FACULTY OF SCIENCES

DEPARTMENT OF CHEMISTRY
RESEARCH GROUP SCRiPTS

# PROGRESS TOWARDS $YBA_2CU_3O_{7-\delta}$ NANOCOMPOSITE THIN FILMS USING CHEMICAL SOLUTION DEPOSITION AND PREFORMED NANOCRYSTALS

**Katrien De Keukeleere**

*H. Rijckaert, J. Hänisch, P. Cayado, A. Meledin, M. Sieger, F. Valles, M. Coll, A. Palau, J. Sierra, P. López-Domínguez, R. Hühne, J. De Roo, R. Hühne, G. Van Tendeloo, P. Paturi, H. Huhtinen, T. Puig, X. Obradors ,M. Hemgesberg, M. Bäcker, and I. Van Driessche*

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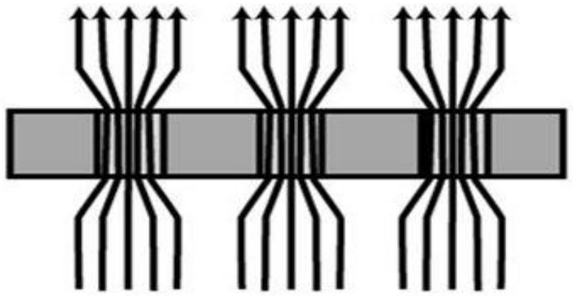
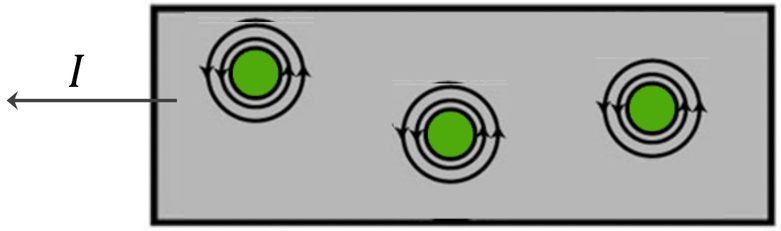
# Vortex pinning



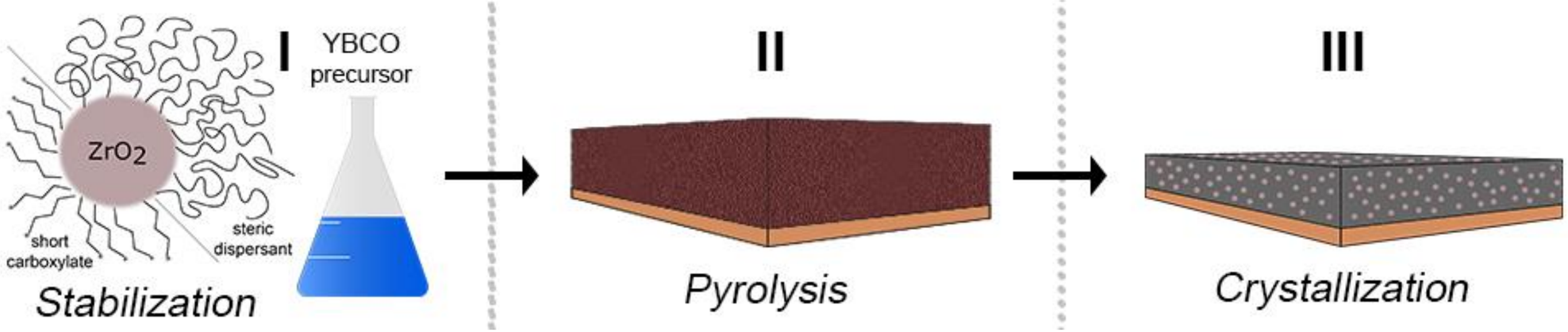



$F_p = F_L = I_c \times B$

*Lorentz force*
*Movement of vortices*
***Energy dissipation***

*Pinning force =Lorentz force*
***Pinning of vortices***

# CSD approach

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# PREFORMED NANOCRYSTALS

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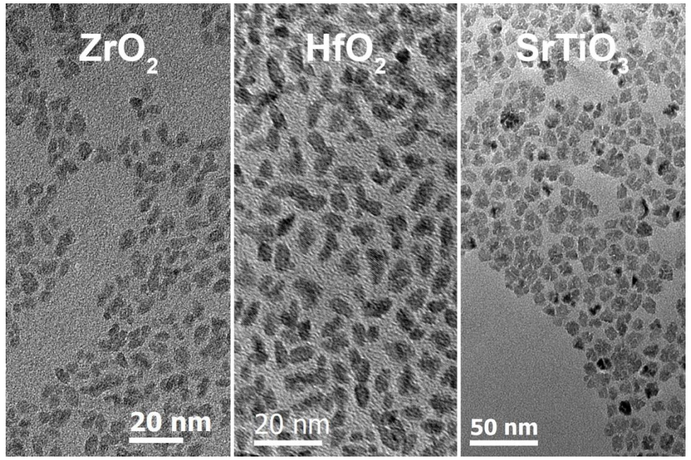
# Nanocrystal synthesis

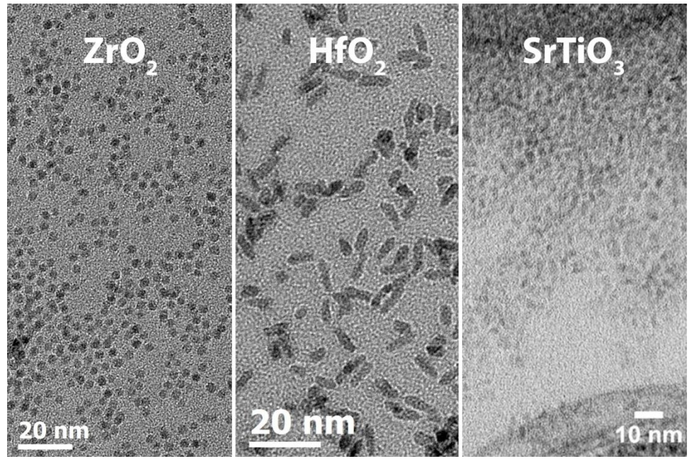
## Microwave synthesis

Metal alkoxide in benzyl alcohol




## Heating-up synthesis

Metal alkoxide in tri-n-octylphosphine oxide




**Goal:**
- Crystalline material
- Particle size: 3-10 nm
- Individual particles, free from agglomeration
- Stable in different solvents and YBCO

