Project acronym: HIPERCHEM

Project full name: High Performance Nanostructured Coated Conductors by Chemical Processing

Project no. 516858

Instrument: NMP

Final Publishable Activity Report

Period covered: from 01/04/2005 to 31/12/2008 Date of preparation: April 2009

Start date of project: 01/04/2005 Duration: 45 Months

Project coordinator name: Professor Xavier Obradors

Project coordinator organisation name: Consejo Superior de Investigaciones Scientificas

Revision [01] 10/04/09

SECTION 1: PROJECT EXECUTION

Summary of Project Objectives:

The overall objective of the HIPERCHEM project was to develop radically innovative low cost technologies for the mass production of nano-structured high temperature superconducting materials based on chemical solution processing. Within this envelope the development of novel nano-structuring methodologies for the growth of epitaxial nanocomposite films and coated conductors with excellent performance were targeted. The originality of the project lay in the fact that two rapid growth rate chemical processing techniques (metal-organic decomposition and hybrid liquid phase epitaxy) were combined to grow epitaxial $REBa_2Cu_3O_7$ high-temperature superconductor films with bottom-up nano-structuring strategies. The purpose was to achieve an artificial network of nano-defects that would immobilize the superconducting vortices and, hence, allow the achievement of high critical currents and give weak magnetic field dependence in films and coated conductors with high thickness.

The first general strategy for nano-structuring the superconducting layers was based on the generation of coherent randomly distributed nano-structures, such as nano-dots. The second strategy was to engineer nanostructures originating at the substrate interface, either based on strain-induced self-assembling principles or on polymer track-etched cylindrical nanopores generated by ion bombardment. The final technological goal was to develop nanostructured coated conductors with a total critical current of 400 A in a 1 cm wide tape and to achieve a reduction in its thickness and magnetic field dependence by a factor of 3 compared to the state of the art.

The specific scientific and technological objectives of the HIPERCHEM project were:

- To provide “proof of concept” that nano-structuring epitaxial films will lead to conductors that carry a total current of 400 A in a 1 cm wide tape and display a reduction in thickness and magnetic field dependence by a factor of 3 compared to the present state of the art.
- To provide “proof of concept” that the chemical processing approaches of MOD and HLPE can produce state of the art CC´s which will be compatible with bottom-up nano-structuring techniques, at growth rates 5 times larger (2 nm/s) than the state of the art and at low cost.
- To select the most promising nanostructuring approaches and demonstrate the feasibility of implementation on a pilot scale by making at least 1 m of CC.
- To protect the new inventions by patents (at least 2) and publish results in scientific journals (at least 12 joint publications)

Contractors Involved

The HIPERCHEM consortium consisted of six contractors from five member states:

Consejo Superior de Investigaciones Scientificas, Institut de Cienca de Materials de Barcelona, Spain {CSIC-IMCAB}

Professor X Obradors (the Co-ordinator): www.icmab.es

The University of Cambridge, Cambridge, UK {UCAM}

Professor J Driscoll: www.msm.cam.ac.uk

The Leibniz Institute for Solid state and Materials Research, Dresden, Germany {IFW}

Dr B. Holazapfel: www.ifw-dresden.de

Nexans SuperConductors GmbH, Hurth, Germany {NSC}

Dr J Bock: www.nexans.de

The Technical University of Vienna, Vienna, Austria {TUW}

Professor H Weber: www.ati.ac.at

The Université Catholique de Louvain, Louvain-la-Neuve, Belgum {UCL}

Dr E. Ferain: www.poly.ucl.ac.be

High Performance Nanostructured Coated Conductors by Chemical Processing (HIPERCHEM)

INTRODUCTION

THE overall objective of the HIPERCHEM project was to develop radically innovative low cost technologies for the mass production of nano-structured high temperature superconducting materials based on chemical solution processing. Within this envelope the development of novel nano-structuring methodologies for the growth of epitaxial nanocomposite films and coated conductors with excellent performance were targeted. The originality of the project lay in the fact that two rapid growth rate chemical processing techniques (metal-organic decomposition (MOD) and hybrid liquid phase epitaxy (HLPE) were combined to grow epitaxial $YBa_2Cu_3O_7$ (YBCO) high-temperature superconductor films with bottom-up nano-structuring strategies. The purpose was to achieve an artificial network of nano-defects that would immobilize the superconducting vortices and, hence, allow the achievement of high critical currents and give weak magnetic field dependence in films and coated conductors with high thickness.

The targeted future applications of this new type of advanced conductors are schematically indicated in Figure 1.

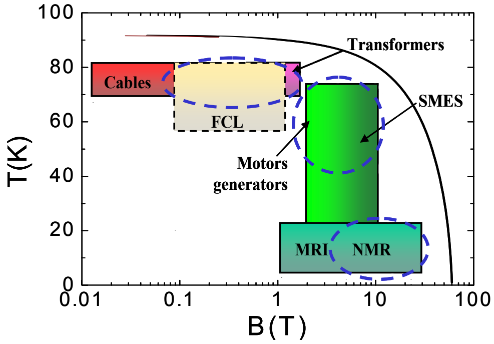


Figure 1 – Situation of the required working regions of the different targeted applications within the magnetic field – temperature phase diagram. The upper limit of the irreversibility line of YBCO is indicated as a continuous line.

The first general strategy for nano-structuring the superconducting layers was based on the generation of coherent randomly distributed nano-structures, such as nano-dots. The second strategy was to engineer nanostructures originating at the substrate interface, either based on strain-induced self-assembling principles or on polymer track-etched cylindrical nanopores generated by ion bombardment. The final technological goal was to develop nanostructured coated conductors with a total critical current of 400 A in a 1 cm wide tape and to achieve a reduction in its thickness and magnetic field dependence by a factor of 3 compared to the state of the art.

The consortium consisted of six partners from five EU Member States: three universities (University of Cambridge, UK; Catholique University of Louvain-la-Neuve, B and Vienna University of Technology, A), two research institutes (Institut de Ciència de Materials de Barcelona, Consejo Superior de Investigaciones Cientificas, E), and the Leibniz-Institut für Festkörper– und Werkstoffforschung Dresden, D) and the industrial company Nexans Superconductors GmbH, D. CSIC-ICMAB was the project co-ordinator under the leadership of Professor X. Obradors.

The work plan was divided into five technical work packages:

WP1 - Innovative Chemical Processing;
WP2 - Nanostructured Templates,
WP3 - Nanostructured REBCO films,
WP4 - Nanostructure and Vortex Pinning,
WP5 - Flexible Nanostructured Conductors

METHODOLOGIES USED

YBCO films were prepared by MOD following the trifluoroacetates route by using anhydrous precursor solutions. Deposition was carried out by spin coating or dip coating. After deposition, films were calcined in fast pyrolysis processes. Subsequently films were grown in wet atmosphere under controlled atmospheres of oxygen. The thickness of the grown films was determined by profilometer measurements. HLPE films were carried out using PLD as deposition method for a continuous feeding. Buffer layers were grown by MOD of carboxilate precursors which were deposited by spin or dip coating. Accurate control of the solution concentration and deposition conditions allowed to control the film thickness in the nanometric range. The buffer layers were grown under controlled oxygen partial pressure at temperatures in the range

900ºC - 1100 ºC. Ultradiluted solutions were used to prepare self-assembled nanostructures. Oxide and metallic interfacial nanotemplates were prepared using polycarbonate (PC) films irradiated with high energy ions and chemically etched (TEP) as chemical reactors filled with MO solutions or by electrodeposition when the buffer layer was metallic.

TGA-DTA and FTIR were used to analyze the MO decomposition process. RHEED and EBSD were used to analyze the surface crystallinity of buffer layer films and the grain size and structure of metallic substrates and buffers grown on top of them. Microstructural analyses were carried out based on TEM-EDX, EELS and SEM investigations. FIB was used for transverse microstructural analysis and XPS and RBS for the surface chemical properties and crystallinity of buffer layers.

Surface topography of the different substrates and buffer layers was characterized by atomic force microscopy. Epitaxial growth of the YBCO and buffer layer films was characterized by x-ray diffraction. A 2D area detector was used in some cases. Pole figures and ϕ-scans were performed systematically while strain analyses were also carried out.

Critical current density of the YBCO films were measured both inductively and by transport methods. Magnetic measurements were performed in SQUID magnetometers up to applied fields of 7T. Transport experiments were performed on bare films and conductors and in lithographically patterned films. Angular resolved measurements were performed in applied magnetic fields up to 9T. MFM at low temperatures were carried out to visualize vortices while a magnetoscan system based on Hall probes and permanent magnets was used for magnetic imaging at larger scales. Computer simulation allowed to determine from magnetic profiles the distribution of critical currents. Neutron irradiation was used in some cases to generate defects on the films for vortex pinning enhancement.

ACHIEVEMENTS COMPARED TO THE STATE OF THE ART

A) Nanostructured templates

There is a large interest worldwide to develop methodologies for large scale preparation of nanostructured templates and the use of chemical solution deposition for that purpose is only at its infancy. Within the scope of HIPERCHEM the interest on nanostructured templates arise from their potential capability to induce specific and controlled defects to pin vortices in superconducting films grown on top of them. Two general approaches were investigated: 1/ Self-assembling and self-organization of nanostructures generated mainly from ultradiluted chemical solutions, but also from cluster deposition methods; 2/ Nanostructures grown from chemical solutions or electrodeposition within track etched polymeric (TEP) nanoreactors. The first outstanding achievement in this field was the generation of self-organized nanodots of CeO_2 on vicinal perovskite substrates (Figure 2). A very strong terrace confinement effect was demonstrated between one single unit cell lattice steps arising from the strong interfacial energy of dissimilar structures [1]. The same compound was also generated as self-organized nanowires when a (011) orientation was generated (Figure 3(a)). This is because the inherent elastic anisotropy of this orientation stabilizes such elongated structures. Shape ratios up to ~100 and lengths in the range of 2 μm where demonstrated with annealing times in the range of 30 min which brings a very remarkable effective ultrafast growth rate of ~60 nm/min [2].

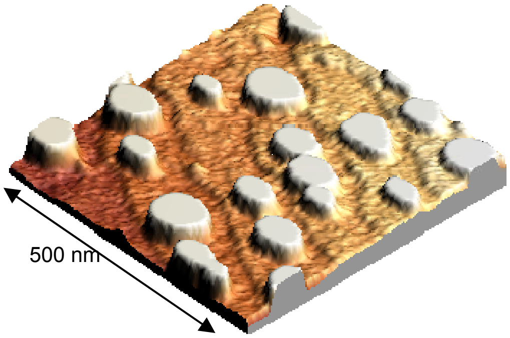
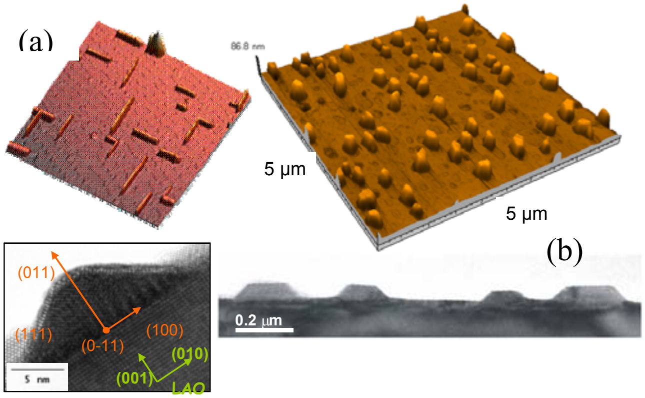
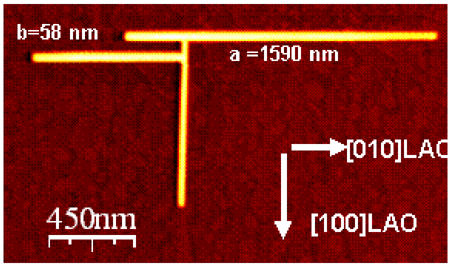
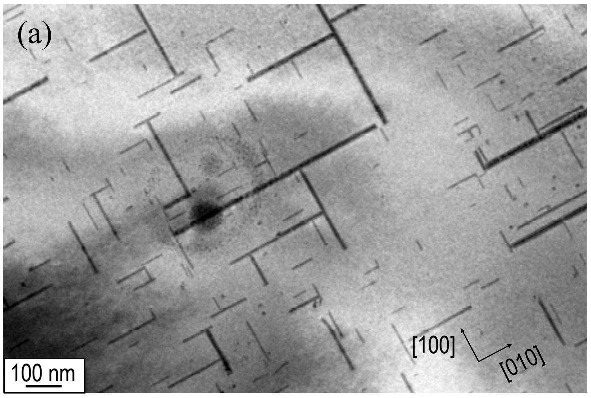


Figure 2 – Self-organized nanostructures grown by CSD on top of single crystals. AFM image of $Ce_{1-x}Gd_xO_{2-y}$ nanodots confined in the center of the terraces of a vicinal substrate having single unit cell steps.



(a)





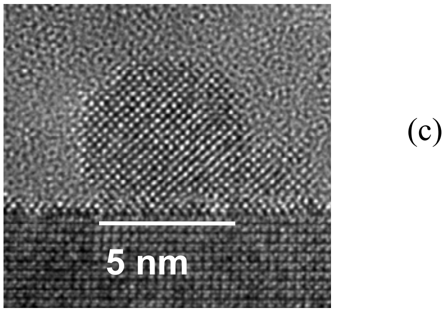


Figure 3 – Self-assembled nanostructures grown by CSD on top of single crystals. Left: AFM and TEM cross section images of $Ce_{1-x}Gd_xO_{2-y}$ nanowires. Planar view of the nanowires networks of CGO. Right: AFM and TEM cross section images of $(La,Sr)_2O_3$ nanodots outcrept on top of $La_{1-x}Sr_xMnO_3$ epitaxial thin films. Lower image: TEM cross section of a $BaZrO_3$ nanodot grown on top of a STO substrate.

