



# GILDA

## Annual Report 2014

## Abstract

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This document resumes the activity of the italian CRG beamline at ESRF (GILDA project) during year 2014. Statistic data on the beamtime use are presented as well as the latest major instrumental advancements and highlight experiments.

## Keywords

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Italian beamline at ESRF

GILDA project

X-ray Absorption Spectroscopy

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Year 2014 has brought decisive facts and news for our users. GILDA has successfully undergone the periodical evaluation by the ESRF Review Panel. The panel applauded the hard work of the beamline staff and recognized the remarkable quantity and quality of science that the beamline has delivered over the last five years despite the challenging landscape for funding and staffing encountered during this period. We would like to thank all our users for their huge contribution (a complete report on the activity in the past 5 years is available on the web page of the beamline). The panel also expressed a positive opinion about the continuation of the project for the forthcoming 5 years. Thus, the user community will profit of the services of the beamline till end 2018, i.e. the moment of the major refurbishment of the ESRF storage ring. These 5 years will be spent in progressively rebuilding the beamline as a dedicated financial support of 1 M€ has been received from CNR. The rebuilding will affect mainly the optics of the instrument as you will find described in the next section and will be carried out in order to affect at minimum the users operation. The project will also change its name so, from Jan 1st 2015, GILDA is formally “retired” and the Italian CRG beamline at ESRF will be named **LISA**, **L**inea **I**taliana per la **S**pettroscopia d'**A**ssorbimento x (Italian beamline for x-ray absorption spectroscopy).

In her last year of activity GILDA has delivered 369 shifts in user mode: 258 for the ESRF beamtime quota and 111 for the CRG beamtime for a total of 24 experiments. The scientific beamline activity has been severely affected by the breakdown of two ball bearings of the complex monochromator mechanics stopping the operation during two full months until the intervention of an external company. From the experimental point of view we have carried out some new experiments using the new instrumentation presented last year (namely XEOL, cryostat for liquid samples and acquisition under excitation) and results will be published in the forthcoming months. Moreover, since the last call for experiment a new form of access, the BAG (Block Allocation Groups) has been launched as reported below.

Year 2015 will be devoted to the finalization of the details of the LISA project by designing a new beamline layout that will be compatible with the present source as well as with the new 3-pole wiggler announced for the future ESRF ring. At present, the Calls for Tender for the monochromator and the mirrors are being finalized and we aim to receive the first new optical elements in one year from now.

After 20 years of operations the Italian CRG at the ESRF will undergo a deep refurbishment process leading to an instrument fully dedicated to X-ray Absorption Spectroscopy and related techniques. For this reason also the name of the project has been changed to **LISA**, **L**inea **I**taliana per la **S**pettroscopia d'**A**ssorbimento di raggi X (Italian beamLine for x-ray Absorption Spectroscopy). LISA will take the X-ray beam from a bending magnet of the ESRF and will be fully compatible with the source (3 poles wiggler) foreseen after the realization of the Phase II of the ESRF upgrade program. LISA will consist in 3 hutches: the first will contain the optical elements, a first experimental hutch for XAS experiments in non-focusing configuration and a second one for experiments with a focused beam.

The beamline, will provide a monochromatic x-ray photon beam in the energy interval 4 - 90 keV, with an energy resolution  $\delta E/E \sim 10^{-5} - 10^{-4}$ , a spot size of about 0.10(H)× 0.15 mm(H) (focused configuration, present source) and a flux at the sample position up to  $10^9 - 10^{11}$  ph/s (depending on the energy). The optical scheme will consist in a collimating mirror, a double crystal monochromator and a toroidal focusing mirror. The first mirror will have a length of 1 m and be cylindrically bent; the incidence angle will be 2 mrad and will be coated with Platinum and Si for efficient harmonic rejection above and below 15 keV respectively. The second mirror will have a toroidal shape and will realize the horizontal and vertical focusing. Two toroids on the same substrate will be realized one coated with Pt and the other with Si. A flat zone between the two toroids will provide a reflecting surface for applications needing a non focused x-ray beam. The monochromator will be a double crystal, fixed exit device, equipped with a pair of flat Si(311) and Si(111) crystals. It will provide monochromatic X-ray beams approximately in the 4-80 keV range. For the harmonic rejection at lower energies (<5 keV) a pair of flat Pt-coated mirrors will be available and their containing vessel will be in the first experimental hutch (EH1). For energies above 40 keV the mirrors will be withdrawn from the beam path and the beamline will operate with an unfocused beam. The option of using collimating refracting lenses inside the Front End for this range of energy is at present under consideration in collaboration with the other CRG groups of ESRF.