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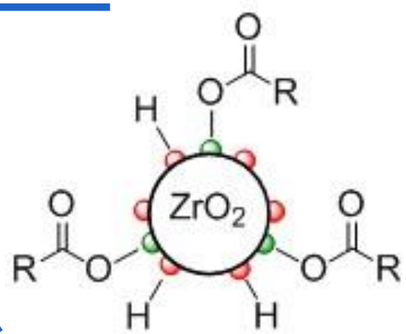
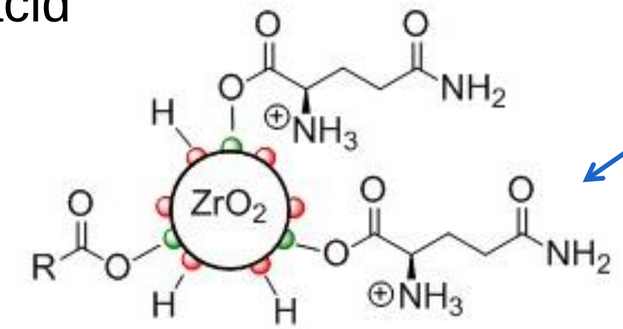
*J. De Roo et al., J. Am. Chem. Soc. (2014), 136, 9650*
*K. De Keukeleere et al., Inorg. Chem. (2015), 54, 3469*
*H. Rijckaert & K. De Keukeleere et al., in preparation*

*J. Joo et al., JACS (2003), 125, 6553-6557*
*J. Tang et al., Chem. Mater. (2004), 16, 1336*
*H. Rijckaert & K. De Keukeleere et al., patent pending*

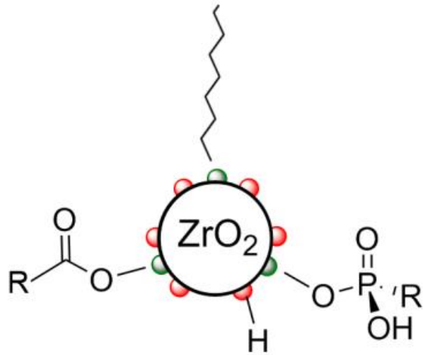
# Nanocrystal stabilization

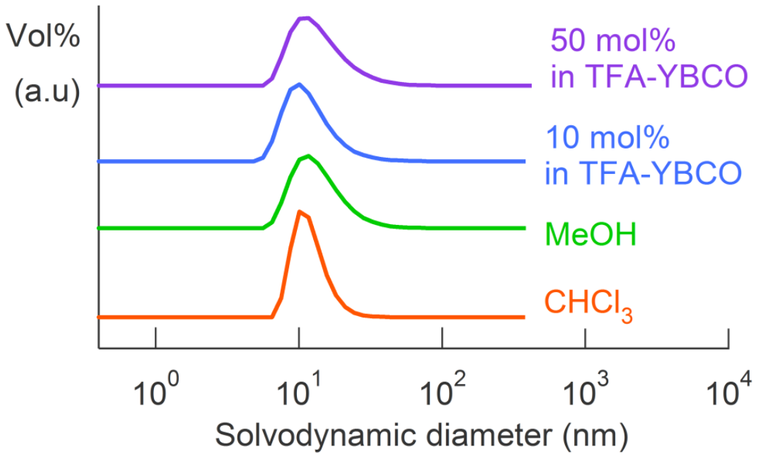


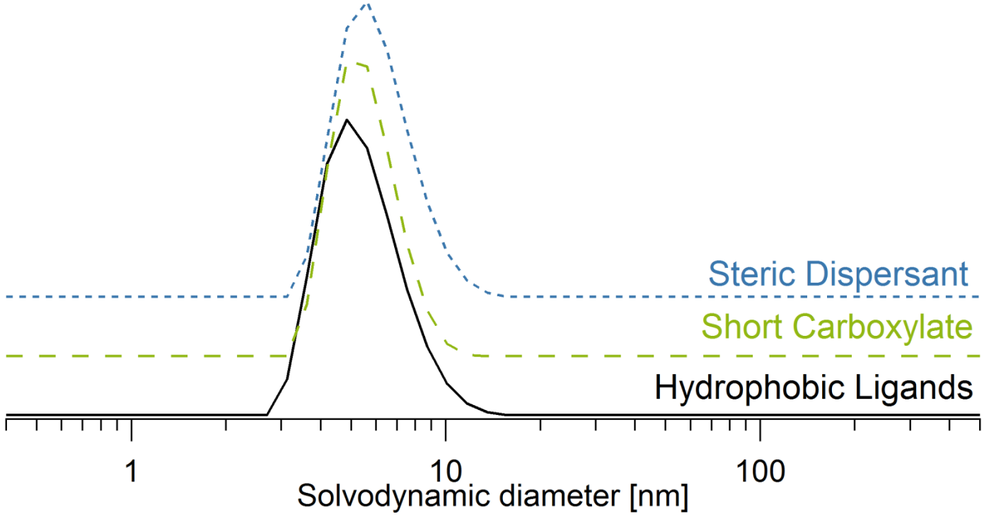

**Charge** and **steric** stabilization
via amino acid




**Steric** stabilization
via short carboxylate or steric dispersant

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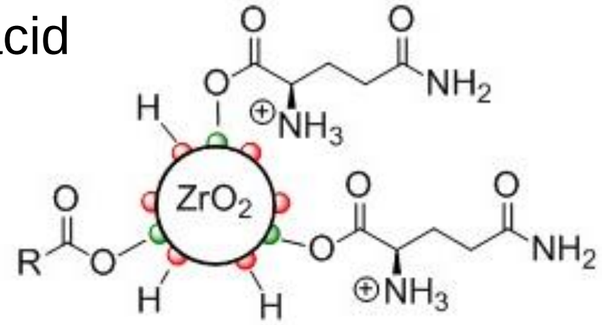
*J. De Roo et al.; Langmuir. (2016) 32; 1962*

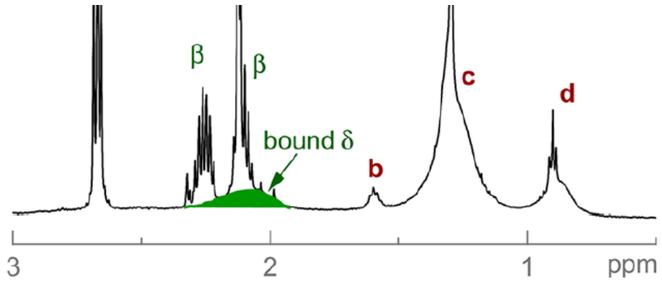
*H. Rijckaert et al.; Chem. Mater. (2017) 29; 1962*