Other alternative interfacial nanodots were also generated, such as $(La,Sr)_xO_y$ on top of epitaxial $La_{1-x}Sr_xMnO_3$ (LSMO) thin films (Figure 3(b)) [3] and $BaZrO_3$ (Figure 3(c)) on top of other perovskite substrates [4]. The establishment of the principles for the production of a wide range of interfacial nanostructures are definitively a very appealing output of this research. While using chemical solutions leads to strain engineered epitaxial nanostructures, using a metal beam cluster deposition technique allowed to generate Y_2O_3 nanodots having a random orientation as they nucleate homogeneously before impinging in the substrate (Figure 4). This achievement allowed to study the influence of crystalline orientation on the inducement of defects on films grown over the nanotemplates.

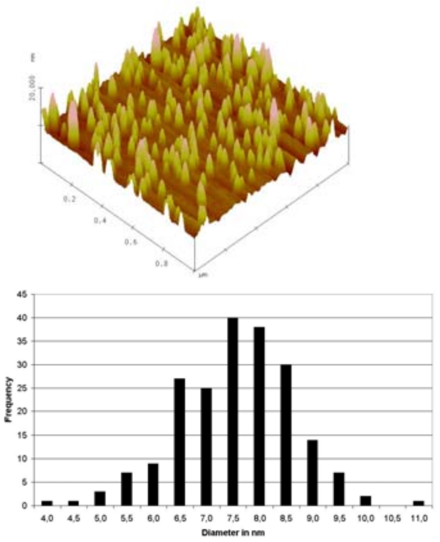


Figure 4 –AFM image of Y_2O_3 nanodots deposited on a STO substrate by means of a metal beam cluster technique. Nanodot size distribution determined from TEM images of nanodots deposited on a grid for direct observation.

The achievement of compatibility between the polymeric track-etched nanoreactors and chemical solutions was a real outstanding output of the project. For the first time single crystalline oxide nanowires were achieved through this methodology when self-sustained polycarbonate (PC) membranes were used [5]. Ferromagnetic manganites (LSMO) were selected as material (Figure 5). In this case even more outstanding results were achieved when the nanostructured transformations of the LSMO nanorod were investigated. While growth at low temperatures lead to polycrystalline nanorods, further high temperature annealing results in a full transformation of the nanostructure. The polycrystalline nanorod turned out into a LSMO wetting layer on the whole substrate and $(La,Sr)_xO_y$ nanopyramids remained at the same position of the track etched nanopores (Figure 6). The driving force for such a drastic transformation was associated to the surface, interfacial and elastic energies trade-off but the crucial role of the short dimension to achieve fast kinetics for phase separation and recrystallization should also be stressed.

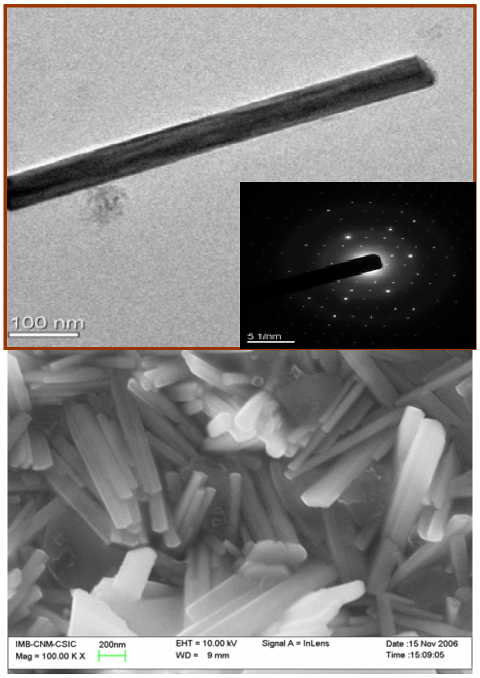


Figure 5 – Lower SEM image: Single crystalline nanowires of $La_{1-x}Sr_xMnO_3$ grown by CSD using self-sustained PC after a TEP process. Upper image: TEM and electron diffraction of a single nanowire showing the single crystalline character. A new monoclinic structure has been obtained through this procedure.

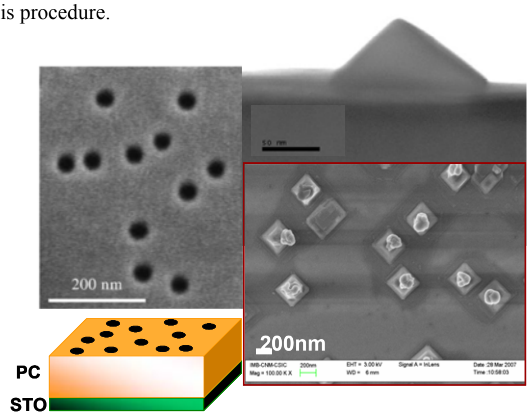


Figure 6 – Left: Track etch nanopores generated on polycarbonate films deposited on top of $SrTiO_3$ single crystals. Right: Nanostructures grown by CSD using the TEP as nanoreactors. A wetting layer of epitaxial $La_{1-x}Sr_xMnO_3$ layer is obtained with $(La,Sr)_2O_3$ nanopyramids growing on the position of the etched nanopores.

A second successful approach to the achievement of interfacial nanostructures was that based on electrodeposition. Here the key issue was the development of the TEP methodology on top of metallic oxide buffers such as $SrRuO_3$. Polycrystalline Ag nanowires were successfully deposited in that case and hence the capability of the combined methodologies demonstrated (Figure 7).

Summarizing, the effort devoted to the generation of interfacial nanotemplate growth through low-cost, large-scale methodologies was extremely successful and several novel methodologies were confirmed for being used in combination of superconducting layers. Additionally, new functionalities are expected for these oxide and metallic nanotemplates.

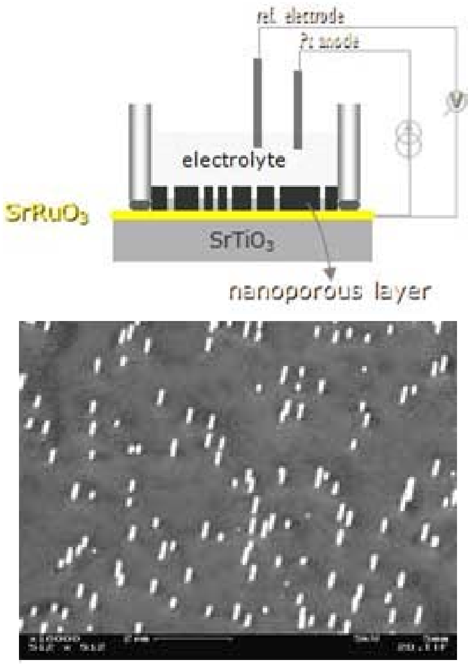


Figure 7 – Schematic of the electrodeposition process and architecture used for growing supported nanorods on the surface of a conductive buffer layer. SEM image of Ag nanorods obtained with this technique.

B) Innovative chemical processing and nanocomposites

Achieving a detailed understanding of the two chemical methodologies for superconducting film growth, i.e. MOD and HLPE, was one of the most demanding objectives in the initial stages of the project. Many varied advances going well beyond the international state of the art were accomplished.

In the case of YBCO film growth by MOD, Trifluoroacetate (TFA) metal-organic precursors were used and, through accurate analysis of the solution purity and of the thermomechanical transformations associated to the deposition and pyrolysis processes, a remarkable reduction of the processing time was achieved (less than 1/10th of the previous processes) [6-9]. Through advanced characterization techniques, such as transversal FIB observation, the mechanisms controlling the formation of porosity could be analyzed (Figure 8) and processing modifications to eliminate pore concentration were defined. It was also particularly useful to analyze the film formation by FTIR, TGA, nanoindentation and mass spectroscopy of the exhaust gases. A very precise identification of the relevant processes generated an extremely useful know-how afterwards being used for the preparation of complex nanocomposite films. An additional accurate analysis of the intermediate microstructural evolution of the films before the high temperature formation of the epitaxial films was carried out by TEM [10]. Particularly the oxide and fluoride nanocrystalline or amorphous phases were identified and the reaction mechanisms was described (Figure 9).

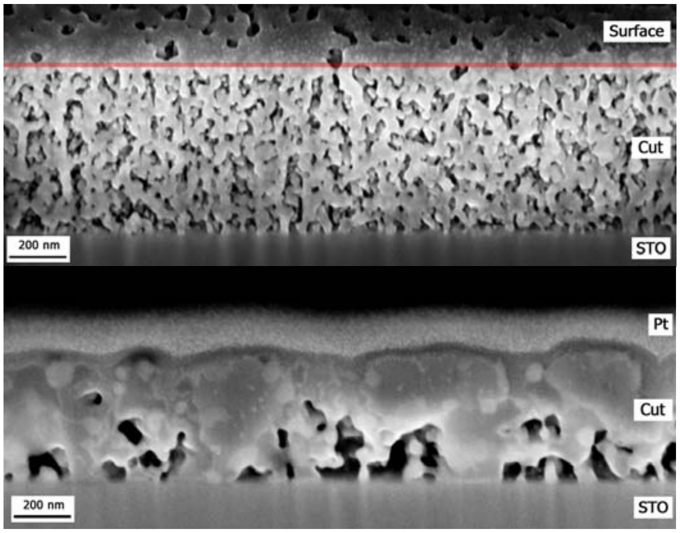


Figure 8 - FIB cross sections of a TFA-YBCO film at different stages of the evolution of the amorphous and nanocrystalline oxide and fluoride precursors, The strong porosity achieved after the pyrolysis is already strongly healed out during the heating process and it is finally practically eliminated after epitaxial growth.

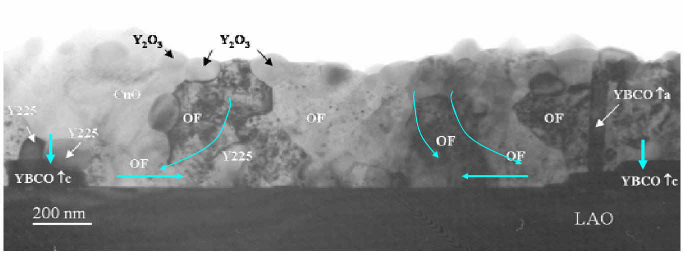


Figure 9 - TEM cross section of a TFA-YBCO film quenched from the nucleation stage. The first YBCO nuclei are already observed at the interface while the different phases defining the growth process and the required diffusion are observed.

Only after a deep understanding of the complex microstructural transformations, and of the processing issues controlling them, was it possible to achieve two very demanding goals of the project: to grow epitaxial films with thickness beyond 1 μm and growing them at rates in excess of ~1 nm/s. For the first objective it required the use of compatible polymeric additives while the second objective was achieved through the development of a low pressure growth methodology allowing to increase the gas exhaust diffusion and hence accelerating the chemical transformation towards YBCO. All these achievements placed the

HIPERCHEM consortium in a competitive position to face the most exciting breakthrough of the project: the development of a chemical methodology for the preparation of nanostructured superconductors.

But if MOD developments achieved a worldwide competitive position, those associated to HLPE growth of YBCO films jumped the consortium in a unique position where practically no worldwide competition exists [11-13]. This technique combines a vapor deposition step (achieved with PLD but also demonstrated with spraying of chemical solutions) with the formation of a thin nanometric liquid layer allowing to achieve extremely high growth rates (~10 nm/s growth rates were demonstrated) and thus very thick films (2-3 μm) could be grown in very short times (Figure 10). The highly advanced mastering degree achieved of the HLPE technique allowed later to face very successfully the challenge of controlling the formation of a precise nanostructure in composite films.

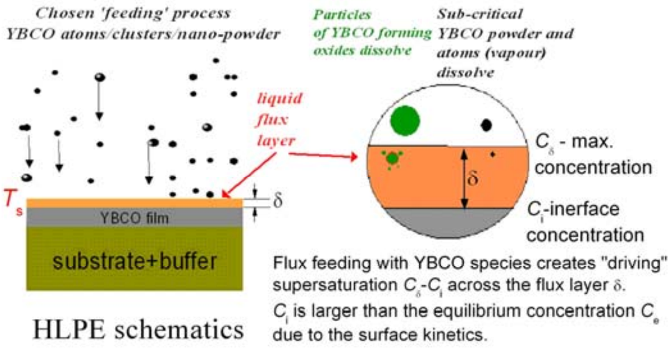


Figure 10 - Schematics of the HLPE growth process.

A successful technology for coated conductors, however, relies not only on having reliable methodologies for superconducting layers growth, it also requires actually to achieve multilayered structures with buffer layers protecting the metallic substrates and transferring the biaxial texture to the superconducting layers. The HIPERCHEM consortium therefore devoted a considerable effort to sort out how dense and highly epitaxial oxide layers could be grown by MOD and how a high quality surface finishing with atomic planarity could be achieved [14-16]. The issue of achieving a high percentage of the film surface displaying atomic planarity flatness, i.e. roughness below ~ 2 unit cells, was found to be highly determinant of the multilayer epitaxial quality and hence of the superconducting performances (Figure 11). It was proposed actually that the atomically flat terraces of the cap layers behave as the most relevant sites for promoting a high density of YBCO nucleation sites which then lead to very dense films without current blocking defects. This development allowed to achieve a worldwide record value for J_c for an "all chemical" multilayer (Figure 12), and established a working basis for the goal of manufacturing high critical current coated conductors [17-19].