The x-ray absorption spectroscopy (XAS) hutch will be realized by merging the existing experimental hutches of the former GILDA beamline. This, in order to exploit the focal point of the toroidal mirror that is foreseen to be placed at about 15 m from the second mirror. The figure below shows the layout of LISA

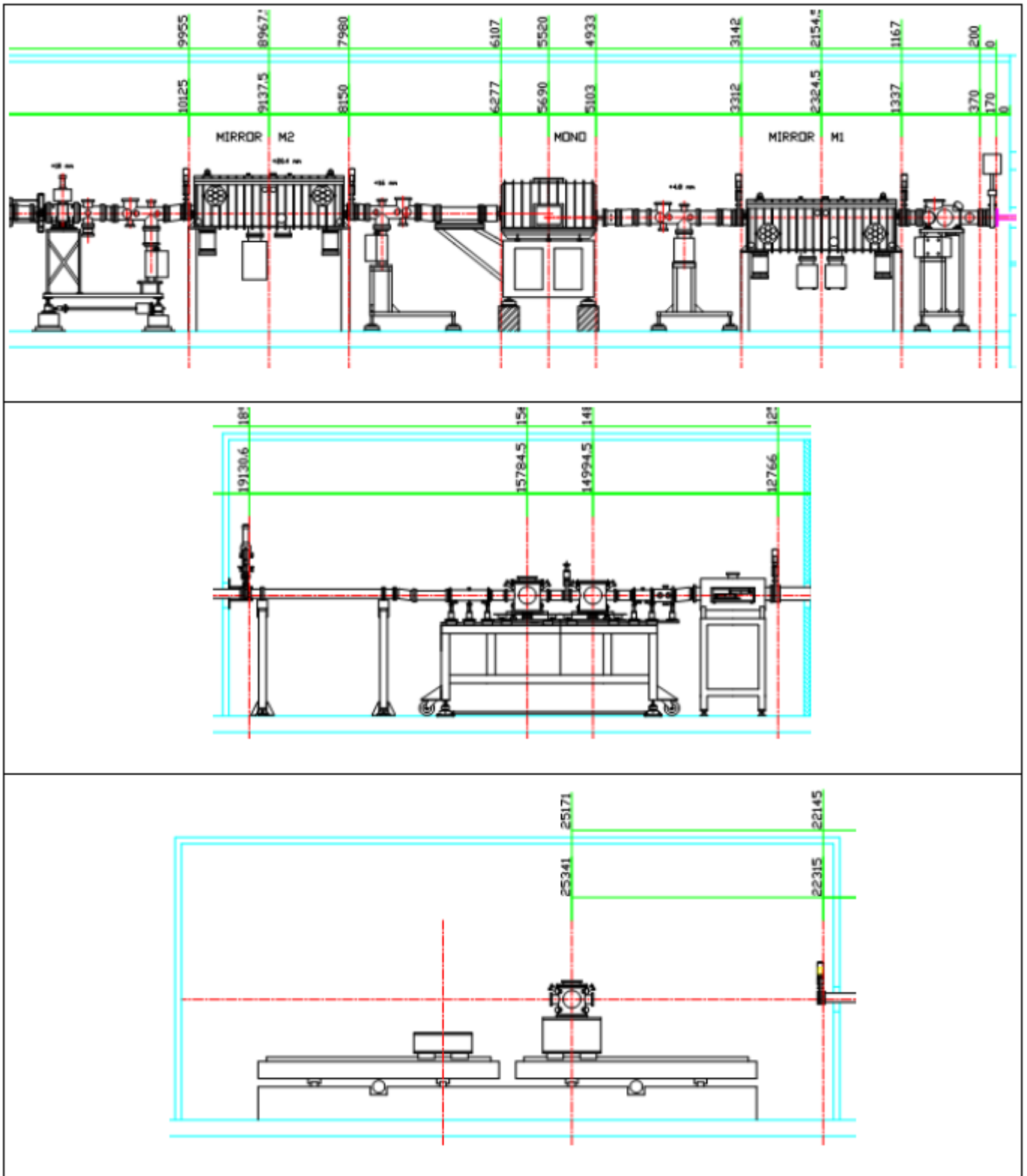


Fig.2. Layout of LISA with the various Experimental Hutches and optical elements. In the upper panel: optic hutche with the collimating first mirror, the monochromator and the second mirror (double toroidal). Central panel: the XAS hutche for measurements in non-focusing mode. Bottom panel the main experimental station for measurements with the focused beam.