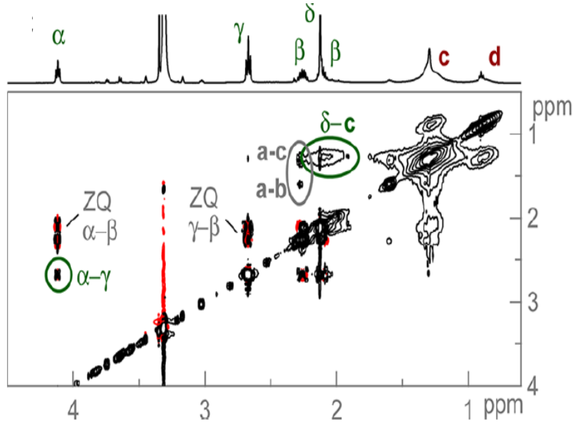
# Nanocrystal stabilization

**Charge** and **steric** stabilization

via amino acid




**Steric** stabilization

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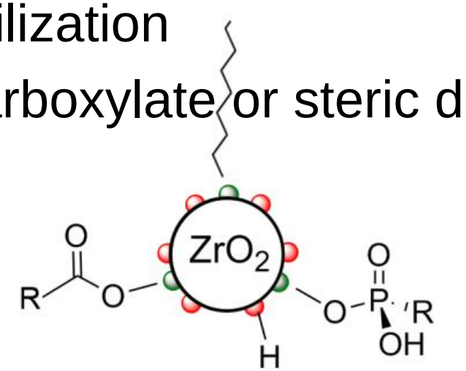


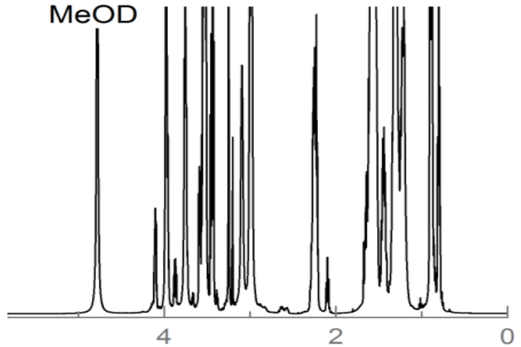


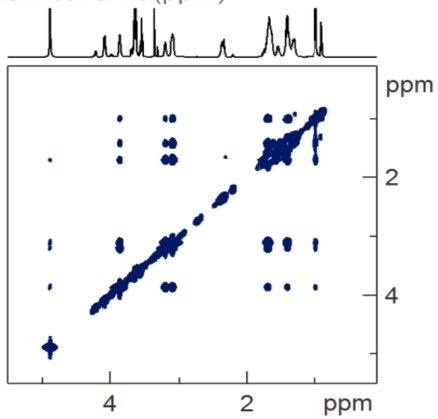


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*J. De Roo et al.; Langmuir. (2016) 32; 1962*

*H. Rijckaert et al.; Chem. Mater. (2017) 29; 1962*

# TFA-BASED NANOCOMPOSITE

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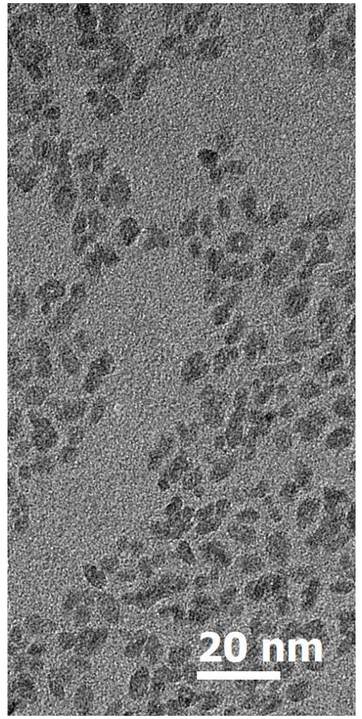
# TFA-based nanocomposite

**$ZrO_2$ nanocrystals**

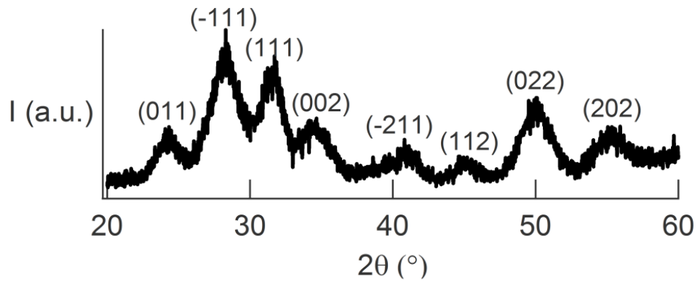



**Microwave synthesis**

$ZrCl_4$ in benzyl alcohol

220 °C – 4h

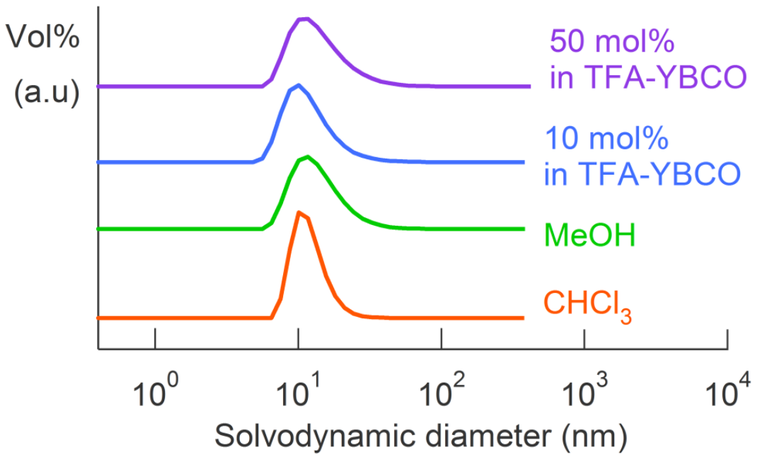
| | | |
|---|---|---|
| TEM | = | 7 nm |
| DLS | = | 11 nm |
| XRD | = | 6 nm |
| Phase | = | monoclinic |
| Rietveld | = | 70% |