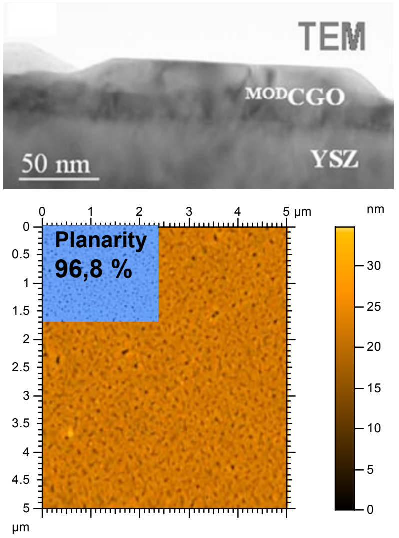


Figure 11 - TEM cross section of a CGO film grown by MOD where the terraced surface is clearly appreciated. AFM image of the surface of a $(Ce,Zr)O_2$ buffer layer growth by MOD on top of a YSZ single crystal. The binarization of the image allows the surface planarization (mask of 1.5nm) to be determined quantitatively.

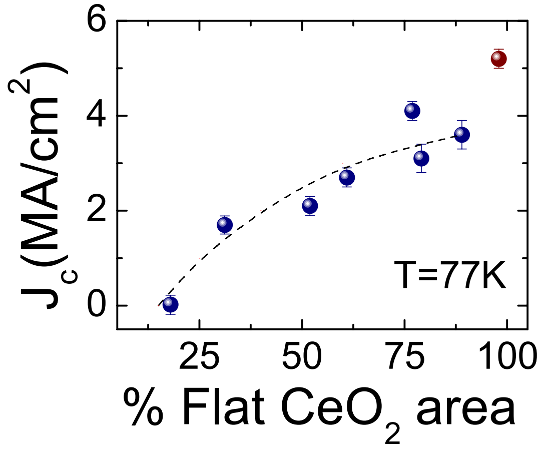


Figure 12 - Dependence of the self field J_c measured at 77 K of TFA-YBCO films grown on top of CeO_2 buffer layers having different degrees of surface planarization A record value of J_c is achieved in the limit of 100 % planarity.

Generating new reliable multi-scale methodologies for analysis of the superconducting properties of films and tapes was considered as a very relevant issue within the consortia, mainly taking into account the microstructural complexity of the developed multilayered films and conductors. Two experimental methodologies are worth of being mentioned as outstanding outputs of the project: 1/ A general approach to the generation of vortex pinning magnetic phase diagrams in superconductors based on temperature and magnetic field dependent critical current measurements; 2/ An inductive fast methodology having enough spatial resolution to analyze the homogeneity of the critical current density in tapes, the magnetoscan technique. Figure 13 shows a typical set of anisotropic $J_c(\theta,T,H)$ measurements from which, through a scaling analysis, the isotropic pinning contribution (in the sense that the corresponding defects do not modify the intrinsic anisotropy of YBCO) is determined and hence the anisotropic contributions sorted out, both of them as a function of temperature and magnetic field [2, 20-22]. The temperature dependence of J_c then allows the contribution arising from weak-isotropic pinning centers and that arising from strong (isotropic and anisotropic) pinning centers (Figure 14) to be determined. The final output of this analysis results in a single phase diagram such as that reported in Figure 15. These diagrams have therefore become an extremely useful instrument to visualize the influence of any approach to the modification of pinning through modified nanostructures. A wide use of this instrument was made during the whole project, as it will be later reported.

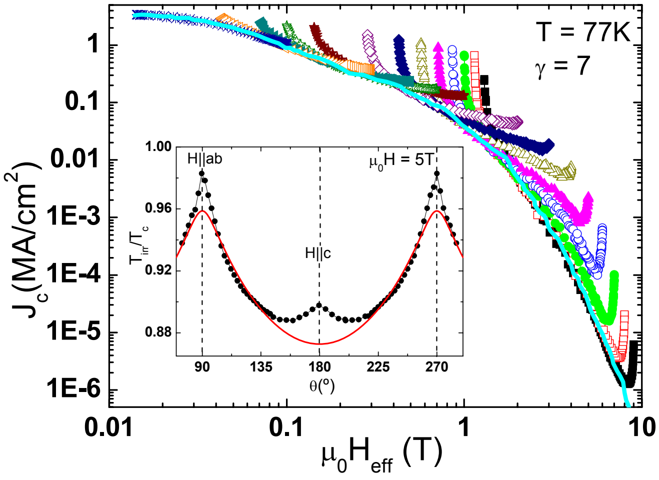


Figure 13 - Inset: anisotropic critical current measured at 77 K at 5 T for the standard TFA-YBCO film. The red continuous curve indicates the isotropic contribution to J_c. Main panel: $J_c(H_{eff})$ dependence for the anisotropic curves measured at different magnetic fields from which the collapsing curve indicates the isotropic contribution and the intrinsic anisotropy is determined.

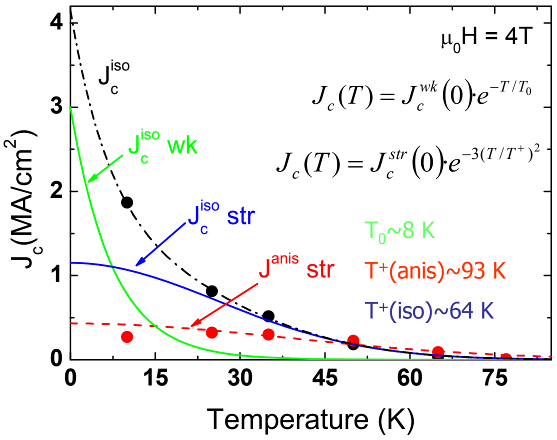


Figure 14 - Temperature dependences of the three contributions considered to the total $J_c(T)$ of a standard TFA-YBCO film measured at 4 T: Weak – isotropic, strong – anisotropic and strong isotropic. The fitted laws for weak and strong temperature dependences are included in the inset.

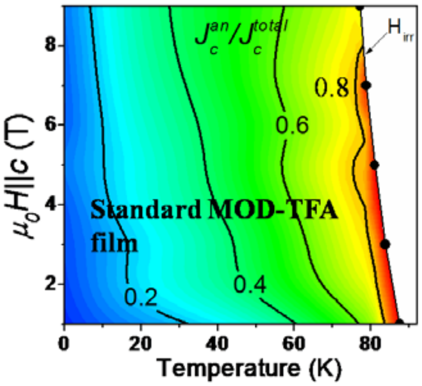


Figure 15 - Strength pinning phase diagram for the standard TFA-YBCO film where color indicates percentage of strong pinning contribution over total measured J_c.

The magnetoscan system is a simple but very ingenious device associating Hall sensors with permanent magnets scanning all together over the films or conductors to be analyzed [23,24]. This allows mapping of the trapped magnetic field distribution and then, through electromagnetic modelling of the process, the persistent current distribution determined (Figure 16). The device has become a fast tracking system of any microstructural inhomogeneity with an improved resolution over existing imaging systems. Although the resolution is not high enough to discern the most relevant inhomogeneity of coated conductors, i.e. grain boundaries (which may be discerned through combined magnetooptic and EBSD measurements), other current blocking defects such as growth instabilities or substrate inhomogeneities could be detected and hence a large use of the device was made to optimize conductor processing. Magnetic imaging at the nanoscale was also demonstrated through the use of MFM (Figure 17), however it has still not been possible to correlate vortex visualization with specific artificially induced pinning centers.

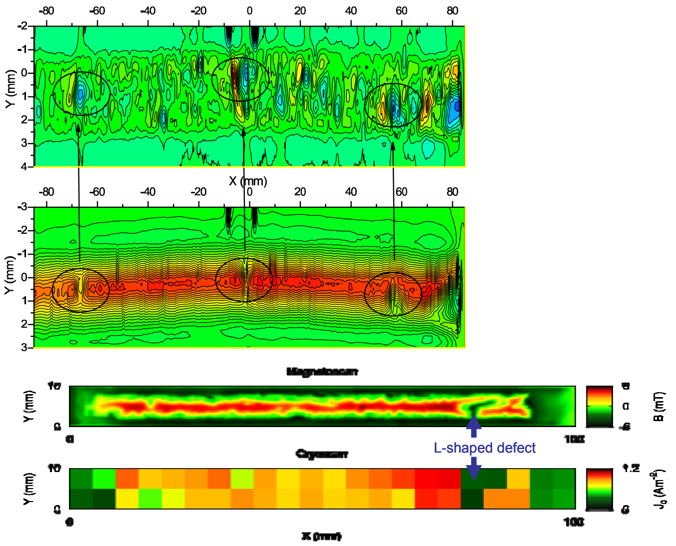


Figure 16 - Upper images: Magnetoscan of a 17 cm long tape where constrictions limiting the current flow are indicated by circles. Lower panel: comparison of the resolution achieved to detect a defect with the magnetoscan and a commercial inductive “cryoscan” system.

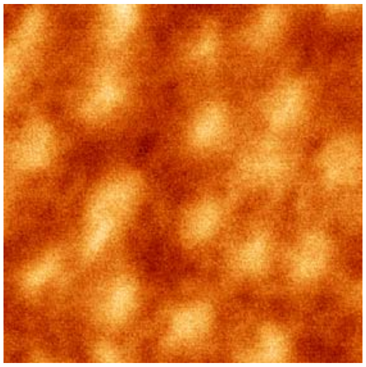


Figure 17 - 4 x 4 µm² MFM image of an off-axis PLD YBCO film cooled down up to the temperature of about 7.7 K in a magnetic field of 3 mT

Before facing the problem of generating artificial pinning centers (APC) it is necessary to understand properly the natural defects appearing in YBCO films grown by MOD and HLPE. This task devoted a considerable effort due to the complex microstructure of these materials where the defects generation mechanisms are strongly linked to processing and, additionally, there is a strong interrelationship among them, i.e. they can not be faced as a simple superposition problem (Figure 18) [25-30]. This analysis was complemented with analysis of the influence of neutron irradiation on the critical currents [31].

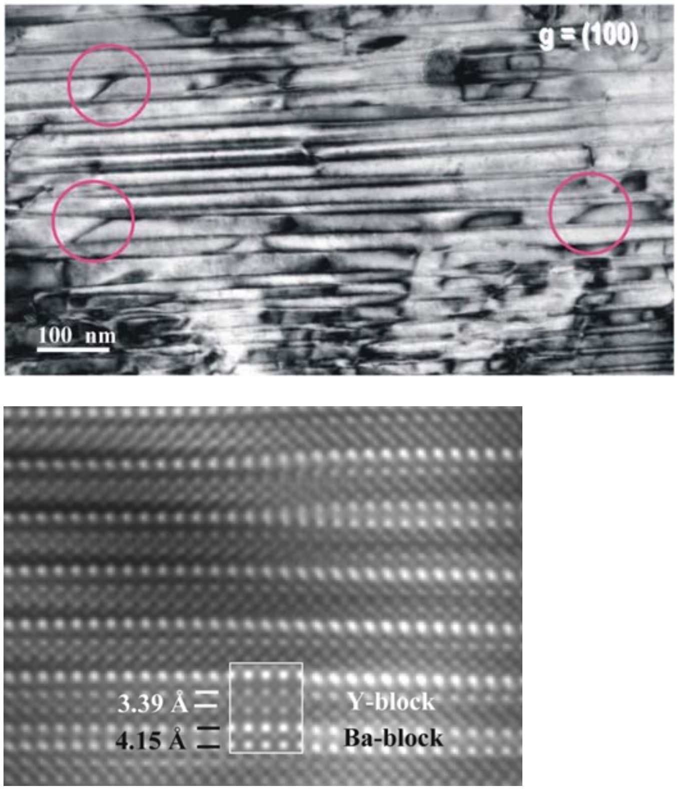


Figure 18 - Upper curve: TEM cross section of a TFA derived YBCO film exhibiting a high density of dislocations pinned at twin boundaries. Segments jumping from one boundary to the next are encircled. Lower curve: High resolution cross section TEM image a Ba-perovskite block intergrowth in the matrix of a TFA derived YBCO film. Inset is an image simulation of the intergrowth.

The first type of APC generated in YBCO films were those induced by interfacial nanotemplates. Many types of combinations of nanostructures and processing conditions were analyzed and, indeed, a strong influence of these nanotemplates on the microstructure and J_c were discerned. Figure 19 displays a typical TEM image where the YBCO matrix is seen to be strongly modified by an interfacial nanodot. Similar influences were made evident in other systems [2].

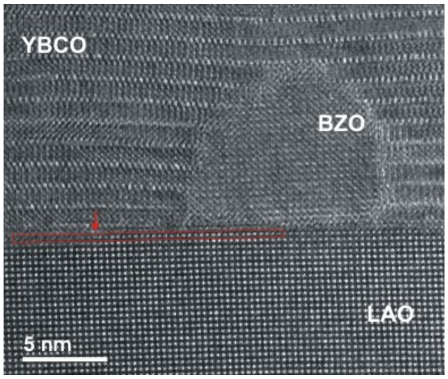


Figure 19 - TEM cross section of a YBCO-TFA film grown on top of a BZO nanotemplate. The influence of the nanodots on the YBCO film is clearly made visible through a strong plane bending and the strain generated on the YBCO matrix.

Figure 20, on the other hand, shows typical anisotropic critical current density measurements of a standard YBCO-TFA film compared with those measured in two YBCO samples grown on top of two different sorts of nanotemplates. As it is made clear, a clear increase of J_c is observed along the c-axis, which indicates that correlated disorder pinning along this direction has been enhanced. This effect is mainly evidenced at high temperatures and magnetic fields and it increases J_c at 7 T and 77 K up to 700 %. This is clearly evidenced by comparing the magnetic phase diagram with that of a standard YBCO sample (Figure 15 vs Figure 21). Note also that while pinning along c-axis is enhanced, that along the ab planes is reduced thus minimizing the overall anisotropy of the film.