A long bench will host the experimental stations. At the moment two stations are foreseen, one with a vacuum chamber for fluorescence/Reflexafs measurements, the other will be an open station with a large space for the installation of user-specific instrumentation (high temp ovens, 2-dimensional detector for joint XAS-XRD measurements, reaction cells).

The former EH1 will host the vacuum chambers and IC detectors for XAS experiments of high quality in transmission mode using a non-focused beam. In this hutch there will also be placed the vessel for the mirror pair for harmonic rejection at lower energies (<5 keV).

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## Differential data acquisition

When the aim of an EXAFS study is to reveal small structural changes induced by external excitations the differential acquisition approach needs to be adopted. This has been used for pump-and-probe experiments on light-responsive compounds but also in case of materials sensitive to magnetic fields, changes of temperature, etc, in which structural differences between the ground-state and excited system are close to the technique limits and there is a high risk of losing such information by the standard subtraction of two full spectra.

In order to obtain XAS spectra in differential mode between two states of the sample (excited and not-excited) specific acquisition software has been now made available on GILDA. The program is based on the standard one and uses the same set of routines to communicate with the acquisition hardware. The sequence of operations is as follows: first of all the program selects a specific energy; then data are collected without any sample excitation and stored in a file; successively the program switches on the excitation (optical excitation, electric field, ...) and data are collected again and stored in a different file. The differential signal is calculated subtracting the unexcited spectrum from the excited one. Preliminary tests of this system showed the capability of measuring reliable differential XAS signals. Users are welcome to propose differential XAS experiments for the next runs.

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## BAG proposals

Starting from the call of November 2014 GILDA has adopted, in addition to the usual rolling access, a new form of access to the beamtime: the Block Allocation Group (BAG). In this mode, similarly to what already put in place at ESRF for Molecular Crystallography, a consortium of users can apply for beamtime over a long time period for the realization of a series of experiments on a common scientific topic. The BAG scheme results to be ideal for the activity of doctorate students or young scientists with Post Doc positions. There will be 2 calls per year with the same deadline as for the conventional rolling access and proposals have to be submitted with the same procedure. The template of the proposal form is the same as for the rolling accesses only the length of the text is allowed to exceed the usual 2 pages. The rules for this form of access are the following:



- The Group submits a project with the same format and procedure as the Rolling Access, clearly stating the BAG nature of the proposal. The length of the BAG proposal will be 4 pages instead of the 2 foreseen for rolling access.
- The project will have duration from 1 to 2 years.
- The main proposer of a BAG cannot submit in the same function other proposals in the same call or if he has another BAG active in that period.
- The proposal will clearly describe the samples used in the whole project and will indicate a suitable requested beamtime for each experimental session.
- The Group will indicate a scientist (preferably a person familiar with the beamline or with the XAS technique) who will act as a liaison scientist with the beamline staff and will coordinate the activity of the Group on the beamline. As for standard proposals, a local contact will be assigned for each scheduled experiment.
- Every 6 months a report on the work in progress must be sent to the proposal committee. Basing on this document, the committee will decide the continuation of the project for the following semester.
- At the end of the project a final report should be presented to the committee and there will be the possibility to further extend the project.

It is foreseen that up to 50% of the total beamtime available on the national quota will be used for BAGs. The launch of this access mode has been a success as at the last run we received 5 BAG proposals and 1 was accepted. Users are invited to propose BAGs and to give us feedback on how to improve this new beamtime request scheme.

