias bilayers, surface and interface spin configurations in thin films and multilayers)
- magneto-elastic, magneto-caloric, shape memory alloys
- Multi-functional nanostructures (heterogenous structures interfacing at least one magneto-functional material)

3. *Nanoparticles dispersed in various media*

- Magnetic nanoentities dispersed in metallic/semiconducting/polimeric matrices. Functionalized nanoparticles., biomagnetic.
- Magnetic nanofluids

Superconducting structures.

Fundamentals and applications.

1. MgB₂ based nanocomposites

- Spark Plasma Sintering and Microwave Sintering
- Additions, substitutions, nanocomposites (*pinning engineering*)
- Characterization, pinning and vortex dynamics models
- Application of X-ray tomography for 3D visualization of MgB₂ (collaboration with INFLPR)
- Bulk magnets, storage, concentrators and other devices
- Wires and tapes
- Irradiation and ion implantation
- Biomedical properties and applications of MgB₂

2. HTS based structures

- Nano-dots and nano-objects for pinning in HTS and other purposes (*pinning engineering*)
- Composite heterostructures: growth principles, pinning fundamentals, vortex dynamics
- Whiskers growth, characterization and application

3. Novel and exotic superconductor; search for new superconductors, 2DEG superconducting interfaces

- Li₂(Pd,Pt)B: synthesis and physics
- Fe-Se-Te and other Fe-based superconductors: vortex dynamics
- oxide superconducting interfaces

4. Nb-based superconductors: vortex dynamics

Synthesis and processing of materials can be defined as:

**Materials + (fields or particles) =
new materials and/or new properties and/or new functionality**

‘Fields’ or particles: electrical, magnetic, thermal, optical,

The key question

How to introduce (pump) in a controlled manner energy into a material for its modification?

- Energy order of magnitude depending on purpose?
- Volume and time scale (equilibrium, non equilibrium) ?
- Energy pumping pattern (e.g. pulsed: on/off) ?
- Material / structure to be modified and its relationship to the field/particle ?

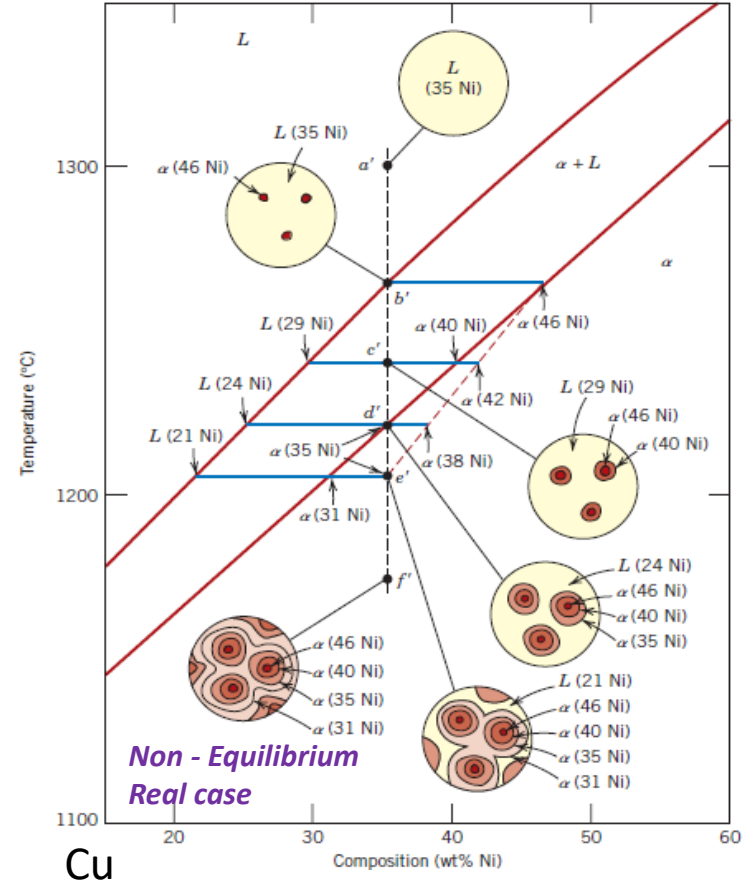
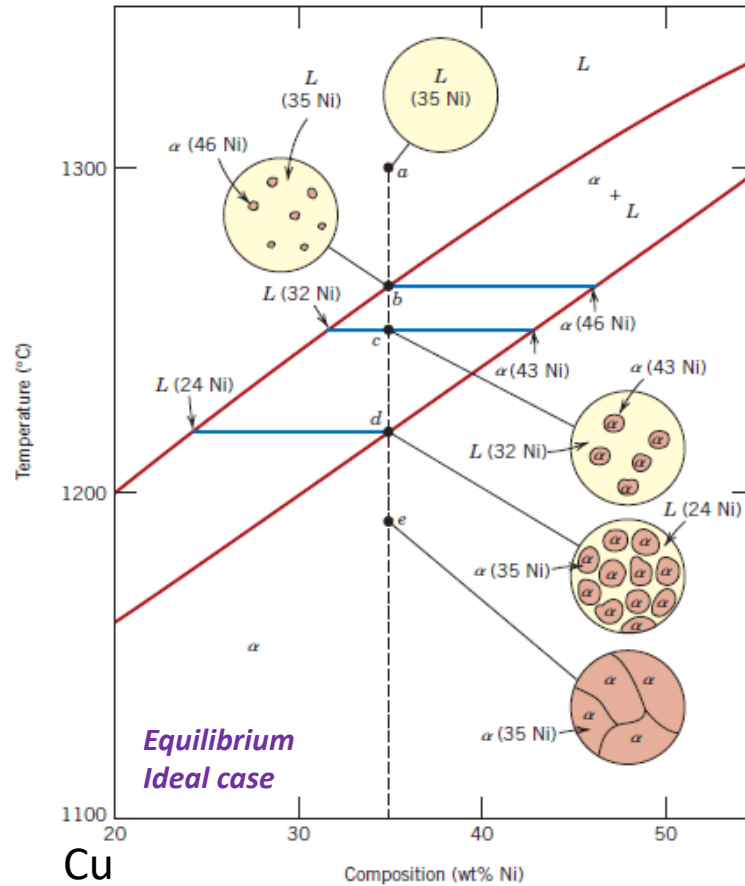