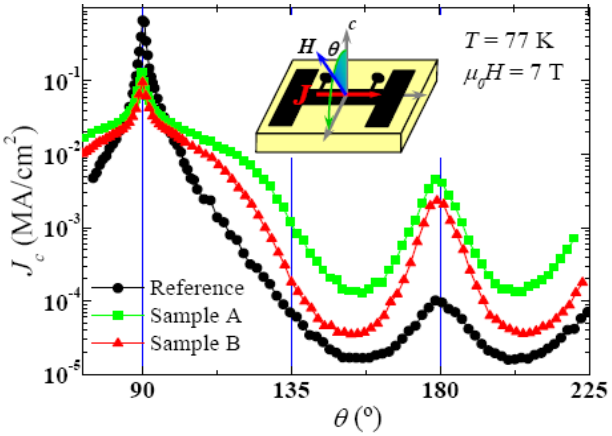


Figure 20 - J_c as a function of θ at 77 K and $\mu_0 H = 7$ T for (●) reference, (■) nanotemplate of CGO nanowires and (▲) nanotemplate of $(La,Sr)_2O_3$ nanodots outcrept on top of $La_{1-x}Sr_xMnO_3$ epitaxial thin films films. Inset shows a representation of the experimental geometry.

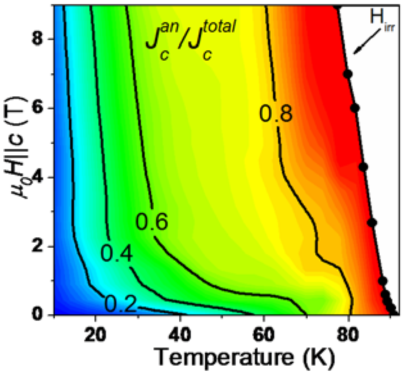


Figure 21 - Strength pinning phase diagram for a YBCO film with APC induced by an interfacial nanotemplate. The color indicates percentage of strong pinning contribution over total measured J_c. Compare with Figure 15 for a standard film.

An additional example of enhanced vortex pinning induced by nanotemplates is indicated in Figure 22 corresponding to YBCO grown by PLD or HLPE on top of the Y_2O_3 nanodots shown in Figure 4, or similar ones grown by PLD [32,33].

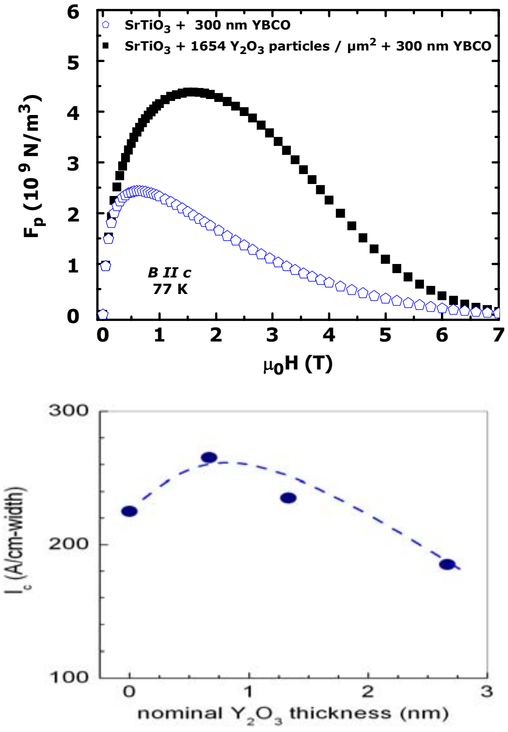


Figure 22 – Upper curve: Comparison of the pinning forces of YBCO films grown by PLD on top of a STO single crystal and of the Y_2O_3 nanotemplate indicated in Fig. 4. Lower curve: Total critical current of a HLPE film grown on top of a Y_2O_3 nanotemplate prepared by PLD and having different concentration of nanodots.

The largest effort and the most outstanding breakthroughs within the project were achieved within the scope of the development of nanocomposites YBCO films, i.e. with different approaches to introduce nanodots within the YBCO matrix which, as will be shown, they become very effective in enhancing vortex pinning. Nanocomposites were grown by the two chemical deposition techniques (MOD and HLPE) and also by PLD because different nanostructural development paths were evidenced in all the different techniques and so very useful complementary knowledge was generated by comparing the growth techniques.

Nanocomposite MOD films were grown only by the so called in-situ technique, i.e. complex metal-organic salts were prepared including the different elements in the concentration required for the final composition of the nanocomposite. The growth process was then tuned to generate two segregated phases. The first issue that is required to grow nanocomposites is to know if different crystallographic phases are immiscible. Several phases have been detected which spontaneously remain segregated, for instance $BaZrO_3$, $BaHfO_3$, Y_2O_3, Au, etc. [34-36,11].

Another fundamental issue is that related to the mutual crystallographic matching (YBCO vs nanodots). Of course this will strongly depend on how the nucleation and growth processes of both crystallographic phases occur. In MOD it was shown that YBCO/BZO nanocomposites are formed in a completely different disposition, as compared to those prepared by PLD [34,21]. Figure 23 shows a 2D XRD pattern of a typical nanocomposite where immediately is discerned that while the YBCO matrix remains epitaxial, the BZO nanodots are oriented at random (ring pattern). TEM analysis shown in Figures 24 and 25 confirm this conclusion. The $BaZrO_3$ or $BaHfO_3$ nanodots are distributed at random within the matrix and they are randomly oriented because they nucleate homogeneously before that YBCO nucleates heterogeneously at the interface. TEM images also show that the incoherent interface generated among the nanodots and the matrix has a strong influence on the YBCO matrix itself which appears deeply strained and defective.

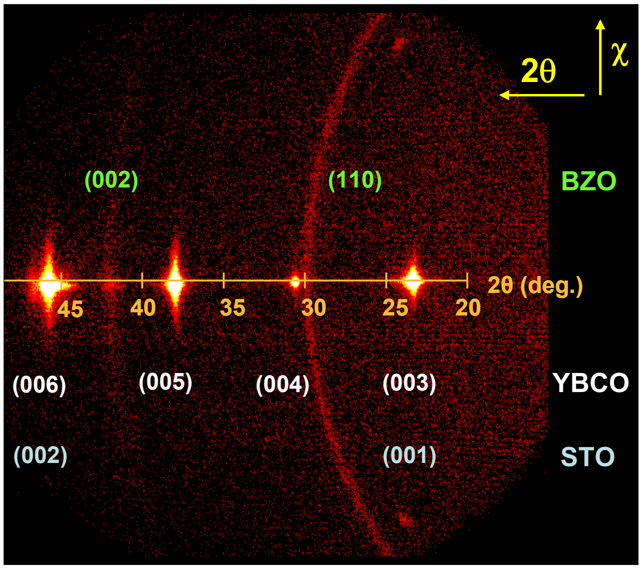


Figure 23 - X-ray diffraction patterns of a YBCO/BZO nanocomposite. The diffraction pattern was obtained with a GADDS equipped with a 2D X-ray detector. The x axis corresponds to 2θ and the rings correspond to χ which varies with constant 2θ. The Bragg peaks of the BZO and YBCO phases are identified. Note the coexistence of diffracted intensities for BZO corresponding to textured and randomly oriented nanodots.

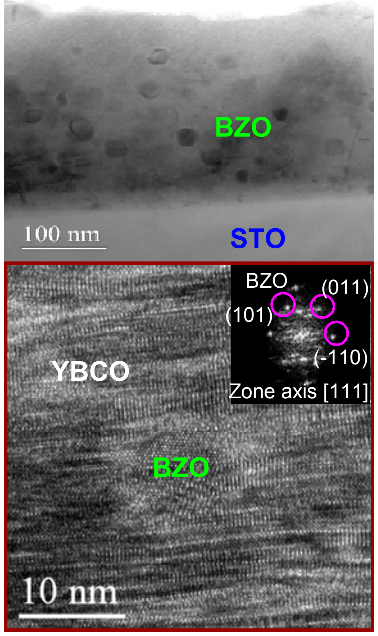


Figure 24 – Upper curve: Low-resolution cross-section TEM image of a nanocomposite film viewed along the [100] direction of YBCO where BZO nanodots are clearly discerned. Lower figure: HRTEM image of a BZO nanodot located at the bulk of the YBCO film where a highly defective matrix is discerned. Inset: FFT of the BZO image showing that the nanodots are not coherent with the YBCO matrix.

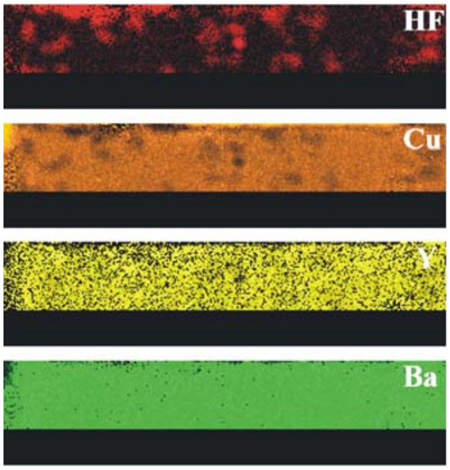


Figure 25 - TEM-EDX cross section image of a YBCO/BaHfO$_3$ nanocomposite prepared by MOD where the BaHfO$_3$ nanodots are easily identified distributed at random within the YBCO matrix.

The consequences of introducing nanodots in the YBCO matrix were immediately made evident through critical current density measurements. Figures 26 and 27 display J$_c$(H) and pinning force F$_p$ when H//c and the anisotropic behavior, respectively. Worldwide record values of J$_c$ and F$_p$ were achieved at very high magnetic fields (3-6 T) and at liquid nitrogen temperatures (T> 65 K) and the goals of the HIPERCHEM project were widely achieved. The achieved performances at such high temperatures overshadows the best performances of the present commercial NbTi low T$_c$ superconducting wires achieved at liquid He temperatures (Figure 26). An additional very beneficial feature of these superconducting nanocomposites is that the effective anisotropy is strongly reduced (Figure 27).

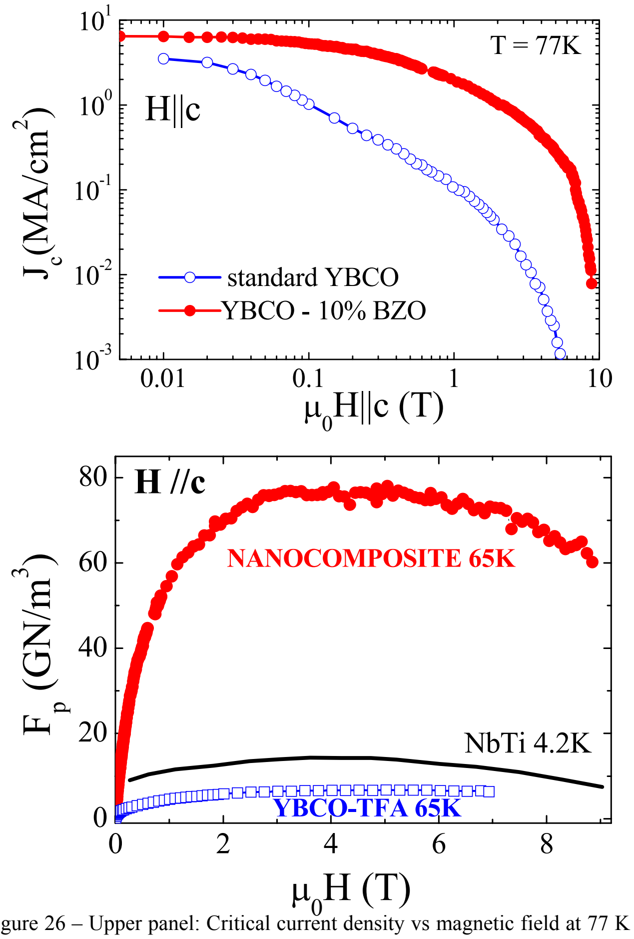


Figure 26 – Upper panel: Critical current density vs magnetic field at 77 K of a YBCO/BZO nanocompossite as compared to a standard TFA-YBCO film. J$_c$ is enhanced by a factor at T. Lower panel: Pinning force of a YBCO/BZO nanocomposite at 65 K, as compared to that measured in a standard TFA-YBCO film and a NbTi wire at 4.2 K.

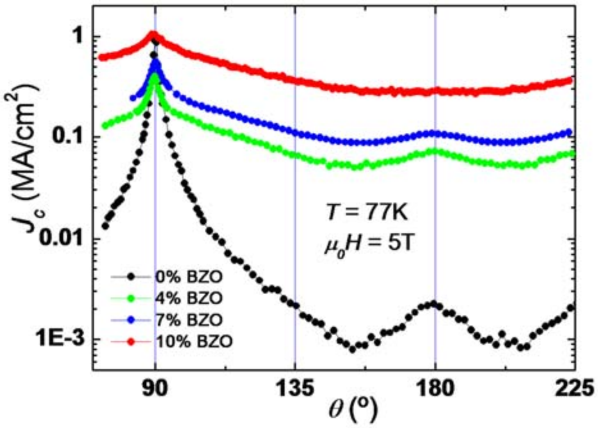


Figure 27 – Anisotropic critical current density measured at 77 K and 5 T for a set of nanocomposite films as compared to a standard TFA-YBCO film. Note that the anisotropy is strongly reduced in the nanocomposites.

A very thorough analysis of the nanostructure and the physical properties of such nanocomposites was carried out to identify the critical parameters responsible of the very beneficial effects of the new nanocomposite materials. Particularly, the magnetic flux pinning phase diagram immediately established that the observed breakthrough was actually due to an enormous enhancement of the effectiveness of the strong – isotropic contribution to vortex pinning (Figure 28).