## Fischer–Tropsch synthesis: EXAFS study of Ru and Pt bimetallic Co based catalysts

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Co/Ru and Co/Pt bimetallic catalysts supported on silica prepared by successive impregnation were characterized, in particular with EXAFS spectroscopy, and tested in Fischer Tropsch synthesis reaction. Both the CO conversion and the total yield towards desired products were tripled thanks to the action of low amount (0.5 wt%) of noble metal. Different interactions of the two noble metals with cobalt in bimetallic catalysts (BMCs) were demonstrated by EXAFS.  $\text{Ru}_{1-y}\text{Co}_y$  solid solutions with hexagonal close packed structure form were identified with  $y$  in the 0.1–0.23 range for all the concentration of noble metal. In the Co/Pt BMCs the structure of Pt containing phase is strongly dependent on Pt amount. Co and Pt form the intermetallic compound PtCo for low Pt amount (0.1%) with a face centered tetragonal structure. Differently for the sample with higher loading of Pt (2%) the EXAFS spectrum could be fitted using a structural model derived from the close packed cubic structure of Pt. The local structures identified with low loading of noble metal in BMCs are the most suitable for the interaction of cobalt/ruthenium and cobalt/platinum interactions in FTS mechanism.

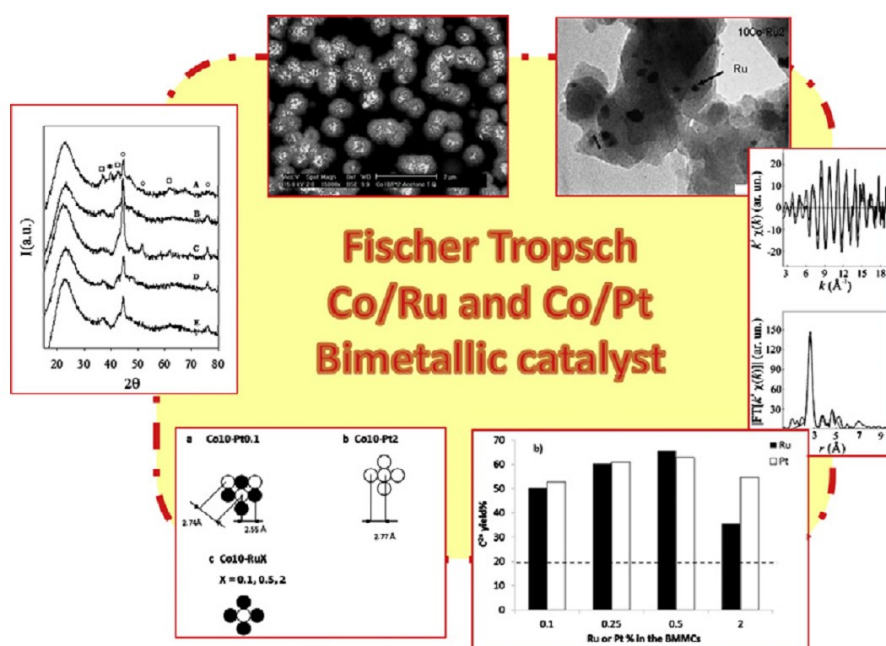


Figure 1, clockwise: TEM-SEM of the investigated samples; Ru-K edge EXAFS and corresponding FT for Co10–Ru0.1; FTS results. CO conversion 5 (a) and  $\text{C}^{2+}$  yield% (b) vs. Ru or Pt wt percentage in BMCs catalysts. The dotted line represents the value obtained using MMC catalyst (Co10); hypotheses of the Co–Pt and Co–Ru ensembles structure in the bimetallic samples as deduced from EXAFS; XRPD power diffraction patterns of samples reduced for 3 h at 623 K in hydrogen atmosphere: A: Co10–Pt2; B: Co10–Pt0.5; C: Co10–Ru2; D: Co10–Ru0.5; E: Co10. The symbols indicate the diffraction peaks relative to: hexagonal Co (circles); CoO (square) and Pt (asterisks).

### Studying the surface reaction between NiO and Al<sub>2</sub>O<sub>3</sub> via total reflection EXAFS (RefLEXAFS)

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The reaction between NiO and (0001)- and (1102)- oriented Al<sub>2</sub>O<sub>3</sub> single crystals has been investigated on model experimental systems by using the RefLEXAFS technique. Depth-sensitive information is obtained by collecting data above and below the critical angle for total reflection. Data analysis was based on the CARD code and a detailed description of the reactive systems was obtained. This demonstrates the full maturity reached by CARD at present and the latest version (with examples and user manual) is available on the dedicated web page: <http://www.esrf.fr/computing/scientific/CARD/CARD.html>. In particular, for (1102)-oriented Al<sub>2</sub>O<sub>3</sub>, the reaction with NiO is almost complete after heating for 6 h at 1273 K, and an almost uniform layer of spinel is found below a mixed (NiO + spinel) layer at the very upmost part of the sample. In the case of the (0001)-oriented Al<sub>2</sub>O<sub>3</sub>, for the same temperature and heating time, the reaction shows a lower advancement degree and a residual fraction of at least 30% NiO is detected in the RefLEXAFS spectra.