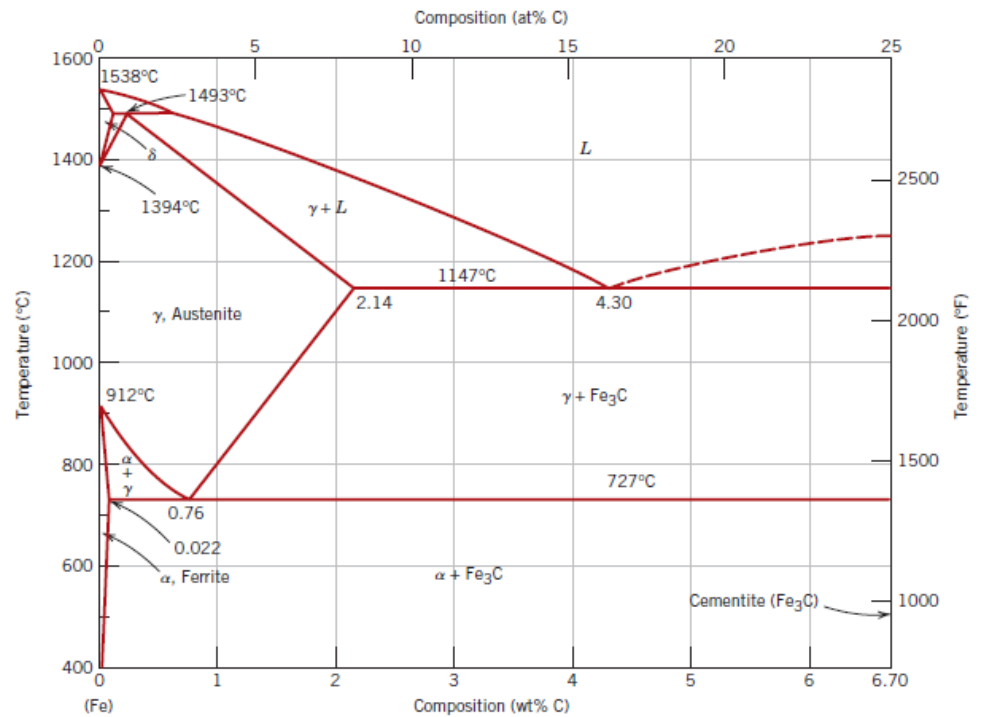
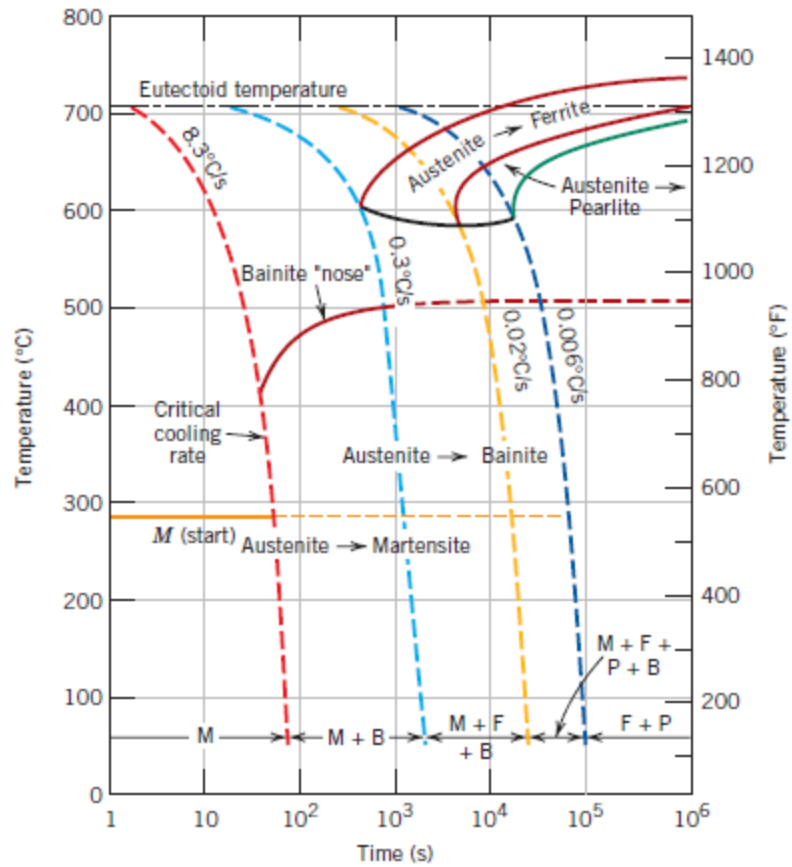
Unconventional and far from equilibrium novel technologies