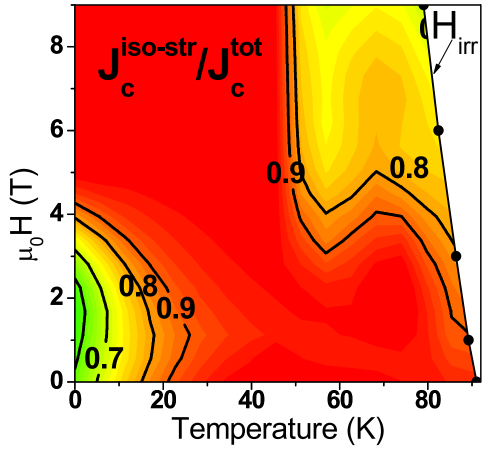


Figure 28 – Strength pinning phase diagram for a YBCO/BZO nanocomposite film with APC. The color indicates percentage of strong isotropic pinning contribution over total measured J_c. Compare with Figure 15 for a standard film. This is the first YBCO superconducting materials where the isotropic pinning contribution becomes dominant at high temperatures and magnetic fields.

The origin of this phenomenon is still not completely clarified but X-ray diffraction analysis of residual strain through the so-called Williamson Hall plots clearly showed that the BZO nanodots induce a strong enhancement of the rms strain within the YBCO matrix. This effect should be coupled with the strong enhancement of the incoherent interfacial energy which might be reduced through enhancing the defect concentration and deforming the lattice to a greater extent than a coherent interface where the influence is much more localized. Through an accurate tuning of the amount of non coherent interfaces, based on BZO and Y_2O_3 secondary phases, it could be demonstrated that the amount of randomly oriented nanodots controls the effective anisotropy and the pinning enhancement.

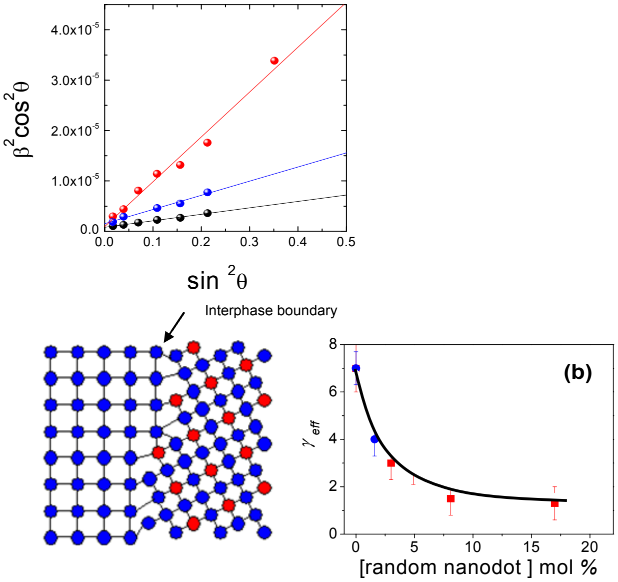


Figure 29 – Upper panel: Williamson Hall plots of two YBCO nanocomposites having 10 % mol of a secondary phase (red: BZO, blue: Y_2O_3), as compared to pure YBCO. The residual strain is determined by the slope and it is enhanced from 0.09 % to 0.25 %. Lower panel left: Schematic representation of a non-coherent interface where the strong disorder is clearly evidenced. Lower panel right: decrease of the mass anisotropy with the concentration of randomly oriented nanodots in the nanocomposites.

Other strategies were successfully deployed to generate nanostructured YBCO films with APC. Figure 30 shows, for instance a YBCO/Au nanocomposite film grown by HLPE following the indicated path [11]. Due to the fact that Au diffuses very fast within the YBCO matrix at high temperature, Au nanodots were introduced within a STO buffer layer and then a smoother introduction of the nanodots within the YBCO matrix was achieved with the flat nanodots remaining epitaxial and randomly distributed within the YBCO matrix (Figure 30). The effectiveness of this approach is clearly demonstrated through the observation of a higher continuous increase of the total critical current of YBCO films with thickness, as compared to HLPE films grown on conventional substrates (Figure 31).

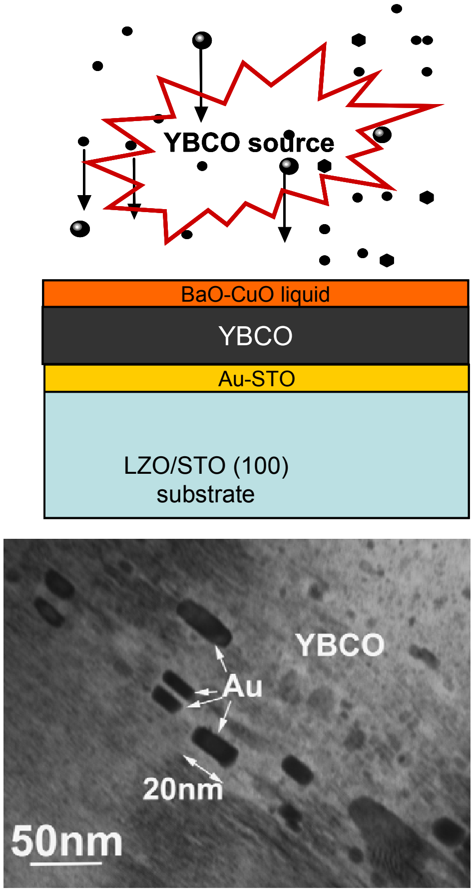


Figure 30 – Upper figure: Schema of the HLPE YBCO growth process on top of a LZO buffer layer covered by a STO-Au cap layer. Lower panel: Low resolution TEM cross section image where the flat Au nanodots are clearly discerned.

Novel and more complex YBCO nanocomposite nanostructures were also generated by means of PLD establishing the fundamentals of the strain nanoengineering required to control the structure of these compounds [28,29,33]. PLD can be carried out through a co-deposition route where the secondary phase nucleates on the YBCO growing interface and hence it becomes coherent or semicoherent, depending on the lattice misfit. Epitaxy is in this case three dimensional and hence, depending on the lattice misfit along c-axis and within the ab-plane, anisotropic strains can be generated. Also, kinetics may play a role on the self-organizing process. When kinetics is too fast the nanodots are randomly distributed but if thermodynamics controls the nanodots growth self-organized nanorods may be achieved. Figure 32 show a schematic representation of 3D epitaxy for a YBCO/Gd_3TaO_7 nanodot [36] together with TEM images of this pyrochlore structure and the perovskite-derived phase $Y_2Ba_4CuZrO_y$, as typical examples.

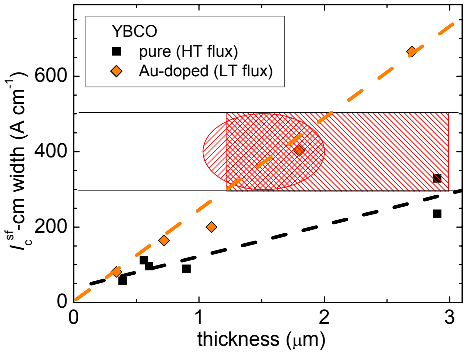


Figure 31 – Dependence of the self-field critical current density of HLPE films with their thickness. Two different slopes are shown corresponding to conventional YBCO films and YBCO/Au nanocomposites grown by means of the modified buffer layer technique.

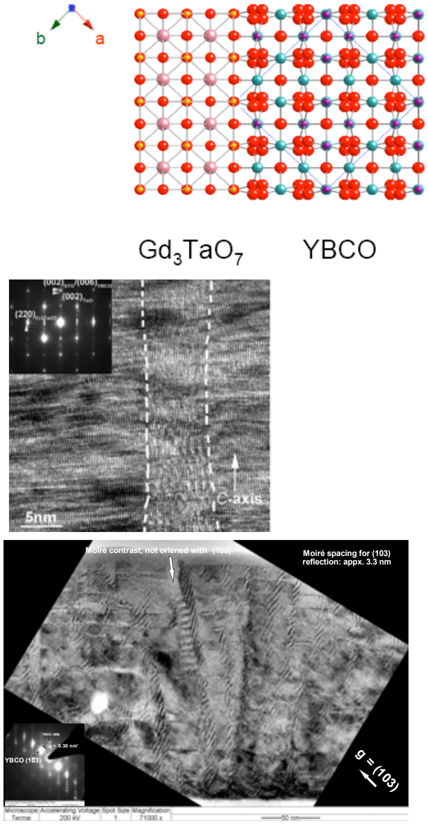


Figure 32 – Upper panel: Lattice matching of the YBCO structure with a RE_3TaO_7 pyrochlore where the lattice parameters can be tuned through the size of the RE ions. Middle panel: TEM image of a YBCO/Gd_3TaO_7 nanocomposite showing the formation of long columnar ordering of the secondary phase. Lower panel: TEM image of a YBCO/$Y_2Ba_4CuZrO_y$ nanocomposite quasi-multilayer showing the formation of different columnar ordering evidenced by the Moiré contrasts

The last example actually corresponds to a quasi-multilayer growth process allowing to enhance the degree of 2D order of the secondary phases through sequential deposition of a very thin film of the secondary phase and of YBCO. The degree of intermixing and the generated strain clearly controls the vortex pinning enhancement effect.

In conclusion, a very wide body of new knowledge was generated within the scope of HIPERCHEM concerning vortex pinning enhancement by APC which put the consortium in a very competitive position worldwide.

C) Flexible conductors

Several conductor architectures are promising alternatives for coated conductors but within the HIPERCHEM consortium the efforts were concentrated in those indicated in Figure 33. The final goal was to achieve an "all chemical" architecture with high performances, however, other deposition techniques, both for the buffer layers and the YBCO layers were used as intermediate steps towards optimization of the deposition and growth methodologies. The most investigated cap layer was doped CeO_2, although growth directly of YBCO on LZO cap layers was also investigated. Also other buffer layers architectures were sometimes investigated, such as those where the seed layers on NiW were perovskites ($CaTiO_3$) or Y_2O_3.

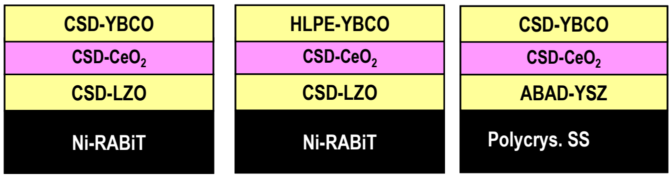


Figure 33 – Main conductor architectures investigated within the project. The NiW RABiT substrates were coated first with MOD-LZO buffer layers and long lengths of these flexible metallic substrates were achieved. Growth on top these substrates was either continued with MOD-CeO_2 cap layers and TFA-YBCO films or with HLPE YBCO films. PLD or other vacuum deposition methods were also explored as intermediate strategies towards the "all chemical" low cost conductors.

The use of polycrystalline stainless steel substrates covered with biaxially textured YSZ layers by means of Alternating Beam Assisted Deposition (ABAD) by Bruker HTS GmbH appeared as a very suitable flexible metallic substrate where the multilayered MOD structure previously developed in single crystals could be transferred. The great advantage of this metallic substrate is the resistance to high temperature anneal under oxidative atmospheres. For that reason a high surface planarity was achieved in a $Ce_{1-x}Zr_xO_2$ (CZO) MOD cap layer (Figure 34). This allowed to achieve high quality TFA-YBCO films on top of it (Figure 35) and hence high critical current self-field densities J_c=1.6 MA/cm^2 (percolative currents) were measured while the typical features of granular conductors were still made visible in magnetization hysteresis loops (Figure 36) [37,38]. The percolative J_c value is well below the record value of intragranular J_c achieved with single crystalline multilayers and hence it confirms that J_c(H) at self-field is still dominated by granularity effects, as demonstrated by the existence of a low magnetic field range where J_c is below that observed in single crystals while above a crossover field both values converge (Figure 35) [38-40]. The ^{ABAD}YSZ/SS substrates were also successfully used to grow YBCO/BZO nanocomposites and it was shown that the vortex pinning benefits were obtained, though further minimization of the granularity effects should be achieved (Figure 37) [41].

As a search for alternative conductor architectures, the Hiperchem consortium was successful in defining a new IBAD – based architecture where all the buffer layers grown on a Hastelloy substrate had a metallic character (Figure 38), an appealing feature for some specific applications such as Fault current limiters. RHEED analysis demonstrated that a biaxial texture was indeed achieved.

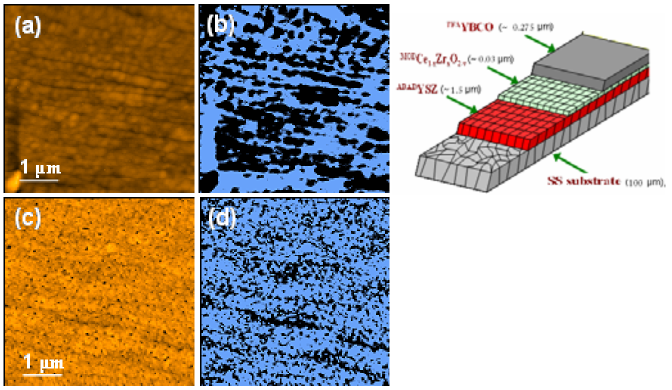


Figure 34 - In the left side are the AFM images of the surface of (a) an ^{ABAD}YSZ substrate as grown and (c) of the (Ce,Zr)O_2 buffer layer growth by MOD on top of it. The images in the right side correspond to their binarization after applying a planarizing mask of 1.5nm. As-grown ^{ABAD}YSZ layer exhibits a 45% of flat area, i.e. blue region in (b) whereas the ^{MOD}CZO buffer layer improves the planarity up to a 75% of area fraction, blue region in (d). Right image: schema of the used conductor architecture.