*K. De Keukeleere et al., Inorg. Chem. (2015) 54, 3469*

**Ligand Exchange/Phase transfer**

Via glutamine and TFA




*J. De Roo et al.; Langmiur. (2016) 32; 1962*

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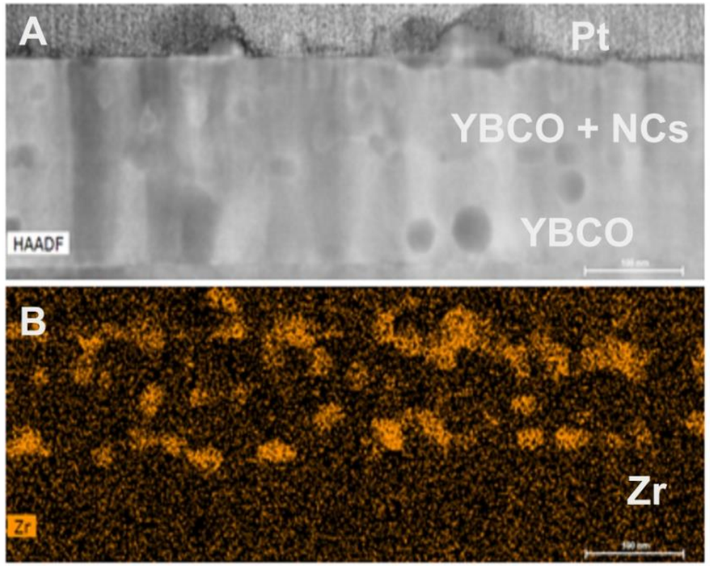
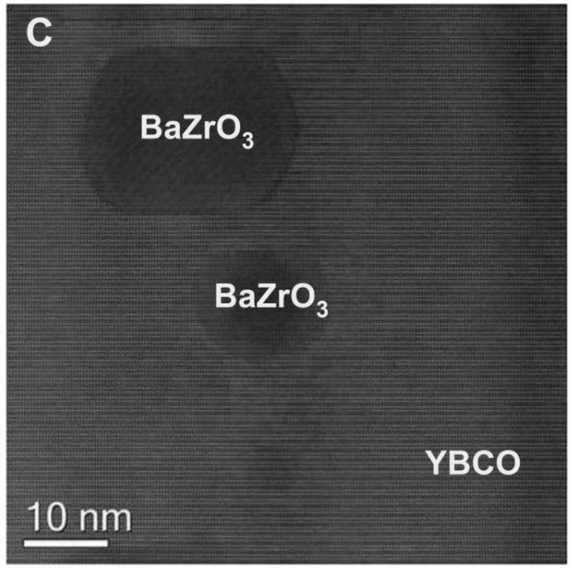
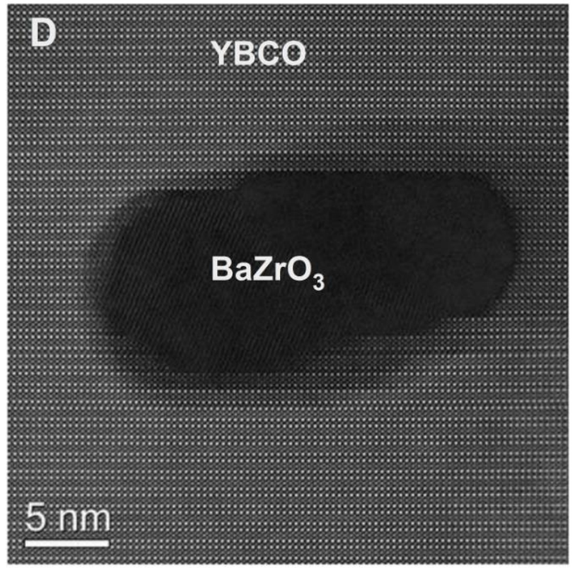
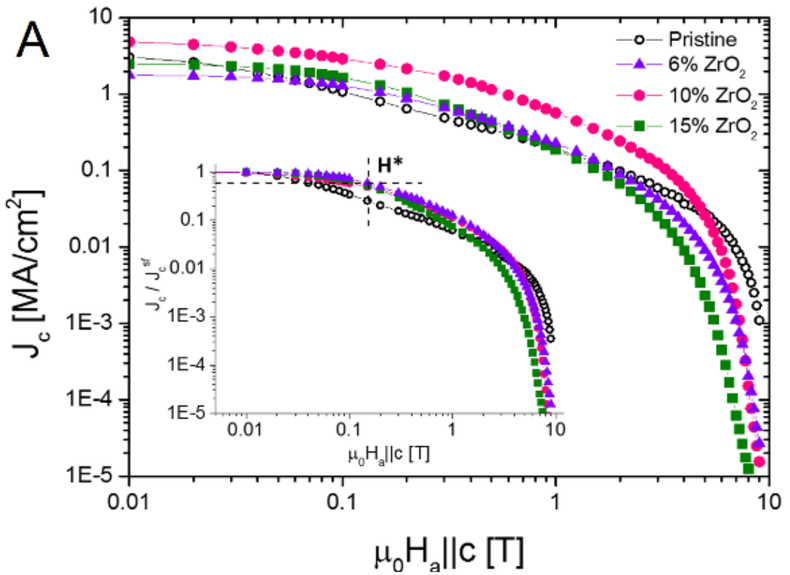
# TFA-based nanocomposite

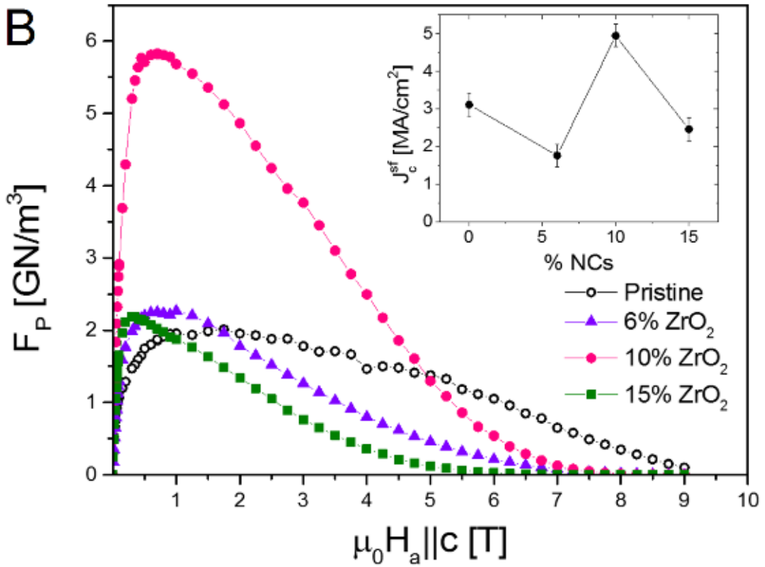
Seed layer




**Zr- phase present throughout the YBCO layer**

**Some growth/agglomeration occurs**

**<u>Small amount of stacking faults</u>**

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*K. De Keukeleere, P. Cayado et al., Adv. Electr. Mater. (2016) 2, 1600161*