**X-ray based
technologies**

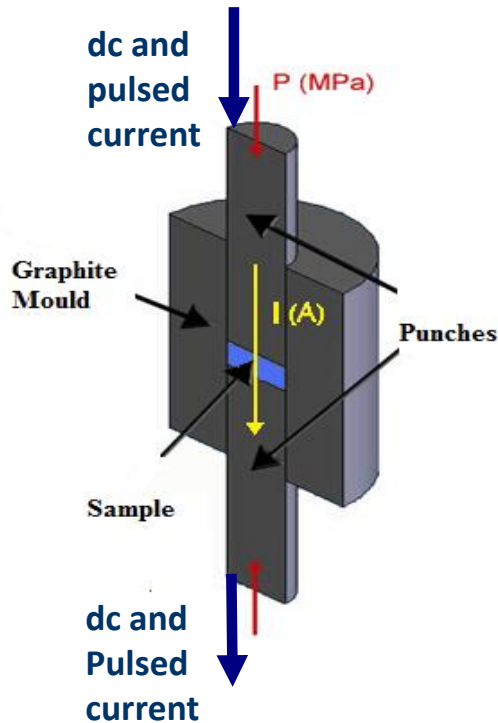
Solid solution





WD Calister Jr and DG Rethwisch, *Materials Science and Eng, An Introduction*, Wiley, 8th Edition

Spark Plasma Sintering (SPS)



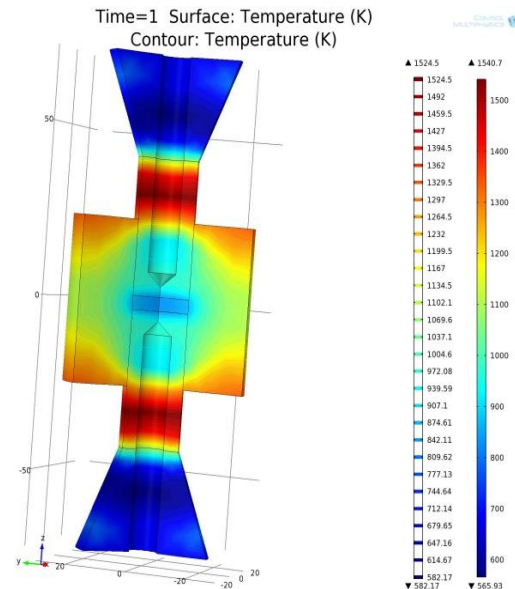
Thermal and non-thermal (debatable) particular features

- rapid heating, cooling (resistive heating)
- **activated** diffusion (electrodifussion?) leading to 'boundary cleaning'
- Discharges and occurrence of plasma states (?): modified boundaries
- hot spots, heating from inside to outside as for MW heating (?)
- moderate or high pressures can be applied

Consequences:

- preserves nano-size,
- high density bulks (especially important for difficult to sinter materials)
- short processing times,
- technique far from equilibrium possibly introducing defects
- Metastable, new or difficult-to-synthesize phases are obtained

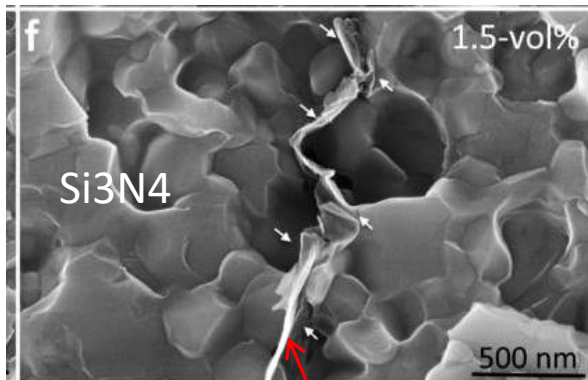
Temp distribution at 867 deg. C (Cu)



FCT-5, Germany

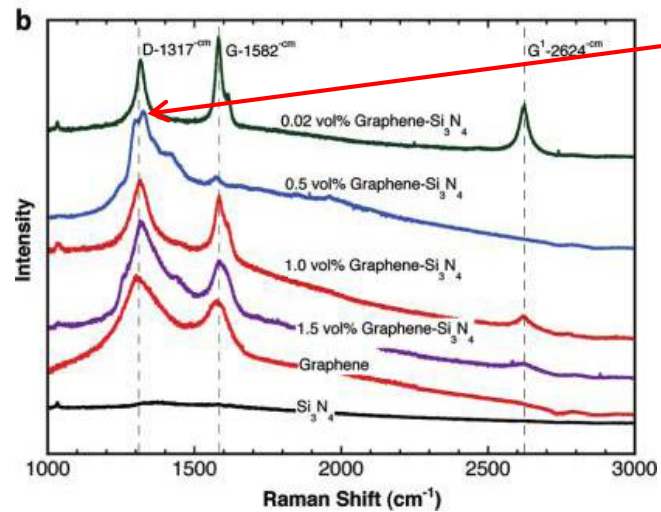
Literature

-SPS can result in formation of phases not available in normal conditions!