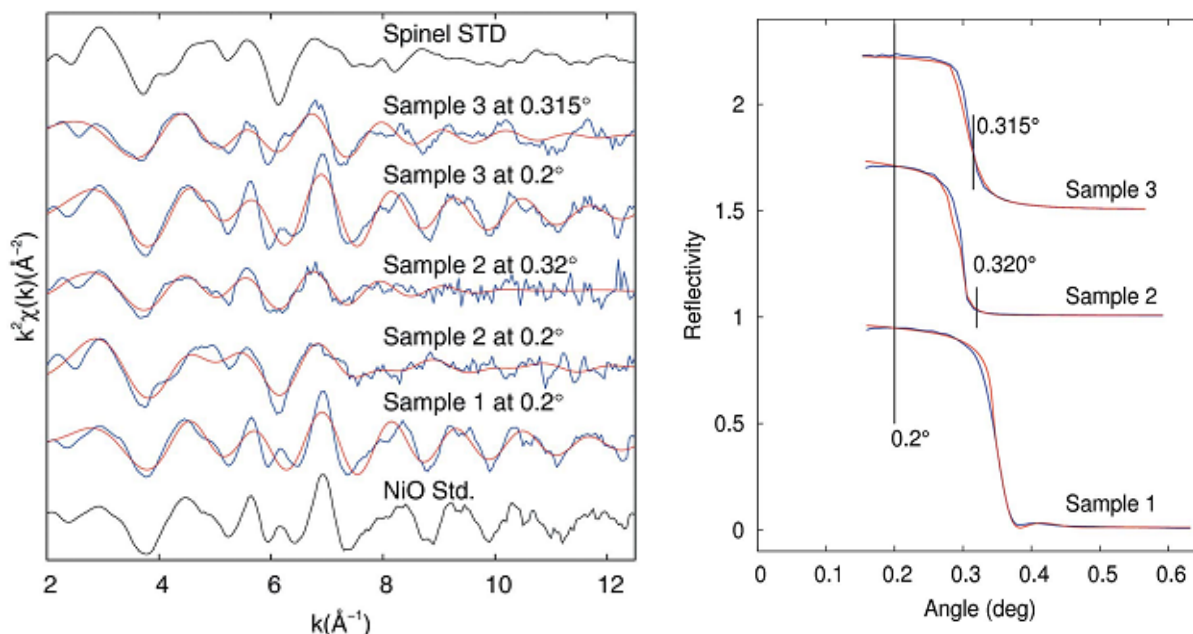


Figure **Left**: RefLEXAFS spectra of the 3 samples (with best fitting curves in red) compared with the model compounds NiO and Al<sub>2</sub>NiO<sub>4</sub> spinel. Sample1\_ Unreacted NiO on Al<sub>2</sub>O<sub>3</sub>, Sample\_2: NiO layer deposited on the (1102) face and annealed for 6 h at 1273 K, Sample\_3: NiO layer deposited on the (0001) face and treated like Sample\_2. Data collected at different incidence angles reflect the different environment on Ni with depth. **Right**: Reflectivity (vertically scaled) of the samples at 8100 eV with the data collection angles marked.

Publication: [Tommaso Costanzo et al. J. Synchrotron Rad. (2014). 21, 395–400].

## Study of the influence of the low temperature cap layer on InGaN/GaN quantum dots structure

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In optoelectronic devices only capped quantum dots can be applied. Nevertheless, the capping layer may change the structure and morphology of the quantum dots. Therefore a key issue is to determine the structure of dots before and after capping process. For this purpose the x-ray methods such as *Extended X-ray Absorption Fine Structure* (EXAFS) and *X-ray Diffraction* (XRD) are well established. Using the former method the local atomic structure around In atoms in InGaN/GaN and GaN/InGaN/GaN structure was determined. The EXAFS results are presented in Fig. 1. The XRD data, in particular asymmetrical XRD reciprocal space maps, contain information about lattice parameters parallel and perpendicular to the sample surface, thus the strain and chemical composition of the quantum dots can be assessed. The example of the GID and AXRD analysis is presented in Fig. 2. Results obtained from both methods indicate the coexistence of two crystalline InGaN phases, namely with low and high In content.