# LOW FLUORINE-BASED NANOCOMPOSITE

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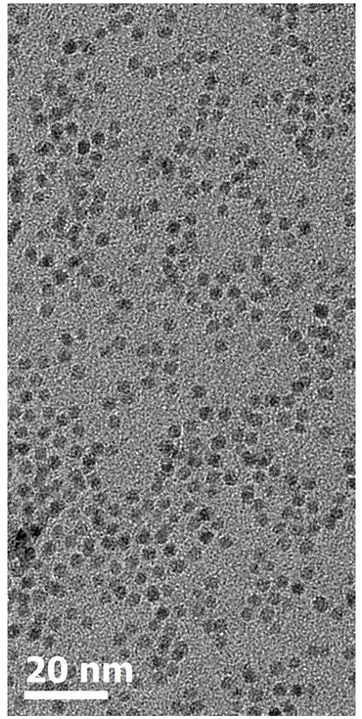
# Low-fluorine nanocomposite

**$ZrO_2$ nanocrystals**

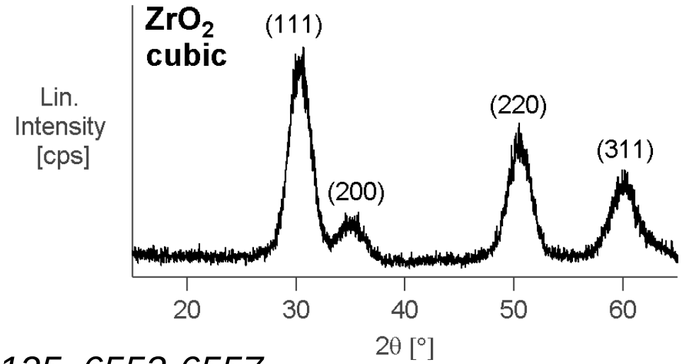



**Heating up synthesis**

$Zr(OC_3H_7)_4$, $ZrCl_4$ and TOPO

340 °C – 2h

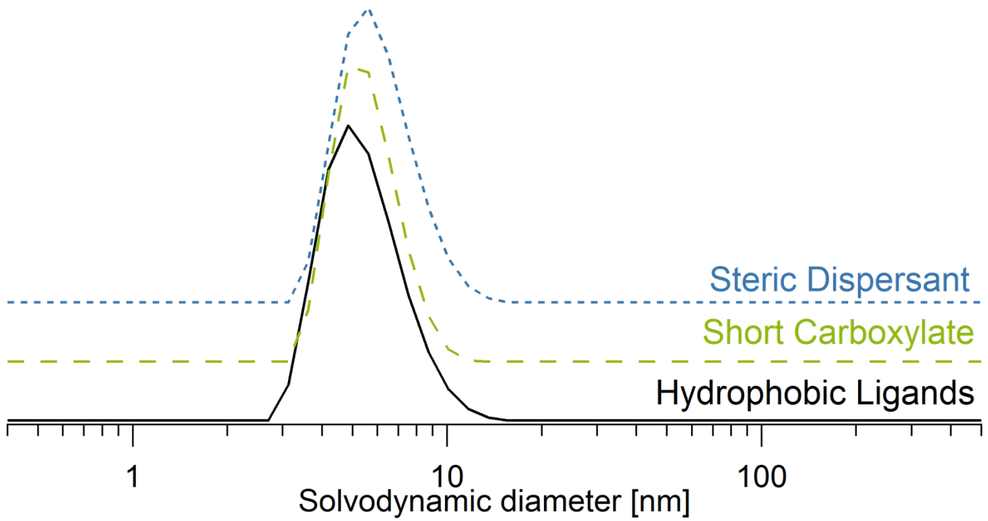
| | | |
|---|---|---|
| TEM | = | 3.5 nm |
| DLS | = | 6.4 nm |
| Scherrer | = | 3.5 nm |
| Phase | = | cubic |
| Rietveld | = | 80 % |




*J. Joo et al., JACS (2003) 125, 6553-6557*

**Ligand Exchange/Phase transfer**

Via steric dispersant or short carboxylate




*H. Rijckaert et al.; Chem. Mater. (2017) 29; 1962*

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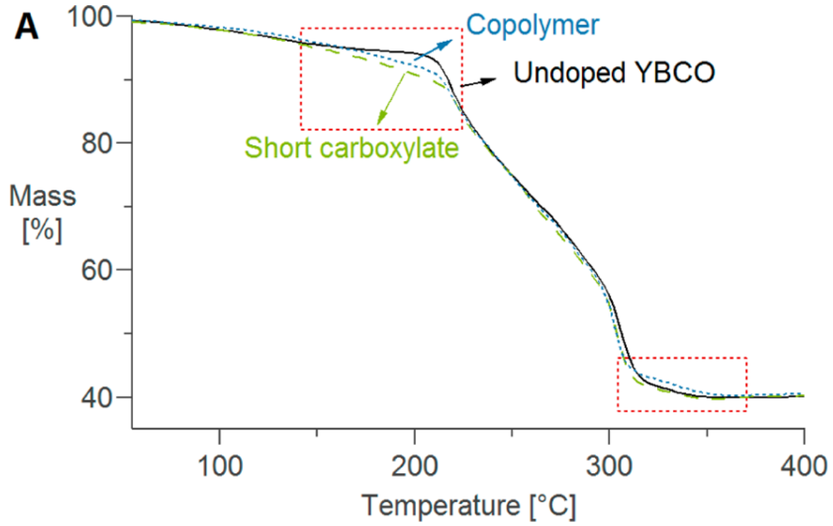
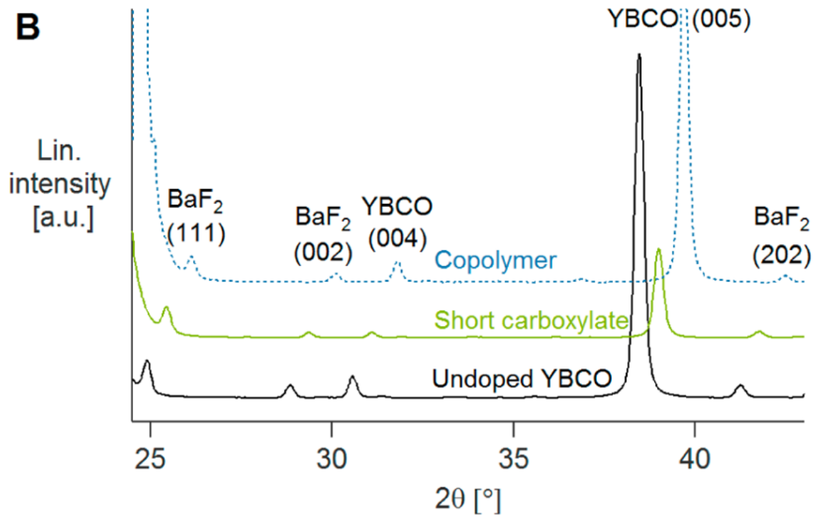
# Low-fluorine based nanocomposite