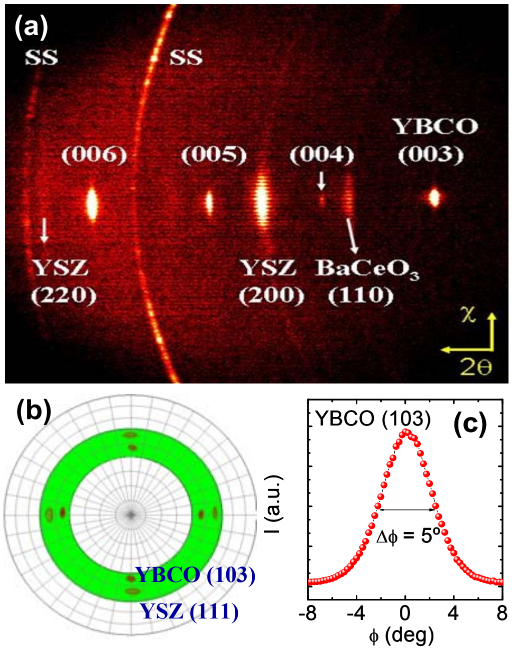


Figure 35 - (a) X-ray diffraction pattern obtained with an area detector of a ^{TFA}YBCO film grown on CZO buffered ^{ABAD}YSZ/SS metallic substrate. Epitaxial dots for the YBCO and YSZ layers are clearly distinguished of the polycrystalline rings coming from the stainless steel substrate. (b) Pole figure of the ^{TFA}YBCO film and of the ^{ABAD}YSZ layer showing the epitaxial relationship between them. (c) Detail of the ϕ-scan of the (103) YBCO reflection. Arrows show the value of the full width at half maximum.

Concerning RABiT substrates, the first large effort was devoted to understanding the issues controlling deposition and growth of the $La_2Zr_2O_7$ (LZO) pyrochlore which behaves as an efficient barrier against atomic diffusion under the YBCO growth conditions [42].

The consortium developed a processing methodology for long metallic substrates using a tubular furnace having a length of ~3 m where through dip coating deposition continuous processing might be achieved. NiW RABiT metallic substrates coated with LZO buffer layers were achieved in lengths up to ~10 m (Figure 37) through growth processes performed in a Ar/10% H_2 atmosphere to protect the substrate from oxidation.

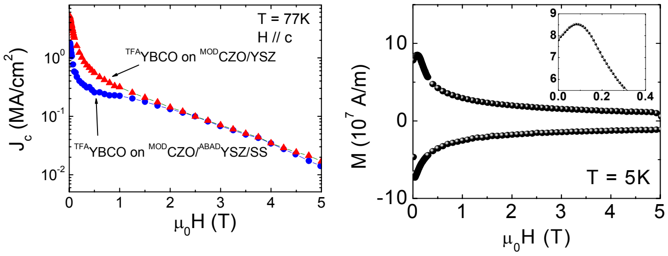


Figure 36 – Left: Critical current density at 77 K as a function of applied magnetic field parallel to the c-axis for ^{TFA}YBCO films grown on ^{MOD}CZO buffered YSZ single crystal (red triangles) and ^{ABAD}YSZ/SS metallic substrate (blue circles). Note that granularity effects are only important below a field value of 2T. Above this field critical current of the coated conductors equals that of the single crystal suggesting that this regime is dominated by the pinning of the grains. Right: Hysteresis loop measured at 5 K where a finite field magnetization peak is measured indicating that the internal field at the grain boundaries is influenced by the demagnetizing field. This feature indicates that the intragranular J_c is higher than the percolative J_c.

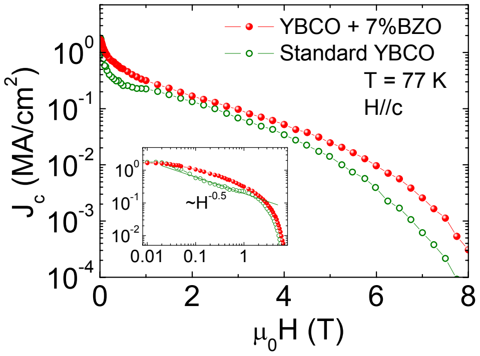


Figure 37 - Field dependence of the critical current density of a ^{TFA}YBCO-BZO nanocomposite coated conductor (solid circles) and of a standard ^{TFA}YBCO conductor (open circles). Log-log plot is shown in the inset. Both films are deposited on ^{MOD}CZO buffered ^{ABAD}YSZ/SS tapes. Introduction of BZO nanoparticles strongly enhances vortex pinning for the whole regime of applied magnetic field studied. Note that the J_c~$H^{-0.5}$ regime has not been observed in the CC nanocomposite.

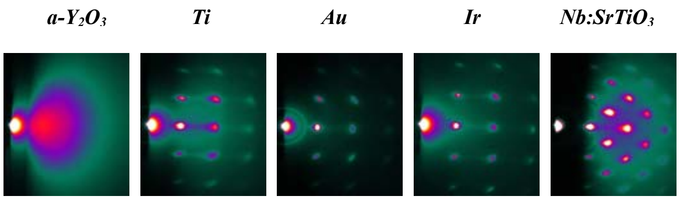


Figure 38 - RHEED analysis of the layers required for IBAD deposition of a full metallic architecture on top of a polycrystalline Hastelloy substrate with amorphous Y_2O_3 on top of it.

The film thickness achieved in a single deposition was typically ~100 nm but total thickness up to ~200 nm were prepared. A very useful tool to quantify non-destructively the epitaxy quality of the buffer films was developed which included the out-of plane and in-plane orientation distribution of the crystallites (Figure 39). This parameter could be also correlated with the surface crystallinity, as determined with RHEED analysis (Figure 39) [43]. A very important issue in the processing of this buffer layer, still not completely solved, is the required tight control of the stoichiometry, and the relationship between the epitaxial quality and the surface planarity. Through analysis of the influence of the XRD defined epitaxial quality factors on J_c of the evaporated YBCO layers (Figure 40), a quality threshold for finite J_c values was defined. This quality parameter is very likely correlated with the low surface planarity of the CGO MOD buffer layers grown on top of the LZO buffer layers which still displayed limited values (in the range of ~50 %, see Figure 41). TFA-YBCO layers based on the architecture NiW/LZO^{MOD}/CeO$_2$ achieved J_c values up to ~0.5 MA/cm^2 while MOCVD or evaporated YBCO layers displayed J_c up to ~1 MA/cm^2. The main limiting factor for further enhancement of the superconducting performance seems therefore to be linked to the epitaxial quality of the underlying buffer layer. The achievement of epitaxial TFA layers avoiding an excessive oxidation of the substrate required a double step process, a short low temperature anneal to nucleate the first YBCO layers and a second higher temperature anneal to complete the grain growth with a strong electrical connectivity.

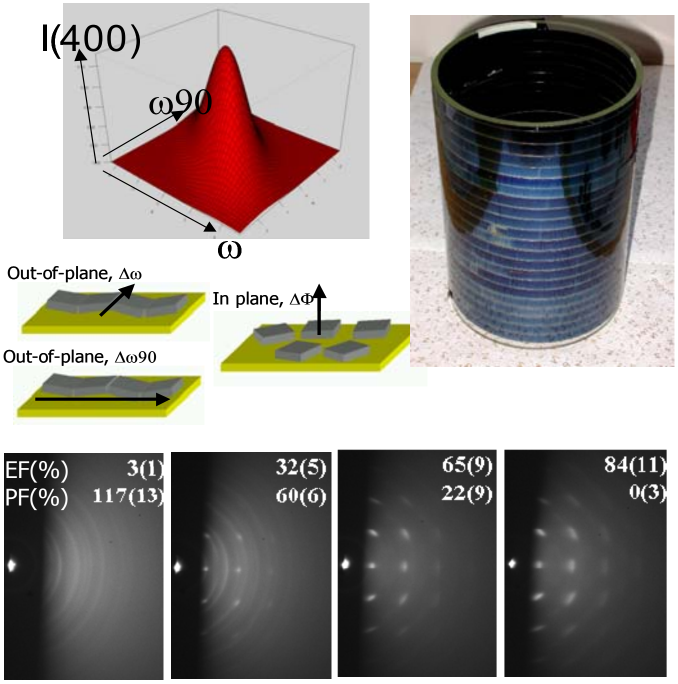


Figure 39 – Upper right image: Photography of a 10 m flexible metallic substrate coated with LZO. Left images: Two dimensional X-ray diffraction texture analysis of flexible metallic substrates as a tool of quality control. The epitaxial fraction and polycrystalline fraction correlate with RHEED analysis of the surface crystallinity (lower panel). Measurements carried out for the LZO buffer layer used in the conductor architecture NiW/LZO^{MOD}/CeO$_2$/YBCO.

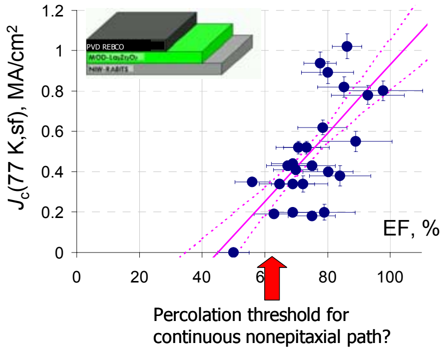


Figure 40 - Dependence of self-field J_c of YBCO films grown on top of CeO$_2$/^{MOD}LZO/NiW substrates on the epitaxial quality factor defined from X-ray diffraction. The limiting value for finite J_c values is defined from this plot.

HLPE YBCO layers were also successfully grown on top of the ^{MOD}LZO/NiW substrates by using a thin PLD-YBCO layer or also on other cap layers introduced to further protect from the detrimental effects of impurities. Figure 42 shows the self field critical currents achieved on RABiT tapes as compared to single crystal substrates by optimizing the growth temperatures. The conclusion is that I_c values of 300 A/cm-w have been achieved in ^{MOD}LZO buffered NiW substrates which correspond to J_c values at 77 K in the range of 2 MA/cm^2. Similar values were also obtained in BZO doped films on the same substrates.

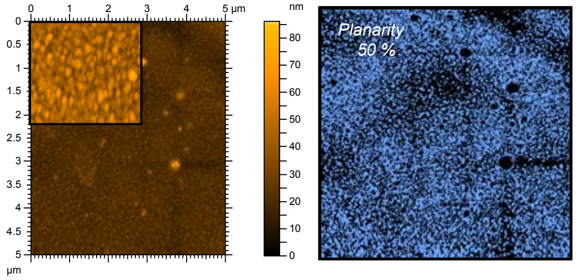


Figure 41 - On the left side are the AFM images of the surface of a ^{MOD}CGO/^{MOD}LZO/NiW substrate grown in Ar-H$_2$ atmosphere. Right image: Binarized image after applying a planarizing mask of 1.5nm is quantified in ~50 %.

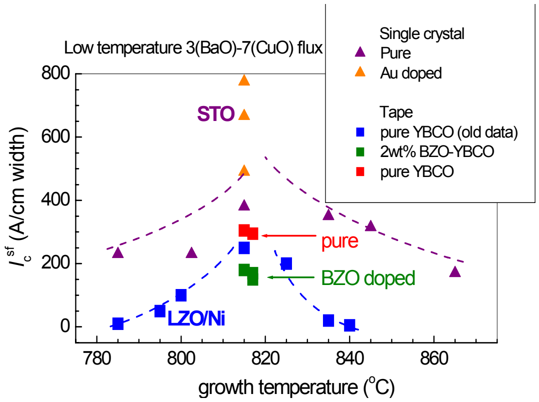


Figure 42 - Dependence of self-field I_c of YBCO films grown on top of ^{PLD}YBCO/^{MOD}LZO/NiW and single crystal substrates vs the growth temperature.

The homogeneity of several conductors grown on ^{MOD}LZO/NiW metallic substrates was analyzed through the magnetoscan technique, as indicated in Figure 43. These analyses showed that high local J_c values are actually achieved through the “all chemical” approach but some inhomogeneities still persist.

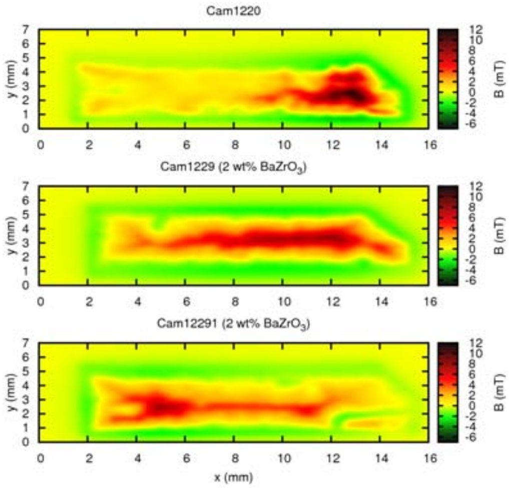


Figure 43 - Magnetoscan images of HLPE YBCO and YBCO/BZO nanocomposite conductors grown on ^{MOD}LZO/NiW substrates where some inhomogeneities can be discerned.

Manufacturing of conductors in the range beyond 1 m was demonstrated by the HIPERCHEM consortium by using a 6 m reel to reel furnace fully equipped for buffer layer growth (Figure 44). Buffered 10 m pieces of metallic substrates are routinely obtained with this tubular furnace. YBCO conductor pyrolysis was performed in static mode in a tubular furnace and the short pyrolysis process of YBCO films with thickness of ~500 nm could be fully reproduced (Figure 45).

Figure 44 - Photograph of a long (6 m) tubular furnace with controlled atmosphere and 16 zones of temperature control. Buffer layer growth and YBCO pyrolysis can be performed in this furnace.