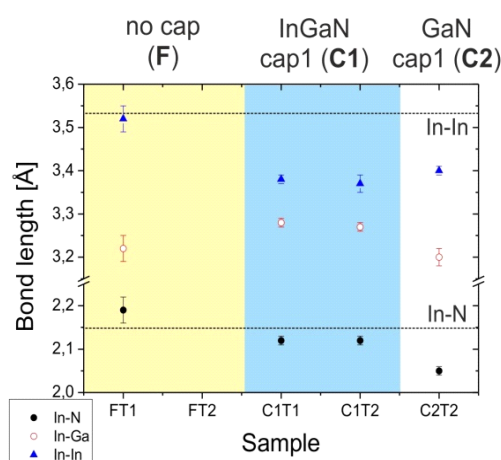


Fig. 1—Bond lengths In-N, In-Ga and In-In obtained from the EXAFS data analysis for un- and capped InGaN quantum dots.

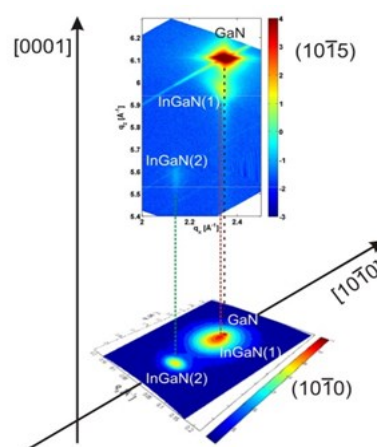


Fig. 2—GID on the c-plane and AXRD in c-plane maps along the surface normal

## EXAFS investigation of the role of Cu on the chemical order and lattice distortion in L1<sub>0</sub> FePtCu thin films

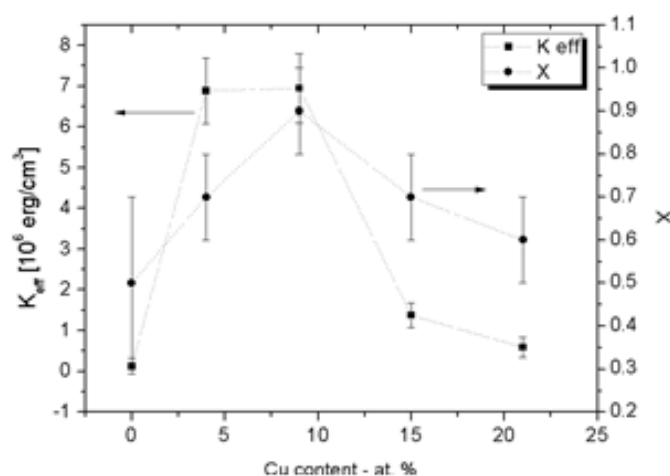
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Nowadays, the development of new advanced technologies in the field of information storage and electronic devices requires a great effort in improving the performance of magnetic materials that are at the basis of the device's functioning. Among them, the chemically ordered FePt binary alloy is of significant interest due to the peculiar atomic arrangement within the crystallographic cell, which induces, through the spin-orbit interaction, a high magnetocrystalline anisotropy (MCA). It has been recently shown that the addition of Cu at different concentrations allows tailoring the MCA, and decreasing the Curie temperature being the most important parameters for the technological application. For these systems, a careful determination of the structural and atomic arrangement becomes essential in order to understand the influence of the additional elements on the magnetic properties.

An EXAFS characterization of ternary FePtCu alloys with different Cu content has been performed at the Cu-K and Pt-L<sub>III</sub> edges in order to describe the local environment around these elements and to compare the structural evolution as a function of the Cu concentration. The significant polarization dependence of the absorption spectra confirmed the high degree of crystallographic order, as previously evidenced from both the magnetic characterization and XRD measurements. In particular, the main effect of Cu atoms in the alloy is a linear reduction of the *c/a* ratio, while the non-monotonic behaviour of the chemical order is consistent with the variation of the MCA (figure).