| Ligand | $d_s$ | Polydispersity | $T_{decomp}$ | $J_{c,sf}$ (77 K) |
|---|---|---|---|---|
| | nm | index | °C | MA cm$^{-2}$ |
| **Copolymer** | 6.4 | 0.32 | 340 | 5.1 |
| **Citric acid** | 5.8 | 0.86 | 250 | 2.3 |
| **Tartaric acid** | 5.9 | 0.91 | 265 | 1.6 |

**Superconducting properties are depending on the used ligands and the nature of nanocrystals**

*H. Rijckaert et al.; Chem. Mater. (2017) 29; 1962*

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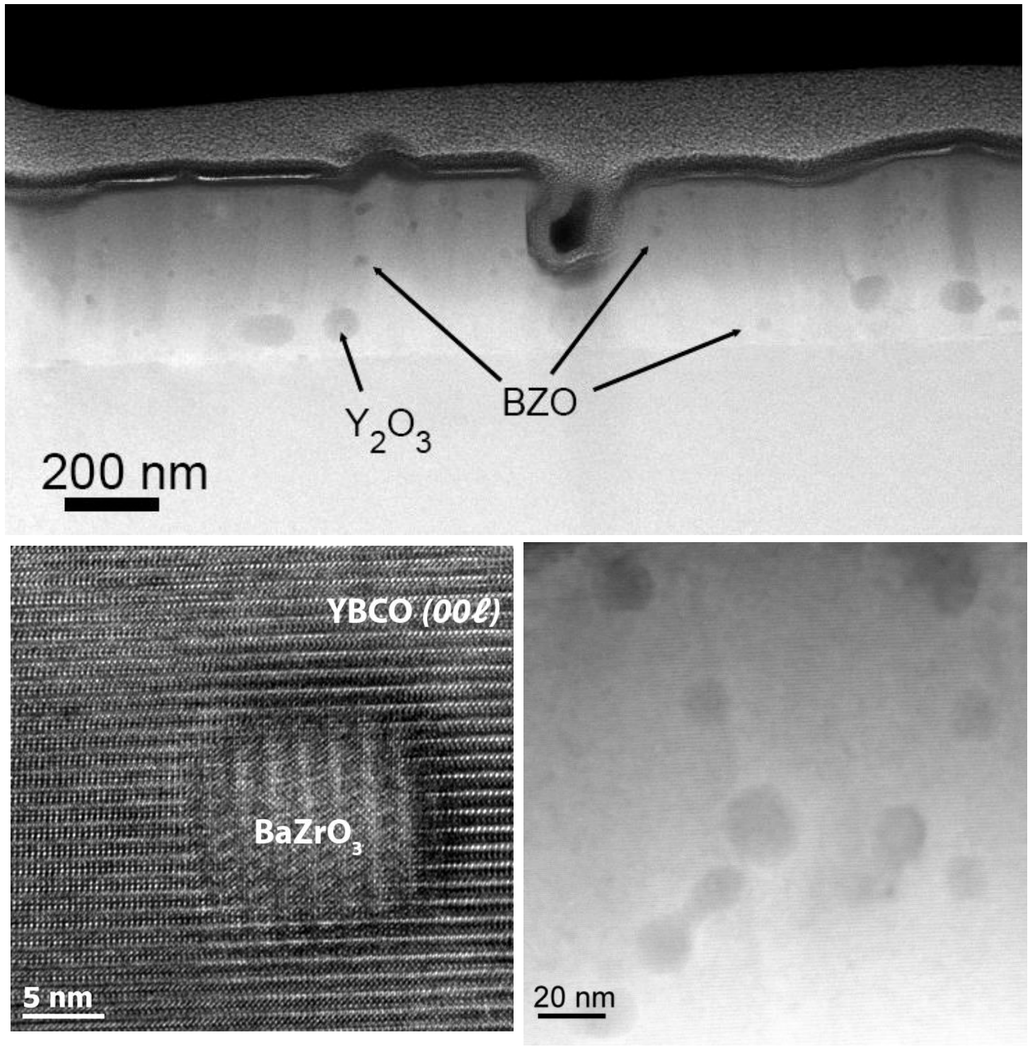
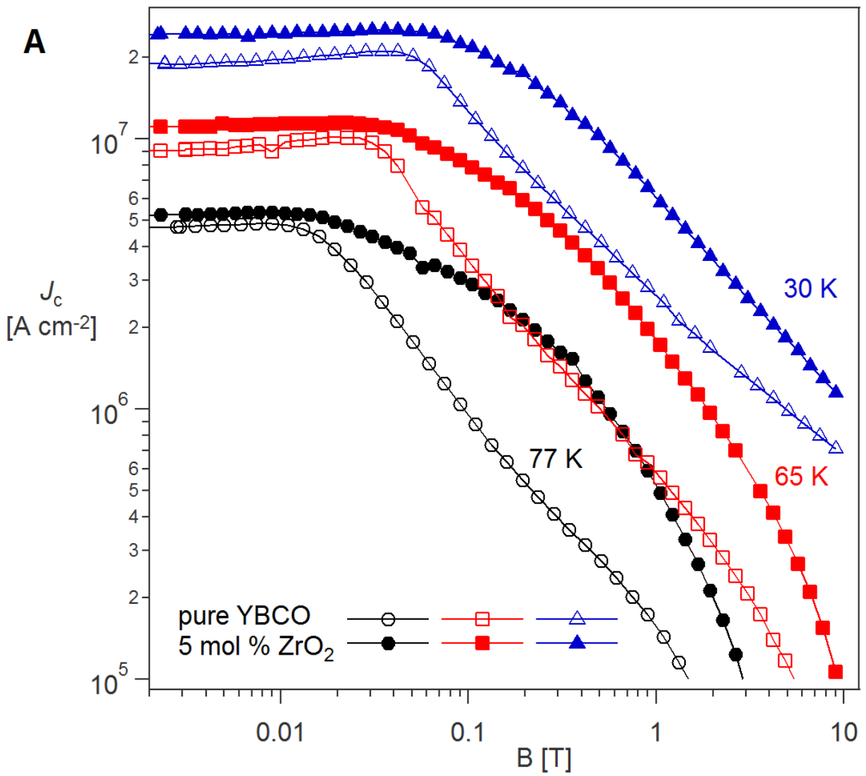
# Low-fluorine based nanocomposite