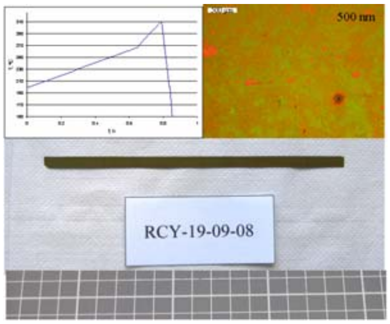


Figure 45 – Upper images: Schema of the short pyrolysis process (~ 1h) followed for 1 m long YBCO conductors. Optical microscopy showed that high quality and homogeneous precursors were indeed achieved. Lower panel: Photograph of a 0.4 m conductor.

The growth steps of the YBCO layers was performed in a batch-type furnace where the conductor is wrapped around a drum (Figure 46). Although biaxial YBCO film growth is achieved, further improvement of the process is required for long length tape production. The batch furnace approach appears to be appealing in the midterm for medium length tape production, however in the long term a continuous reel to reel process is preferable. Reel to reel process, however, requires that a full optimization of the different processing steps is achieved.

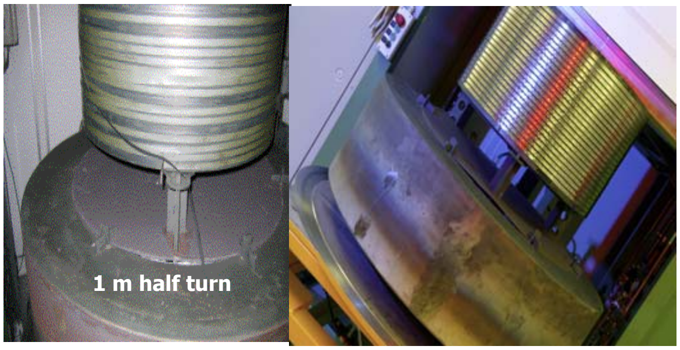


Figure 46 – Photograph of the drum where the YBCO tapes are wrapped in the batch type furnace for YBCO high temperature growth.

In conclusion, highly significant progress was achieved by the HIPERCHEM consortium on the practical implementation of chemically based methodologies for coated conductor manufacturing by MOD. High performances has been already demonstrated, including the introduction of nanostructures behaving as APC, in well protected metallic substrates (ABAD substrates). The achievement of cap layers with a high degree of surface planarity was identified as an essential issue. Further very significant progress was also achieved in the development of RABiT substrates for an “all chemical” approach to CC were also achieved, particularly in the long length growth of buffer layers. MOD YBCO and HLPE YBCO conductor growth was demonstrated in short lengths with high performances, while MOD conductor processing in the range of 1m was also demonstrated but further optimization is required to achieve the performances already demonstrated in short lengths.

IMPACT ON RESEARCH SECTOR AND INDUSTRY

The HIPERCHEM project has generated new knowledge, materials and methodologies which will have a high scientific impact in coated conductors and in other areas of superconductivity, as well as in other fields. Nanostructuring is an area that will strongly benefit from the outputs generated within HIPERCHEM. The general principles developed within the project for nanostructure generation based on strain engineering enables self-assembly and self-organizationto be used as a general route to generate nanostructured materials, with whatever functionality that is required 0D and 1D interfacial nanostructures and 3D nanocomposites have been shown to be feasible by using solution chemistry. Also novel approaches to grow thin films and multilayers with nanoscale control (2D nanostructures) have been created. In the case of 0D and 1D nanostructures, additionally, assisted self-assembling processes have been developed.

Many types of functional or multifunctional materials could benefit from these novel chemical approaches towards nanostructuring, particularly those where large scale structures are required. Ferromagnetism, ferroelectricity, multiferroics, nanoionics (chemical sensors, electrolytes), photovoltaics, semiconductors and metallic behaviour etc can be mentioned. In all these fields new and modified functionalities and/or properties have been derived from the interfaces and hence an advanced control of their properties should be achievable through an accurate control of the structure and composition at the nanoscale. Until now most of the efforts towards nanofabrication in functional oxides have been based on top-down approaches and vacuum deposition methodologies. The chemical routes followed within HIPERCHEM for the generation of oxide nanostructures open up a completely new avenue which is still in its infancy and through further research novel functionalities and devices should be achieved. Also the thin film growth based on MOD and HLPE has registered an outstanding development within HIPERCHEM. The Consortium are therefore convinced that, similarly to techniques such as PLD which became a commercial tool with the advent of high temperature superconductors, the chemical approaches to film growth will become a low cost alternative with a high commercial attractiveness.

Within the area of superconductivity itself it is expected that many developments carried out within the scope of HIPERCHEM will bring a noticeable progress. New concepts, ideas and methodologies related to the problem of vortex pinning and critical currents have been devised. Complex issues related to nanostructure control through TEM investigation and how the existing defects influence vortex pinning at different magnetic fields and temperatures, as well as its anisotropic behaviour, have been clarified and hence effective methodologies for a steady increase of the critical currents are now possible. The advances relating nanostructure and vortex properties will contribute widely to the development of superconducting materials in a broad sense, not only in relationship to coated conductors development.

Concerning the industrial impact of HIPERCHEM, the advances in scalability of chemical methodologies for coated conductor production it should first be stressed.. These methodologies include deposition methods, large furnaces with accurate control of temperature, gas flow and atmosphere, as well as the characterization methodologies for long length conductors (microstructural and superconducting properties). It should be noted that these engineering developments can also be of interest in other areas where large scale production of nanostructured materials at low cost is also required.

Three different types of conductors have been shown within HIPERCHEM to have a strong potential for industrial competitiveness:

1/ Hybrid chemical/physical conductors;
2/ All chemical conductors;
3/ HLPE based conductors.

In the first case one of the buffer layers is grown through a vacuum deposition method while the rest of the layers in the conductor architecture are based on MOD deposition and growth. These conductors have already demonstrated competitive performance and they can be classified as “medium cost”. In the second type of conductors the full oxide architecture of the conductor is based on MOD and the biaxial texture arises from the RABiT substrate. This approach has the potential for the lowest cost but the performance are still slightly lower than those from the first method. Finally, in the third conductor approach HLPE growth of the superconducting layer is combined with MOD buffer layers. The hurdle here is to scale-up the process which could become medium cost.

Other significant developments of industrial interest carried out by the consortium were the generation of a fully metallic buffer layer architecture by means of IBAD deposition on Hastelloy and also a fast characterization system, magnetoscan, with a strong potential as an in-situ processing control tool for long length conductors.

In conclusion, the consortium has advanced consistently the development of chemically-based conductors in many aspects and has placed Europe in a position to compete worldwide in several specific conductor architectures.

A complete list of the exploitable results of the HIPERCHEM project which have been described here are listed in Table 1.

Exploitable number	Exploitable result
1	Metal-organic solution compositions corresponding to different crystallographic phases
2	Hybrid liquid phase epitaxy (HLPE) process for fabrication of HTS coated conductors and other functional oxides
3	Knowledge for the growth of high quality MOD or HLPE superconducting conductors with a single buffer layer grown by MOD.
4	Knowledge for the growth of high quality mixed oxide buffer layers by MOD on RABiT substrates
5	Knowledge on the growth process of interfacial oxide nanodots by MOD
6	Magnetoscan technique
7	Knowledge for the incorporation of nanoscaled oxide particles into REBCO using physical vapour deposition.
8	A processing route to forming high J_c conductors by incorporating Y_2O_3 (and other) nanoparticles using a HLPE method
9	Novel TiN based conducting buffer architecture for use as an internal shunt in HTS tapes.

Table 1 – List of exploitable results generated in the project

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SECTION 2: DISSEMINATION AND USE

Summary of the Project:

The overall objective of the HIPERCHEM project was to develop radically innovative low cost technologies for the mass production of nano-structured high temperature superconducting materials based on chemical solution processing. Novel nano-structuring methodologies would be developed for the growth of epitaxial nanocomposite films and coated conductors with excellent performance. The originality of the project lay in the fact that two rapid growth rate chemical processing techniques (metal-organic decomposition and hybrid liquid phase epitaxy) were combined to grow epitaxial $REBa_2Cu_3O_7$ high-temperature superconductor films. The purpose was to achieve an artificial network of nano-defects that would immobilize the superconducting vortices and, hence, allow the achievement of high critical currents and give weak magnetic field dependence in films and coated conductors of high thickness.

The Project Consortium

The consortium consisted of six partners from five EU Member States: three universities (University of Cambridge, UK {UCAM}; Catholique University of Louvain-la-Neuve, B {UCL} and Technical University of Vienna, A {TUW}), two research institutes (The Institut de Cienca de Materials de Barcelona part of the Consejo Superior de Investigaciones Cientificas, E {CSIC-ICMAB} and the Leibniz-Institut für Festkörper– und Werkstoffforschung Dresden, D {IFW}) and the industrial company Nexans Superconductors GmbH, D {NSC}. CSIC-ICMAB was the project co-ordinator under the leadership of Professor X. Obradors.

The Exploitable Results

Nine exploitable results were developed by the project consortium over the 45 Months duration of the contract. The details of each result are given below.

Market Considerations

Since the main applications for the results are in high temperature superconductors where the technology will be based on different combinations of the results the market considerations are the same in many cases. The background to these is therefore summarised here.

The high current superconducting tapes have very wide applicability for power systems and high field magnets. The sectors involved in the whole chain value are very wide: metallurgical for substrates, chemical for metallorganic precursors, ceramics or materials production for the conductor production through integration of the elements, electrical systems production by integrating the new conductors in new power systems (cables, motors and generators, fault current limiters, transformers), instrumentation for bio-medicine through production of high field magnets for NMR and MRI, high field magnets for fusion reactors, electrical energy production and distribution through use of new superconductor systems with enhanced efficiency and reliability.

Further development of the whole products of the chain of value is required. Particularly at the level of components of the new conductors and its full integration (metallic substrates, chemicals, complex multilayered conductors) more research and development is required to simplify the process and architectures and to achieve a lower cost in the production stage. Industrial development of conductors to achieve high throughput processing methodologies and hence increase the conductor yield is a requirement to be able to reach the industrial scale production. At least 3-4 years of further development with investments in scale up equipments will be required to reach industrial products. Power and magnet demonstrators can be produced with the conductors already existing, however the specific performances and cost reduction required to achieve a significant market penetration of the power systems needs still to be further advanced. Expectations for very significant market penetration (~ 50%) have been made for ~2020.

Result No:1 - Metal-organic solution compositions corresponding to different crystallographic phases

Description:

New conductors having an oxide multilayered epitaxial structure prepared by metallorganic decomposition (MOD) can be envisaged now on top of several types of metallic substrates. To enhance the superconducting performances of YBCO layers at high magnetic fields and high temperatures a key requirement has been to enhance vortex pinning in order to achieve high critical currents. A key development of this project has been to find a suitable methodology for in-situ growth of two immiscible crystallographic phases and hence generate a nanocomposite which may enhance the performance due to the high interfacial surface between two distinct phases. The compounds which have been prepared from complex chemical solutions are adequate for YBCO and the secondary phases were $BaZrO_3$, $BaHfO_3$ and Y_2O_3, although the methodology is applicable to many other secondary phases. The typical size of the secondary phases is ~20 nm and they have a strong influence on the vortex pinning properties of the YBCO superconductor. Actually a worldwide record in terms of vortex pinning force was achieved through this methodology. ($F_p \sim 80 GN/m^3$ at 3-5 T at 65 K).

These complex metallorganic solutions have been shown to stand short pyrolysis times (<1 h) in metre long tapes and hence they are compatible with fast manufacturing rates.

The newly developed compositions will become the main choice for all the high performance conductors to be developed in the near future and the fact that they have been achieved based on a low cost chemical solution methodology leads to a very prominent competitive advantage to the consortium.

Market Applications:

Further development of the whole products of the chain of value is required. Particularly at the level of components of the new conductors and its full integration (metallic substrates, chemicals, complex multilayered conductors), more research and development is required to optimize the compositions and the process of the nanocomposite conductor production. The initial rise of market penetration of superconducting power systems is expected for 2012-2013 hence the growth of coated conductor production should occur in parallel to that.

Stage of Development:

The new chemical solution compositions have been widely investigated at the laboratory scale and they have been demonstrated in the present most suitable conductor architectures. However, further investigation of the microstructural modifications on the superconducting matrix is required in order to achieve more processing control in long length conductor production.

Collaboration Sought or Offered:

Collaborations are sought for scaling up the nanocomposite conductor production and for manufacturing control tools and also for the wide scale production of chemical solutions and the distribution. The corresponding investment for the scaling up development is also sought.

Knowledge on thin film oxide nanocomposite film preparation by means of a high throughput technique such as MOD is offered. Many fields using functional or multifunctional oxides can be potential users of the generated methodologies and know how. The offer can be effective at all levels, from consultancy to information exchange, development contracts or training.

Collaborator Details:

Industrial experts on materials manufacturing for design of conductor production systems (furnaces, quality control, automatization, etc.). Laboratory or industrial experts on nanoparticle emulsion production for further development of ceramic functional nanocomposites.

Intellectual Property: .

A patent application has been filed in 2006 related to this result.

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Result No:2 - Hybrid liquid phase epitaxy (HLPE) process for fabrication of HTS coated conductors and other functional oxides

Description:

HLPE is a rapid, thick film growth method. It relies on growth of the phase of interest through a thin liquid layer. The innovation of using HLPE is the very fast growth, faster than any other thin film oxide growth method. There are several possible applications which require thick oxide epitaxial films of high quality, including thermal barrier coatings, actuators, as well as superconductor current leads, and power and magnetic devices.

Market Applications:

The sectors include energy (power transmission), and electronics sectors (actuators), and transport (thermal barrier coatings on metals). Translation into products will require another 3 years of research and development.