SPS

graphene



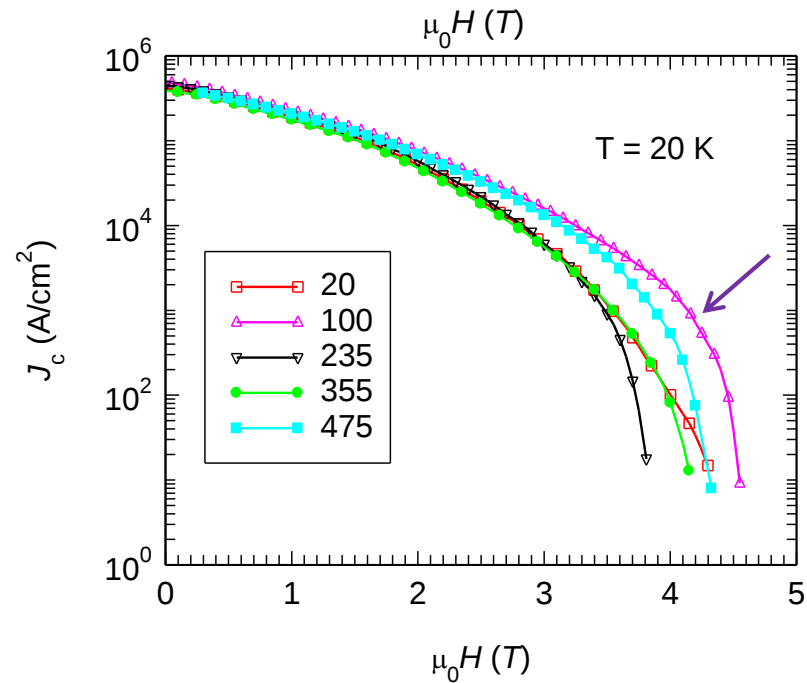
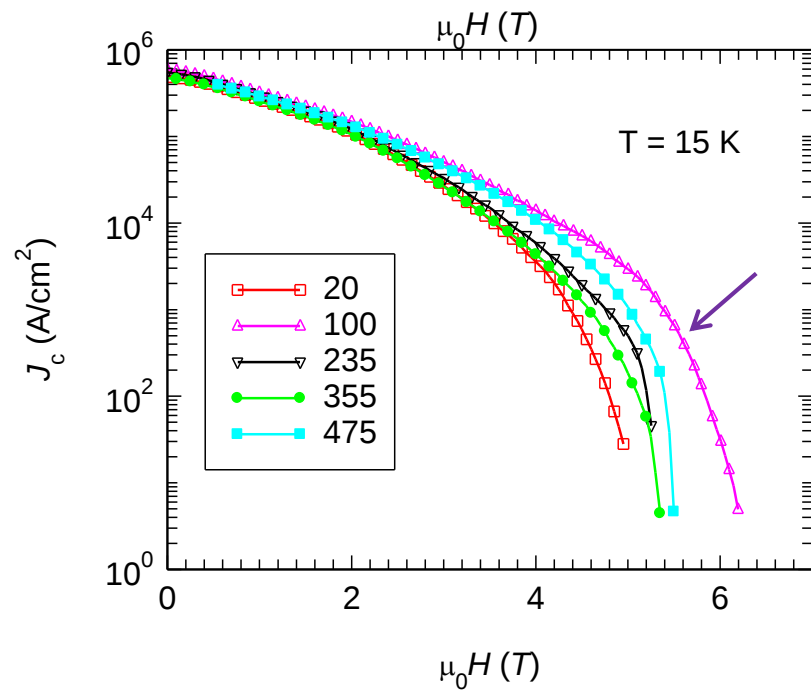
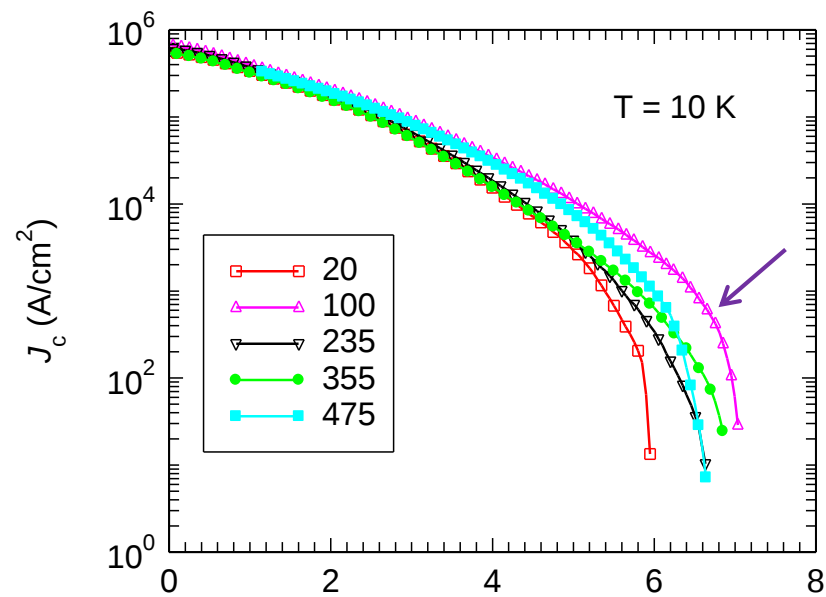
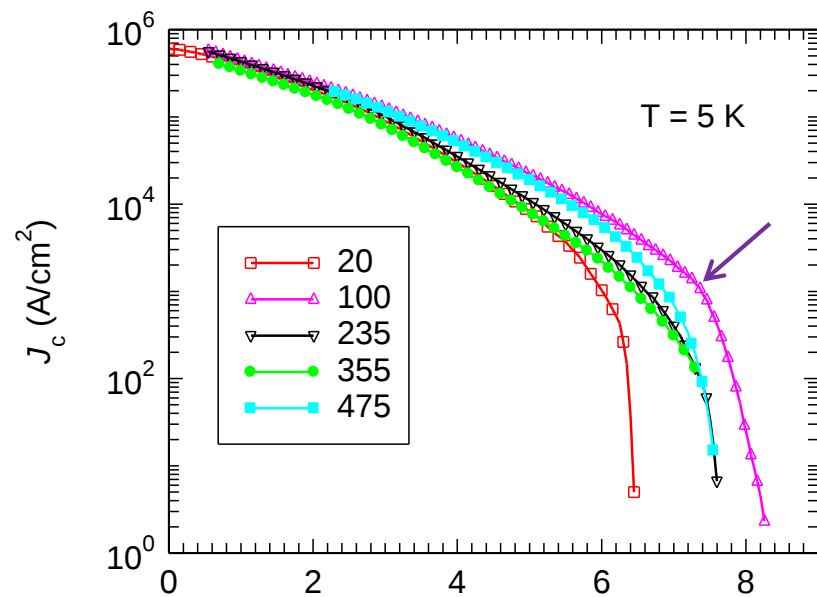
Diamond !!?