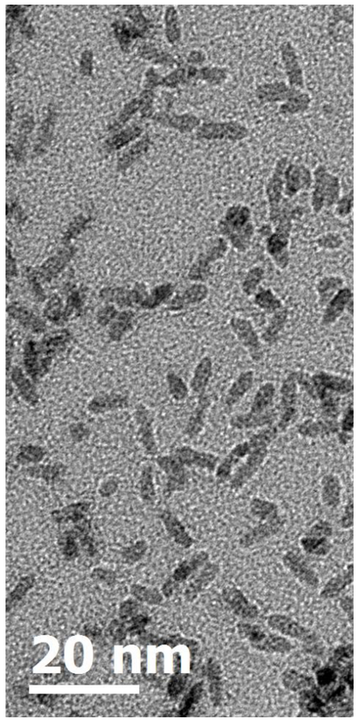

**Homogeneous distribution of randomly oriented 10 – 25 nm particles in YBCO layer**




*H. Rijckaert et al.; Chem. Mater. (2017) 29; 1962*

# Low-fluorine nanocomposite

**$HfO_2$ nanocrystals**

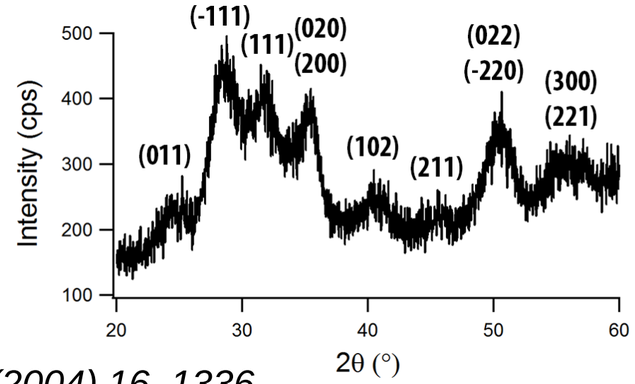



**Heating up synthesis**

$Hf(OC_3H_7)_4$, $HfCl_4$ and TOPO

360 °C – 2h

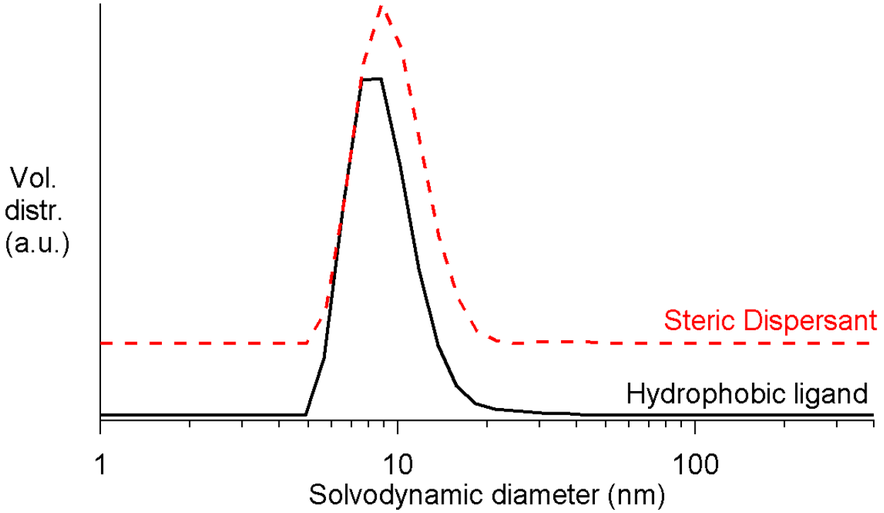
| | | |
|---|---|---|
| TEM | = | 2.3-8.0 nm |
| DLS | = | 10.2 nm |
| Scherrer | = | 3.5 nm |
| Phase | = | monoclinic |
| Rietveld | = | 70% |