The good quality of the absorption spectra and the quite simple substitutional model used to fit the EXAFS signals made this study complementary to the approaches usually applied to determine the long-range order and the structural parameters in L1<sub>0</sub> alloys. Moreover, this method represents a useful tool that is able to experimentally distinguish the effects of the chemical ordering and of the lattice distortion through the study of a diluted third element in the L1<sub>0</sub> alloy providing experimental support of theoretical predictions.



Comparison between the effective magnetic anisotropy constant  $K_{\text{eff}}$  (left) and the chemical order parameter  $X$  (right) calculated by EXAFS, as a function of the Cu content

**Highly photo-catalytically active  $\text{Co}_3\text{O}_4$  nanostructured urchins**R. Edla<sup>1</sup>, N.Patel<sup>1,2</sup>, M.Orlandi<sup>1</sup>, N.Bazzanella<sup>1</sup>, V.Bello<sup>3</sup>, C.Maurizio<sup>3</sup>, G.Mattei<sup>3</sup>, P. Mazzoldi<sup>3</sup>, A.Miotello<sup>1</sup><sup>1</sup>Physics Dept., University of Trento, Italy, <sup>2</sup>Physics Dept., University of Mumbai, India,<sup>3</sup>Physics and Astronomy Dept., University of Padova, Italy

Hierarchical 3D urchin-like nanostructures are promising for wastewater treatment through heterogeneous photocatalysis. Their high surface area facilitates catalysis by providing a larger solid-liquid interface. In this work a porous coating assembled with hierarchical 3D  $\text{Co}_3\text{O}_4$  urchin-like particles was synthesized by pulsed laser deposition (PLD) starting from cold pressed Co-B powder target, followed by thermal oxidation (representative SEM images in the left and right Figure). An EXAFS experiment (Co K-edge, GILDA beamline-ESRF) of Co-B-O coatings after urchin formation has been performed to enlighten the possible presence of other amorphous Co-oxide phases that would not be detected by diffraction-based techniques. The EXAFS spectrum of Co-B-O coating on glass, annealed at 600°C for 4h, is reported in right Figure and compared with the similar spectrum of  $\text{Co}_3\text{O}_4$  crystalline powder. The full EXAFS analysis indicate that the Co site in the Co-B-O coating is very similar to the average one in  $\text{Co}_3\text{O}_4$  powder, showing that the most part of Co forms  $\text{Co}_3\text{O}_4$  crystals and no other relevant phases are present. During photocatalysis, urchin-like particles synthesized by PLD displayed significantly higher (~ 5 times) degradation rates when compared to chemical urchins for degradation of methylene blue dye via a photo-Fenton reaction in presence of  $\text{H}_2\text{O}_2$  and visible light.