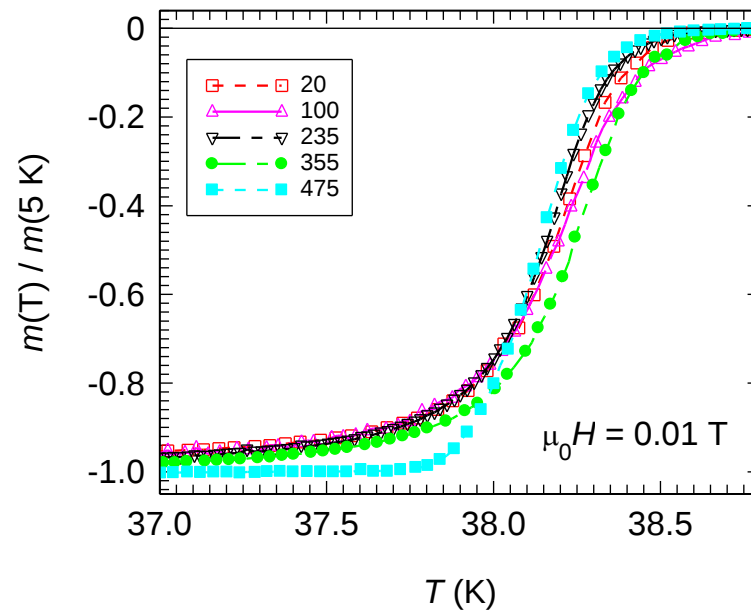
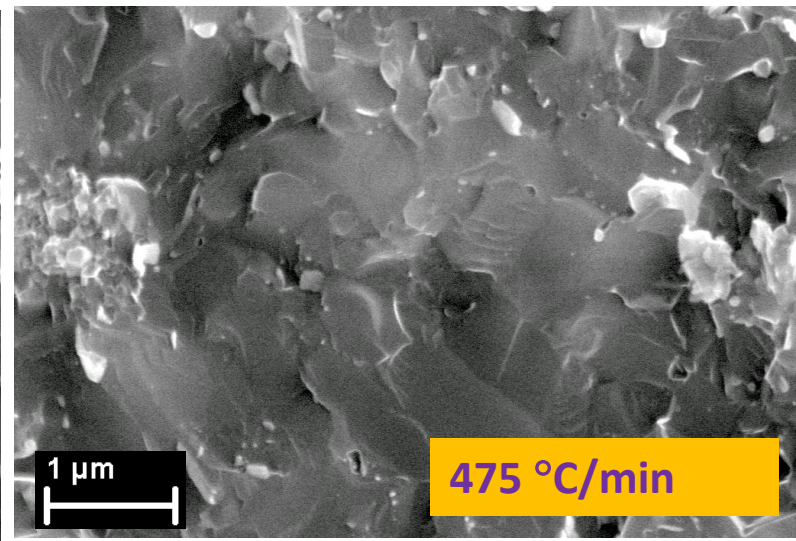
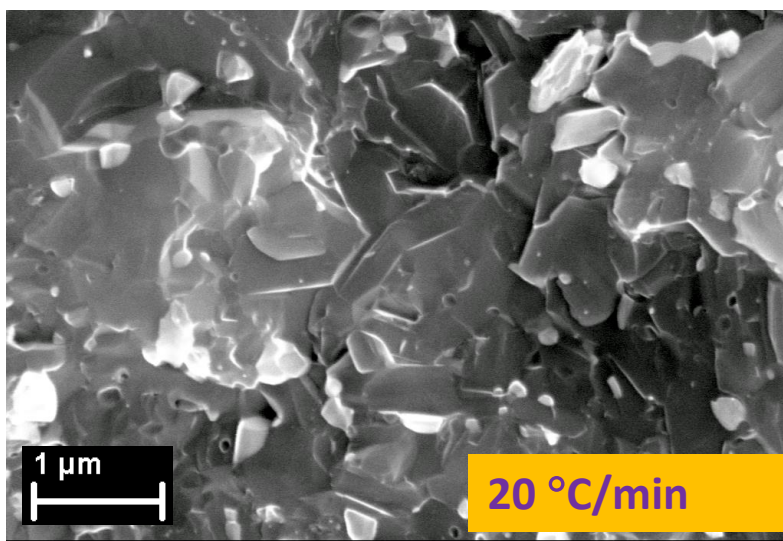
ACS Nano 5 3182 2011

Samples (we use MgB₂ commercial powder)
Heating rate

Sample	Heating rate (°C/min)	Apparent density $\rho a^{SPS}/R^{SPS}$ [g/cm ³]/[%]	Lattice parameter for MgB ₂		Average crystallite size of			Content wt [%]	
			a [Å]	c [Å]	MgB ₂ [nm]	MgO [nm]	MgB ₄ [nm]	MgO	MgB ₄
pw	Powder MgB₂	-	3.0866	3.5226	75 ±7	-	-	-	-
‘20’	20	2.64/99.3	3.0811	3.5249	175 ±41	77 ±1	70 ±20	8.1	13.6
‘100’	100	2.54/95.1	3.0824	3.5270	143 ±38	100 ±12	77 ±25	8.1	11.6
‘235’	235	2.64/99.0	3.0819	3.5264	177 ±57	84 ±10	74 ±17	8.1	13.6
‘355’	355	2.65/99.5	3.0819	3.5253	190 ±60	78 ±3	71 ±22	7.1	11.1
‘475’	475	2.64/99.0	3.0823	3.5260	121 ±25	70 ±5	68 ±18	6.9	11.0

*Crystallite size is shown for a comparative analysis, considering that Williamson-Hall method is not reliable above 100-150 nm.