*J. Tang et al., Chem. Mater. (2004) 16, 1336*

**Ligand Exchange/Phase transfer**

Via steric dispersant

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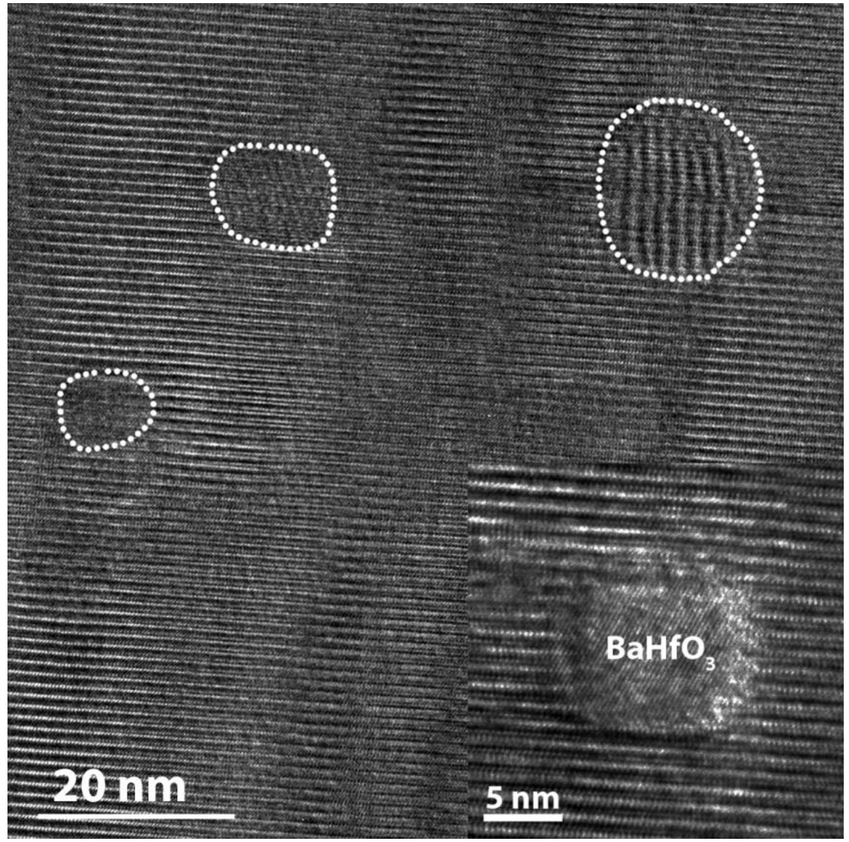
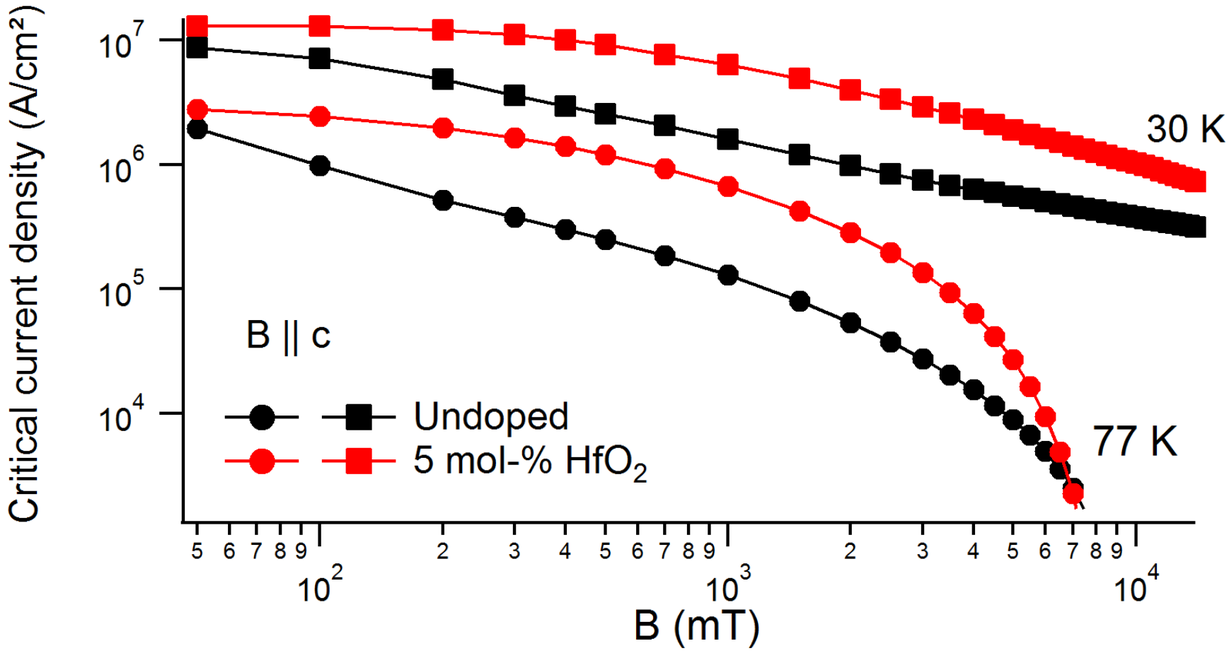
# Low-fluorine based nanocomposite




**Homogeneous distribution of randomly oriented 8 – 20 nm particles in YBCO layer**

More details @ Poster 1MP2-02

# CONCLUSION

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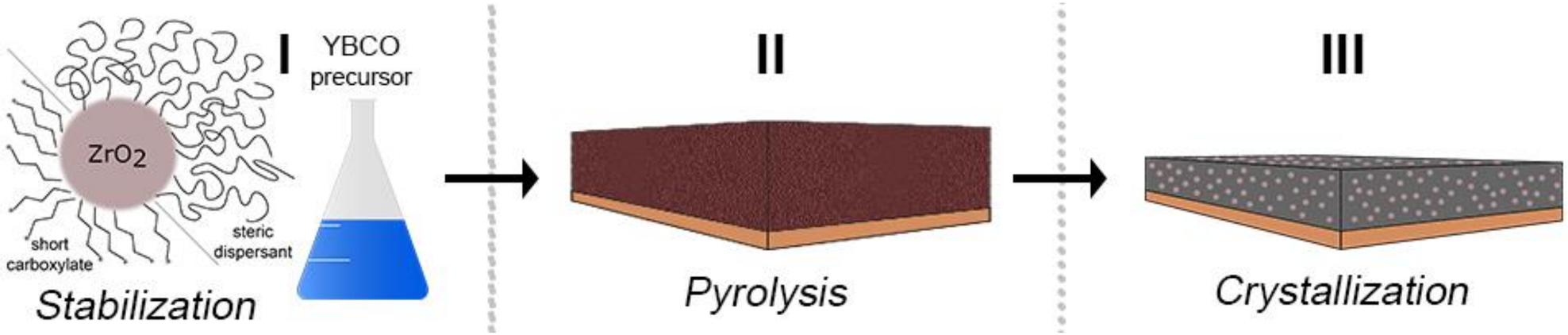
# Conclusion




- Chemical solution deposition methods
- Pré-synthesized nanocrystals
- Importance of ligands
- Introduction in both TFA-based as low fluorine YBCO precursors
- Epitaxial YBCO films with improved $J_c(H)$ decay

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# Acknowledgements


**SCRiPTS**
*H. Rijckaert, J. De Roo, J. Sierra, P. López-Domínguez, I. Van Driessche*

**d-Nano**
*J. Bennewitz, M. Rikel M. Falter, M. Bäcker*

**ICMAB-CSIC**
*P. Cayado, S. Ricart, T. Puig, X. Obradors*

**BASF**
*A. Meyer, M. Hemgesberg*

**University of Turku**
*H. Huhtinen, P. Paturi*

**IFW Dresden**
*M. Sieger, R. Hühne*

**KIT**
*J. Hänisch, R. Nast*

**EMAT (UAntwerp)**
*A. Meledin, G. Van Tendeloo*