Stage of Development:

Laboratory prototype

Collaboration Sought or Offered:

Financial support is sought to scale up the process

Collaborator Details:

SME or large company who will provide funds for UCAM to undertake the work and who will provide substrate materials

Intellectual Property:

Know-how of UCAM

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Result No: 3 - Knowledge for the growth of high quality MOD or HLPE super-conducting conductors with a single buffer layer grown by MOD.

Description:

New conductors having an oxide multilayered epitaxial structure prepared by Metallorganic Decomposition (MOD) can be envisaged now on top of textured metallic substrates, such as NiW alloys having a RABiT (Rolling Assisted Biaxial Texture) processing origin. These metallic substrates can be buffered with a single oxide layer of the pyrochlore $La_2Zr_2O_7$ phase and then the superconducting YBCO layer can be grown by HLPE. This single multilayer combination is one of the simplest reported so far for coated conductors and hence it has the potential for being competitive for high current applications. The main advantage of the new development is the simplicity of the architecture and the fact that HLPE YBCO layers can be produced at high processing rates and with large thickness.

Market Applications:

Markets targeted include new power systems (cables, motors and generators, fault current limiters, transformers), instrumentation for bio-medicine through production of high field magnets for NMR and MRI etc. Application of superconductors in some of these product areas is already a reality today albeit using high cost tapes. Thus once lower cost HTS tapes are available (in ~ 3-4 years) then translation into some product areas could happen relatively quickly.

Stage of Development:

The combination of buffer layer and YBCO growth methodology showing that the simplest conductor architecture can be achieved has been demonstrated at the laboratory scale, however the single separate stages are already more advanced: MOD buffer layer at the demonstrator level and YBCO layers by HLPE through a reel-to-reel system.{See Result 2}

Collaboration Sought or Offered:

UCAM require an industrial partner to supply substrates to try to develop long length coating using HLPE.

Further funding will be sought by NSC to further optimise the process and explore the possibility of substituting PLD with other techniques (MOCVD and MOD). Industrial partners with experience in PLD and MOCVD will be sought.

Collaborator Details:

Small or large company sought.

Intellectual Property:

The partners NSC and UCAM have submitted an application for a European patent

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Result No:4 - Knowledge for the growth of high quality mixed oxide buffer layers by MOD on RABiT substrates

Description:

New conductors having an oxide multilayered epitaxial structure prepared by metallorganic decomposition (MOD) can be envisaged now on top of textured metallic substrates, such as NiW alloys having a RABiT (Rolling Assisted Biaxial Texture) processing origin. The typical compositions of the developed buffer layers are $Ce_{1-x}M_xO_2$ (M=Gd, Zr) and they have an improved microstructure (epitaxy quality, density, surface planarity, etc.) which lead to enhanced conductor performances when superconducting $YBa_2Cu_3O_7$ (YBCO) layers are grown on top of them. The main advantage of the new development is that simple conductor architectures become feasible for the “all chemical” approach to high current superconducting tapes. This approach can be classified as “low cost” and hence the generated knowledge will allow to advance the scaling up of the conductor production.

Market Applications

The main application is in HTS (high temperature superconducting) tapes for power systems (cables, motors, fault current limiters) and for magnets for MRI, NMR etc. These developments will take another 3-5 years assuming that funding can be secured to continue the R&D.

Stage of Development:

Deposition of MOD layers on metallic substrates has been widely developed at the laboratory scale and the industrial scalability has been demonstrated with production lengths above ~10 m. These buffered RABiT conductors can be already considered an industrial product because other laboratories and industrial developers can use them for further advancement of their industrial products. Nevertheless much larger lengths will be required for full industrial power systems and magnets.

Collaboration Sought or Offered:

Collaborations are sought for scaling up the conductor production and manufacturing control tools. Agreements with the manufacturers of metallic substrates are required to secure the integration of the full tape in a single competitive product at reduced costs. The corresponding investment for the scaling up development is also sought.

Knowledge on thin film preparation of oxides by means of a high throughput technique such as MOD is offered. Many fields using functional oxides can be potential users of the generated methodologies and know how. The offer can be effective at all levels, from consultancy to information exchange, development contracts or training.

Collaborator Details:

Industrial experts on materials manufacturing for design of conductor production systems (furnaces, quality control, automatization, etc.). Users of electroceramic films which may have interest in using MOD as a manufacturing process.

Intellectual Property:

The know-how is held by the partners involved.

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Result No: 5 - Knowledge on the growth process of interfacial oxide nanodots by MOD

Description:

Nanostructured oxide templates grown on ceramic or semiconducting substrates and having varied shapes and sizes. The preparation methodologies are based on high throughput chemical solution techniques and hence have a low cost while they can be used for large area applications. Many functionalities of the nanotemplates have been already shown to be of interest (magnetic, ionic conductivity, catalysis, ferroelectric, metallic contacts, etc.). The main advantages of the developed techniques are low cost, as compared to the usual vacuum deposition techniques, and the high surfaces or long lengths which can be achieved.

Market Applications:

The wide sector of the electroceramics is the main final user of the new functional nanostructured oxides. These advanced materials can be used in several sectors: electronics, magnetism, sensors, catalysis, energy related devices, etc. Also the chemical sector producing the metallorganic precursors will benefit from the new products generated with these new functional electroceramics. At least 2-4 additional years would be required to develop new specific applications based on these nanostructured templates while the required market penetration time of the devices based on them could differ in each case.

Stage of Development:

All the new nanostructured templates which have been produced are only obtained at the laboratory stage and also only in some specific cases some progress in the development of devices demonstrators has been made. It is clear that further R&D effort is required to reach the demonstrator and industrial production stages.

Collaboration Sought or Offered:

Collaborations are sought for further development of new applications based on the interfacial nanostructures and also for nanoscale control of the structure and properties. The corresponding investment for the scaling up development is also sought.

Knowledge on preparation of nanostructured oxides (nanodots, nanowires, nanorods, etc.) by means of a high throughput technique such as MOD is offered. Many fields using functional oxides can be potential users of the generated methodologies and know how. The offer can be effective at all levels, from consultancy to information exchange, development contracts or training.

Collaborator Details:

Research developers with experience of nanoscale characterization of functional properties and development experts on nano-micro device integration. Final users of functional electroceramics which may help to precisely define the required performance and the opportunities arising from these new nanotemplates.

Intellectual Property:

The know how is held by the partners involved

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Result No:6 - Magnetoscan technique

Description:

Experimental and theoretical work to adapt the magnetoscan technique, originally developed for large bulk high temperature superconductors, for long length coated conductors was successful. The magnetoscan technique allows the local critical current density of the superconducting tape to be assessed through a single "central line scan" of the local magnetic field generated by the super-currents induced by a large permanent magnet, e.g. during processing.

Market Applications:

Depending on the processing conditions (e.g. batch versus reel-to-reel), the technique can be used for quality assurance (i.e. post-processing quality control) or for detecting defects locally immediately after completing the last deposition step (i.e. a few meters behind the last production step during processing).

For both types of instrument a development time of 18 months will be needed for the production of an industrial prototype device.

Stage of Development:

Laboratory prototype

Collaboration Sought or Offered:

Collaborations offered to European (Nexans, Bruker HTS) and non-European (Industrial Research Ltd.) industry.

Collaborator Details:

NSC (Hürth, Germany): Design and construction of an "in-situ" device for reel-to-reel production; design and construction of a post-processing quality assurance device.

Bruker HTS (Alzenau, Germany): Design and construction of an "in-situ" device for reel-to-reel production; design and construction of a post-processing quality assurance device.

IRL (Lower Hutt, New Zealand): Design of a reel-to-reel screening device of commercial coated conductors foreseen for the production of Roebel-type strands made from these commercial coated conductor tapes.

Intellectual Property:

Know-how of the design and application of the technique to HTS tapes.

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Result No:7- Knowledge for the incorporation of nanoscaled oxide particles into REBCO using physical vapour deposition.

Two partners are involved in this Result: IFW and UCAM, their plans are described separately below.

IFW:

Description:

New innovative approaches were used to incorporate nanoscaled oxide particles such as $BaHfO_3$, Y_2O_3 or $Y_2Ba_4Cu(Zr,Nb)O_{12}$ into the High Temperature Superconducting layer using PVD methods. One approach is the application of incomplete layers of a metal (as for example Hf of Ir) or an oxide ($Y_2Ba_4Cu(Zr,Nb)O_{12}$) in combination with complete YBCO layers using pulsed laser deposition (quasi-multilayer approach). A second approach is the application of gas-phase prepared nanoparticles with a defined size (as for example Y_2O_3) in the YBCO matrix. Significantly enhanced superconducting properties of YBCO layers in magnetic fields can be achieved using both approaches.
The incorporation of oxide nanoparticles is a major route to improve the magnetic field dependence of the critical current density in the HTSC layer. The used approaches offer the opportunity to vary easily and independently from each other the material, size and density of the incorporated nanoparticles in order to study in detail the influence of these inclusions on the superconducting properties.

Market Applications:

Optimised critical current densities of superconducting layers in magnetic fields are required for several applications based on REBCO coated conductors, as for example motors, field coils or transformers. Further research is necessary within the next couple of years to tune the superconducting properties according to the requirements for a certain application by using nanoscaled oxide particles in the superconducting matrix.

Stage of Development:

The incorporation of oxide nanoparticles was realised so far in small samples in a laboratory environment only.

Collaboration Sought or Offered:

A partner is needed to upscale an optimised process for long length coated conductors. The knowledge on the preparation of nanoscaled oxide particles inside a superconducting matrix is offered for the growth of other oxide heterostructures with different functional properties.

Collaborator Details:

Research institutes, universities, industrial companies working on superconducting conductors or devices.

Intellectual Property:

The know- how is held by the partner.

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UCAM:

Description:

The creation of nanocomposite structures has wide ramifications not only for flux pinned superconductor cable. Other areas of interest are where the combination of two materials with different functionalities is required (e.g. combining a ferromagnet with an antiferromagnet can give exchange bias which is useful in magnetic read heads in computer hard drives, combining a p-type light absorbing oxide with an n-type oxide can give a highly durable all-oxide solar cell, or combining an insulating oxide with a superconductor can give an extremely high performance composite material for clean power transmission, magnetic energy storage or efficient motors/ transformers

Market Applications:

Magnetic data storage, photovoltaics, power transmission, motors and magnets. Time scales are 5-10 years.

Stage of Development:

Laboratory prototype

Collaboration Sought or Offered:

For the nanocomposite research in general, this is early stage research. Only government support is required. For the superconductor work, we are at the stage of seeking industrial partners,

Collaborator Details:

UCAM are liaising with a European supplier of substrates.

Intellectual Property:

IP has been applied for in the area of rare earth tantalate pinning and is in progress with magnetic pinning. (UK patent application has been filed.

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Result No:8 - A processing route to forming high J_c conductors by incorporating Y_2O_3 (and other) nanoparticles using a HLPE method

Description:

Creating of very high Ic superconducting conductors using a variety of additions to the conductor during growth by HLPE.

Market Applications:

Power transmission, motors, magnets for NMR and MRI. Development is likely to take a least 3 years to industrial scale.

Stage of Development:

Laboratory prototype

Collaboration Sought or Offered:

SME or large company to fund ongoing research

Collaborator Details:

UCAM is seeking to partner with a company involved in developing processes for HTS tape manufacture in order to not only secure funding for the developmental research for process scaling up but also to benefit from the required engineering expertise.

Intellectual Property:

Not Applicable

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Result No:9 - Novel TiN based conducting buffer architecture for use as an internal shunt in HTS tapes.

Description:

An electrically conductive buffer architecture was developed based on IBAD-TiN, which can be used as internal shunt between the metallic substrate and the HTSC layer. An amorphous conducting TaNi seed layer was successfully applied for the preparation of highly textured TiN layers on Hastelloy tapes using ion-beam assisted deposition. The texture of the TiN was transferred to the superconductor using a combination of noble metal layers and a final conducting oxide cap. As a result, highly textured, superconducting YBCO layers were prepared on such templates.

The developed buffer architecture is an alternative to presently used commercial architecture types based on IBAD-MgO or IBAD-YSZ. These architectures require a thick metal shunt (several tens of micrometers) on top of the superconducting layer in order to avoid thermal destruction of the superconductor in case of an over current situation. This is closely connected with a reduced engineering critical current density and additional material costs. An electrically conductive buffer architecture could solve these drawbacks as the metallic substrate will be used as shunt.

Market Applications:

The developed buffer architecture is an alternative to presently used commercial architecture types based on IBAD-MgO or IBAD-YSZ. These architectures require a thick metal shunt (several tens of micrometers) on top of the superconducting layer in order to avoid thermal destruction of the superconductor in case of an over current situation.

The conducting buffer architecture might be used for superconducting cables (especially for dc applications), superconducting devices (as for example fault current limiters). The approach might be alternatively applied for other devices, which require a high degree of crystallographic texture in combination with high conductivity.

Stage of Development:

The conducting buffer architecture was realised so far in small samples in a laboratory environment only. Further R&D is required to continue the optimisation of the buffer layer architecture. This includes a reduction in the number of used layers for more cost-efficiency.

Collaboration Sought or Offered:

A partner is needed in order to optimise the buffer architecture and to upscale the process for longer length or bigger areas. The knowledge on the preparation of highly textured template layers on untextured substrates is offered for the growth of other textured functional materials.

Collaborator Details:

Research institutes, universities, industrial companies working on superconducting conductors and devices or other functional materials, which require a high degree of texture alignment.

Intellectual Property:

A patent application was submitted in 2008

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Dissemination Activities

The consortium published 80 scientific papers in leading refereed scientific journals throughout the world describing the results of the HIPERCHEM project. In addition 39 Key Note lectures, 137 conference talks and more than 100 posters were presented at national and international conferences. Further publications and conference papers are in preparation. Full details of these dissemination activities are given in the public part of the website: www.icmab.es/hiperchem