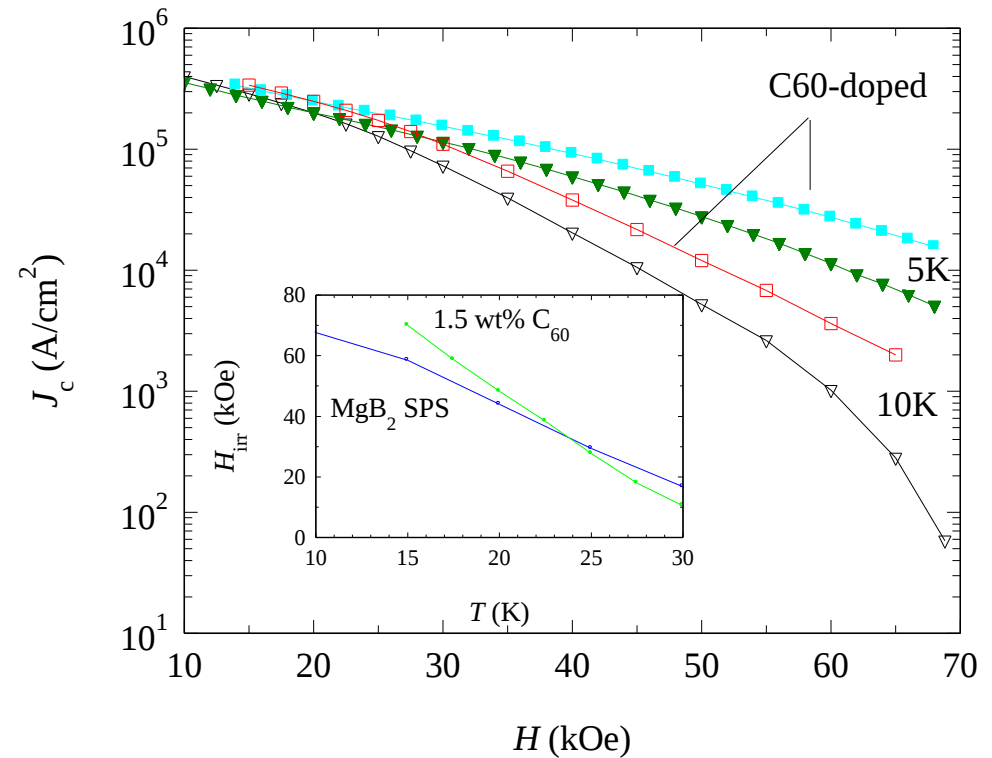
Physica C, 519, 184, 2015

There is an optimum heating rate of ~ 100 $^{\circ}\text{C}/\text{min}$ to maximize $J_{\text{c}0}$, H_{irr} , ($J_{\text{c}0} \times H_{\text{irr}}$), and to partially avoid formation of undesirable flux jumps at low temperatures.

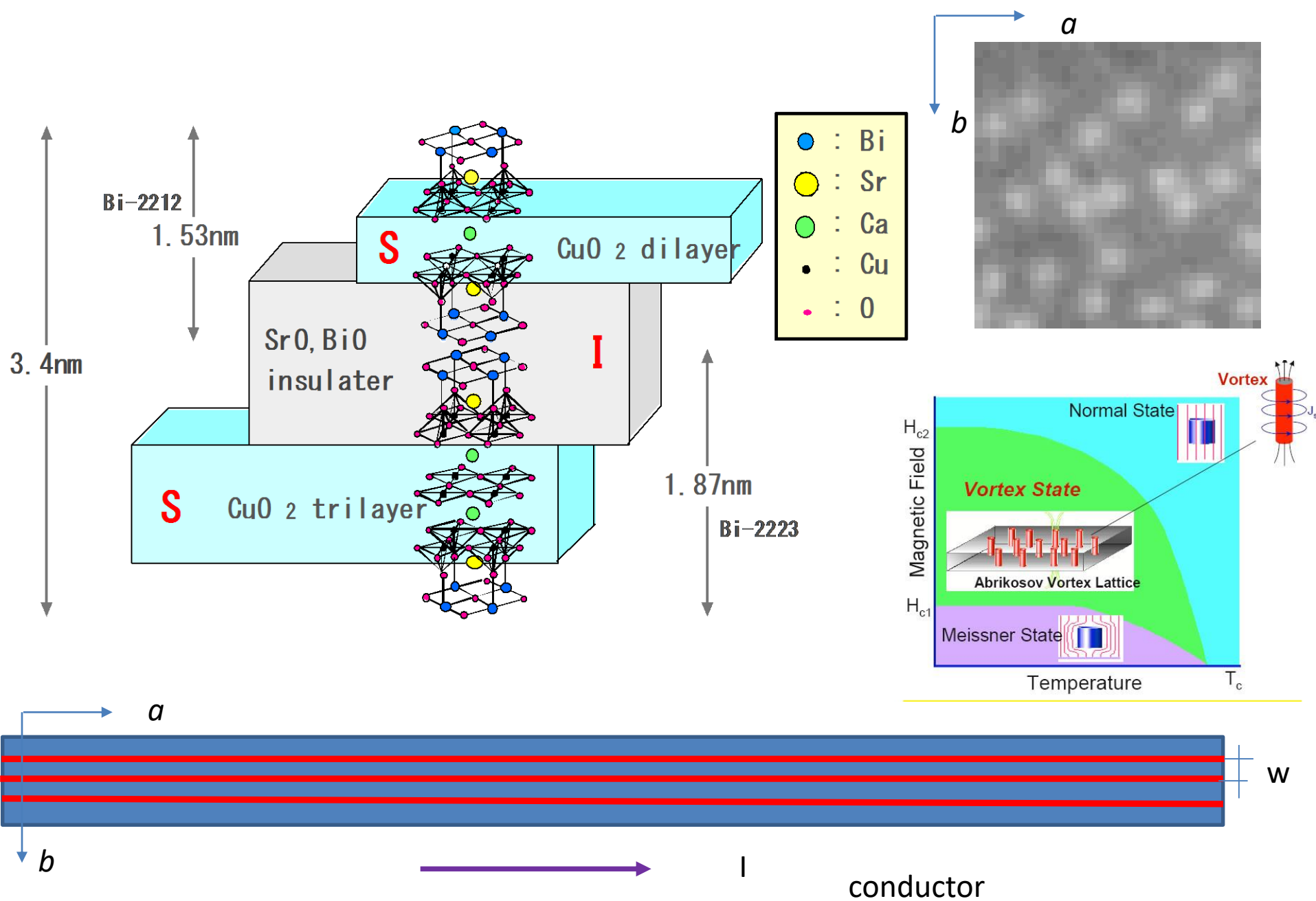
MgB₂ by SPS for different on / off
pulsed pattern of the current
(work in progress)

