2MO4-07 Multi-deposited inkjet printed YBCO on IBAD-MgO architectures for coated conductors

Spin-off technology-based company founded in 2010

- Specializing in scale-up of chemically-derived nanostructured functional oxide thin films.
- Staff: 8 people now + student internships
- Turnover around 250,000 € in 2016 and (project grants, and products sold worldwide: PVD targets and customized furnaces)

+200 m² industrial site 5 km away + 500 m² (to be inaugurated)

Private investor: industrial

Venture capital
Achievements

2010
- Foundation
- 1st financing round
- La Farga enters equity
- Start of activities in superconductivity

2011
- FP7 Eurotapes project

2012
- First research contract
- Signed licence agreement with CSIC

2013
- First sales of PVD targets, furnaces
- Continuous inkjet pilot plant in operation
- Start of activities in solar oxides

2014
- Award from REPSOL FUNDATION
- UV curing of inks

2015
- 2nd financing round (300 k€)
- H2020 project (500 k€)

2016
- 10 meters of HTS tape, new R+D contracts

2017
- Extension to industrial park with 200 m²
Tape architecture

- DC
- PL
- Dual PLD Chamber system

- High-Tc superconductor
- CeO$_2$:RE
- LaMnO$_3$
- IBAD - MgO
- Al$_2$O$_3$ or Y$_2$O$_3$
- Hastelloy

- PLD-1 (100-200 nm) at T1
- RF sputtering-2 (30-50 nm) at T1
- Ion beam assisted deposition with RF sputtering 5-7 nm
- Single Chamber

- Al$_2$O$_3$: by RF reactive or nonreactive sputtering
- Y$_2$O$_3$: by solution deposition planarization

- Cold rolled, annealed, electro polished (60-100 microns)
PILOT PLANT FOR REEL-TO-REEL INKJET DEPOSITION AND PYROLYSIS
Gas extraction system for the removal/filtering of ink solvents and decomposition products
Low-F YBCO multi-deposition pyrolysis in R2R mode by Inkjet Printing: 10 layers, 1 µm thickness

Compact layers after pyrolysis obtained by inkjet printing multi-deposition

Cu-rich interlayers are visible between the multideposited YBCO films.
Observations: still $n=5$ is highest in $I_c$. Slower pyrolysis ramps in $O_2$ produce flatter samples and this is main limitation for $I_c$ enhancement. Growth conditions become more and more relevant too for top results in thicker films.
Characterization of the YBCO samples by IJP and pyrolyzed in R2R mode

Record $I_c$ obtained at OXOLUTIA by inkjet printing and still large room for improvement in the short term

$J_c(77K, sf) \approx 2$ MA/cm$^2$

$I_c = 90$ A/cm-width
Deeper look into the microstructural and magnetic characterization of the LF-YBCO IJP films

Low-resolution TEM cross-section image of the sample, shows the different layers of the CC. We note there is some presence of a reactive phase at the interface between the cap CGO and the YBCO layers, identified to be $\text{BaCeO}_3$ (BCO).

Around 30-40 nm BCO, can be also appreciated from the EDX maps of Ba and Ce.

V.R. Vlad et al. - Inkjet printing multideposited YBCO on GO/LMO/MgO/$\text{Y}_2\text{O}_3$/Al$_2$O$_3$/Hastelloy tape for 2G Coated Conductors
To be published
Deeper look into the microstructural and magnetic characterization of the LF-YBCO IJP films

The formed BCO doesn’t seem to disturb the epitaxial growth of YBCO on top; the major part from the 200 nm of CGO remains unreacted.

High density of short Y248 intergrowths with some triple Cu chains. → Y125 (R. Guzman et al. APL(2013))

V.R. Vlad et al. - Inkjet printing multideposited YBCO on CGO/LMO/MgO/Y$_2$O$_3$/Al$_2$O$_3$/Hastelloy tape for 2G Coated Conductors To be published
Magnetic characterization

Granularity analysis

<2a>=0.80±0.05 µm

J_c^G=49±2 MA/cm² at 5K

J_c^{GB}=15.9 MA/cm² at 5K

J_c^{GB}/J_c^G=0.3

V.R. Vlad et al. - Inkjet printing multideposited YBCO on CGO/LMO/MgO/Y_2O_3/Al_2O_3/Hastelloy tape for 2G Coated Conductors
To be published

In collaboration with E. Bartolomé

J_c^{GB}=15.9 MA/cm² at 5K

J_c^{GB}=6.06 MA/cm² at 50K

J_c^{GB}=1.23 MA/cm² at 77K
SCALE-UP TO 10 METERS:
REEL-TO-REEL INKJET DEPOSITION AND PYROLYSIS OF YBCO\textsuperscript{TFA}

- SUPEROX substrate. \textbf{SuperOx}
- 2 inkjet+pyrolysis cycles.
- Nominal thickness (as-grown): 450 nm approx.
- Inkjet deposition speed: 3.5 m/h, 1 printhead
System for long length YBCO conversion

- Speeds up to 100 m/h, 12 mm width, 50 mbar- 1 bar of total pressure
- Initial testing of **10 meters**
- **Capable of 100 meters** when special insert will be built and installed
10 METERS OF GROWN R2R IJP YBCO$^{\text{LF}}$ ON SUPEROX SUBSTRATE

- Full conversion
- c-axis orientation predominates
- Silver deposition and oxygenation in progress
Transfer to OXOLUTIA: deposition of thick CSD nanocomposites

Ink jet printing colloidal solution + UV curing

Single deposition by ink jet printing

Low F solution (80% reduction)

1 μm grown film with 1 deposition

1.6 μm grown film with 2 depositions

10x10 mm² substrate

IJP nanocomposites 12%
IJP pristine
Spin pristine

3 MA/cm²

Courtesy of Prof. Teresa Puig
Transfer to OXOLUTIA:
Increase of $I_c$ with thickness by IJP deposition of nanocomposites

$I_c(5K, 9T) \times 12$

$I_c(77K, 3T) \times 12$

Courtesy of Prof. Teresa Puig
New production site
-5 km away from Scientific Park of UAB
-500 m²
-Inauguration: 25th September 2017
Acknowledgements

Partners and collaborators:

Projects:
THANKS FOR YOUR ATTENTION!

www.oxolutia.com
Chemical Solution Deposition scalability processes at Oxolutia

Reel-to-reel Ink Jet Printing pilot plant for all CSD Bruker & SuperOx substrates

Homogeneous single pass 100 m SDP layer @ 35 m/h

10 m CeO$_2$ (001)-oriented buffer @ 28 m/h

10 m of YBCO
COATED CONDUCTORS BY R2R INKJET PRINTING ON IBAD/ABAD TEMPLATES

PLANARIZING CHEMICAL LAYERS FOR $^{\text{ABAD}}$YSZ

Ce$_{0.90}$Zr$_{0.10}$O$_x$ CAP LAYERS

10 meters of (001)-oriented (Ce,Zr)O$_2$ buffer printed at 28 m/h

40 meters of nanocrystalline Y$_2$O$_3$ buffer printed at 30 m/h on unpolished stainless steel
The superconducting properties of the sample from the end of the pyrolysed 10 m decreases.

<table>
<thead>
<tr>
<th>Sample’s name</th>
<th>Ic (A/cm-w)</th>
<th>Tc onset (K)</th>
<th>ΔTc (K)</th>
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<tbody>
<tr>
<td>Y285_1K_P</td>
<td>21</td>
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<tr>
<td>Y285_1K_F</td>
<td>12.3</td>
<td>86.8</td>
<td>19.3</td>
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