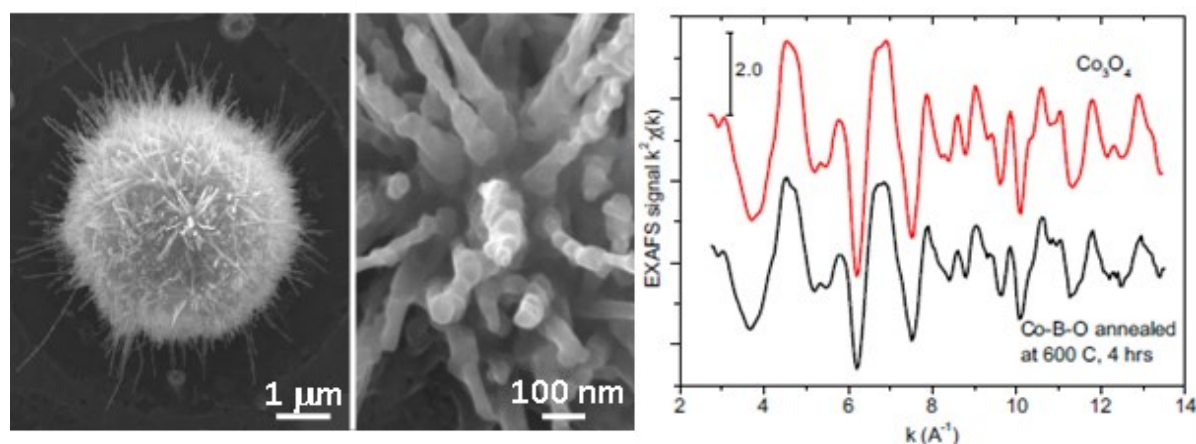


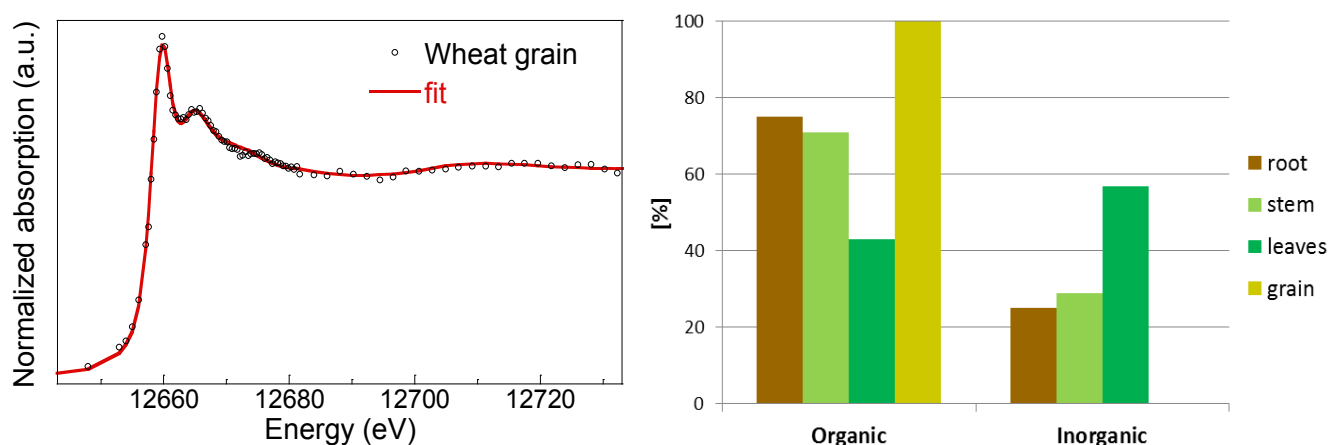
Figure: Left and center: SEM images of  $\text{Co}_3\text{O}_4$  urchin-like particles. Right: EXAFS spectrum of Co-O-B coating on glass after urchin formation upon air annealing at 600°C for 4h, compared to the very similar spectrum of  $\text{Co}_3\text{O}_4$  crystalline powder.

## Selenium distribution and speciation in plant parts of wheat (*Triticum aestivum*) and Indian mustard (*Brassica juncea*) from a seleniferous area of Punjab, India

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The concentration, distribution, and speciation of selenium in different parts of wheat and Indian mustard, grown in a seleniferous area in Punjab, were investigated using synchrotron based (XAS,  $\mu$ XRF) and classical acid digestion and extraction techniques. The analyses revealed a high Se enrichment in all investigated plant parts, with Se levels in the range of 133–931 mg/kg (dry weight). Such high Se enrichment is mainly due to the considerable amounts of easily available Se detected in the soil, which are renewed on a yearly basis to some extent via irrigation. Speciation analysis in soil and plants indicated selenate and organic Se as major Se species taken up by plants, with a minor presence of selenite. The analyses also revealed that the highest Se enrichment occurs in the upper plant parts, in agreement with the high uptake rate and mobility of selenate within plants. In both wheat and mustard, highest Se enrichments were found in leaves. Organic Se species were found in different parts of both plants, indicating that an active detoxification response to the high Se uptake is taking place through methylation and/or volatilization. The high proportion of selenate in wheat and mustard leaves (47% and 70%, respectively) is the result of the inability of the plant metabolism to completely transform selenate to non-toxic organic forms, if oversupplied. Selenium was found to be mainly present in a form that is highly absorbable by human and animal metabolism. In addition to the generally high Se concentration observed in all investigated plant parts, this leads to a considerable health risk for humans and livestock if only local Se-contaminated food is consumed. One positive aspect for human health is the high proportion of methylselenocysteine, which has a low toxicity, but high efficacy towards cancer prevention. The study suggests that to prevent wasting and bring benefits for human health the studied plants could be mixed with plants from non-seleniferous areas to reduce the Se content and obtain methylselenocysteine-enriched food to be exported to Se-deficient areas.



(Left) Linear combination fitting of a XANES spectrum of a wheat grain using the methylselenocysteine (68%) and Se-Cysteine (32%) reference spectra acquired at GILDA-BM8; (Right) proportion of organic and inorganic species in different parts of wheat.

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