MgB₂ added with C60 by SPS

Use of graphene C60 was not successful to produce substitution effects of C for B;
J_c was constant (Lim HJ et al, Phys C, 2008, 468 18)



$$\xi < w < \lambda$$



Diameter of nucleus $D \approx 1/H^2$ (Derango, P. et al *Nature* 1991, 349, 770)

Orientation: ($H > 0T$)

-Bi-2212 only a -axis $\perp H$.

Size: ($H > 0T$)

- aspect ratio (length/width) *is decreasing*.
- maximum length and thickness *are decreasing*
(10 - 40 times lower than average at $H=0T$)

Output: ($H > 0T$)

-is *lower*

Defects: ($H > 0T$)

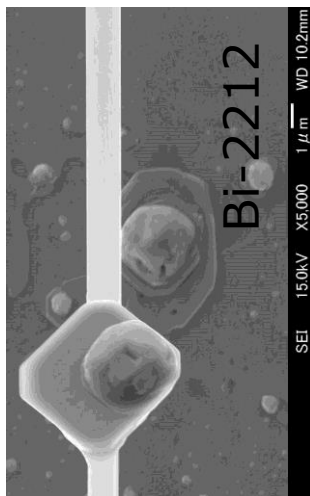
- *less*: less stripes and layers

Crystal quality: ($H > 0T$)

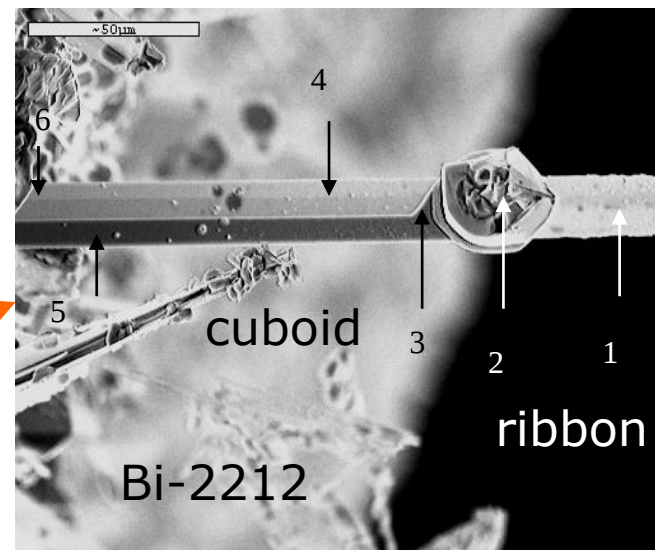
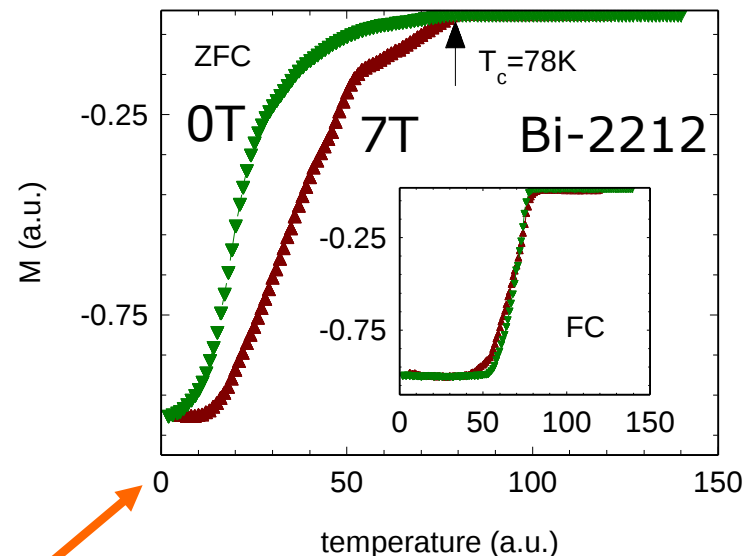
- *narrower* transition

Morphology: ($H > 0T$)

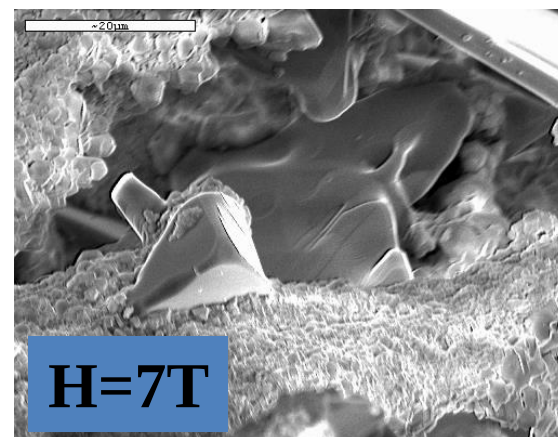
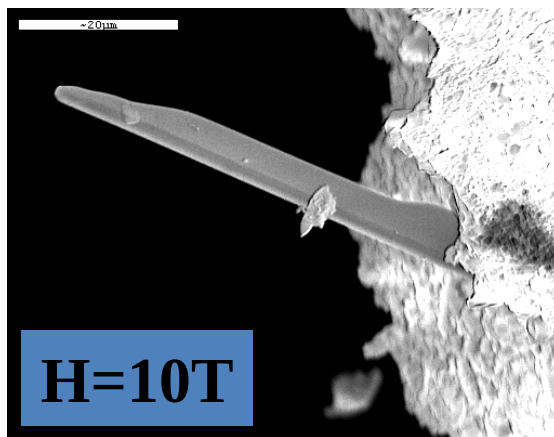
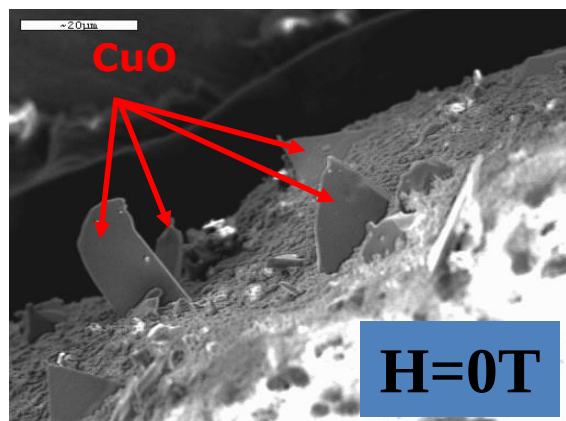
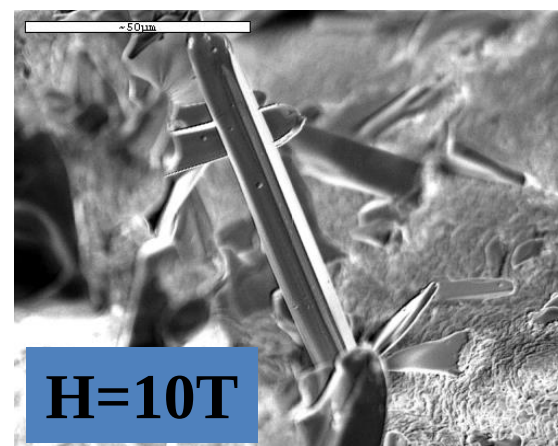
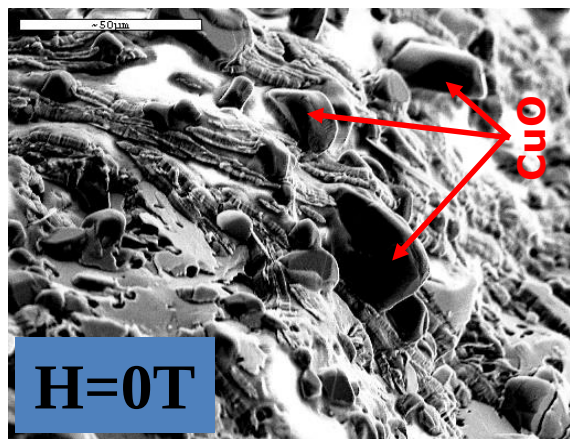
- *cuboid* is possible



Superconducting Bi-2212 whiskers



CuO whiskers



2D or 3D growth

1D growth under H

MoO₃ whiskers

- gas sensing in breadth analysis
- pseudocapacitive materials with insertion/removal of Li

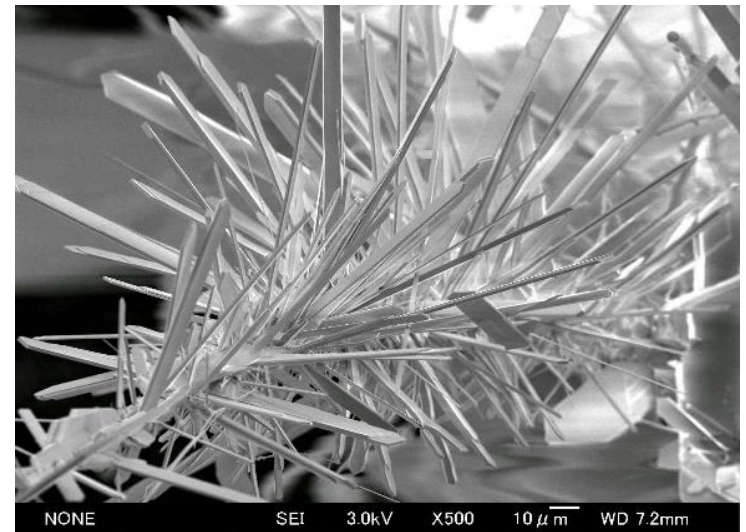
LaSrMnO₃ – films by PLD -magnetic

ZnO (Mn) diluted magnetic films by sputtering

CeO₂-Tb/Yb grown by PLD
for optical purposes and conversion in solar cells

(Sr, Ba)TiO_x for MW and THz

Bi₂(Sr, Ca)₂Co₂O_x thermoelectric whiskers



Thank you for your attention !!!!