ESRF	**Experiment title:** ***Advanced studies of electronic states by soft x-ray resonant Raman scattering***	**Experiment number**: HE1952
Beamline: ID08	**Dates of experiment**: LONG TERM PROJECT from: 3-9/5/06, 25/2/07-5/3/07, 18-24/4/07, 29/8/07-4/9/07, 16-22/7/08, 24-30/9/08	**Date of report**: 19/1/2009
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The present report completes and replaces the one submitted in Oct. 2007.

Report:

The long term project (LTP) HE1952 was intended to pursue our exploration of resonant Raman scattering (RRS) of soft x-rays and of its newest and most advanced applications. In particular we have been focussing on the use of RRS for studying the electronic states of magnetic and strongly correlated materials.
The LTP has two main branches: integrated resonant Raman scattering (IRRS) and high resolution resonant inelastic x-ray scattering (RIXS). We present here a report for both after having used already 2/3 of the beam time originally allocated. Rather than organising the report chronologically we will summarise the main results obtained on each of the two subjects. As stated in the proposal we have restricted as much as possible the technical work and concentrated our efforts on the optimisation of the instrumentation and on some important scientific cases. Our goal was and is to exploit the unique performances of our instrumentation both in IRRS and in RIXS in order to possibly open totally new opportunities in the field of x-ray spectroscopy. We anticipate here that all the runs have been successful: although it was impossible to realise all the experiments originally proposed (mainly for high resolution RIXS) we have a rich set of interesting experimental results. The reference list gives a good overview of our recent results, all based on the LTP.

IRRS (25/2/07-5/3/07)

<u>*Spin resolved dynamics of magnetic Fermi gas*</u>

One of the main issues of the running LTP is to exploit the consequences of what we have seen in our previous LTP on the dynamics of the screening of the intermediate state core hole in magnetic systems [1]. The basic concepts are summarized here for clarity. In resonant scattering, let us consider a convenient scattering channel (e.g., in 3*d*TMs the inner shell channel $2p^63d^n \rightarrow 2p^53d^{\,n+1} \rightarrow 2p^63s^13d^{\,n+1}$ or the valence channel $2p^63d^n \rightarrow 2p^53d^{n+1} \rightarrow 2p^63d^n$) and let us measure the total intensity pertaining to that channel in the chosen emission direction. This integrated signal is measured as a function of the incident photon energy so that an IRRS spectrum is a function of $h\nu_{in}$. Despite the integration, the IRRS spectrum brings information on scattering (a second order process) and differs drastically from the absorption spectrum (XAS). IRRS can be seen as a sort of partial photon yield spectroscopy as well. In our work on elemental Fe, Co and Ni obtained in the previous project [HE1534, and ref. 1] the IRRS magnetic circular dichroism (MCD) in perpendicular geometry gave the evidence that in metallic samples the Fermi gas operates a spin dependent screening of the 2*p* core hole in the intermediate state. We can use the 2*p* lifetime as an atomic stopwatch to evaluate the time scale of that rearrangement of the Fermi gas. In particular we have seen that the screening is faster (it finishes within at most a fraction of a femtosecond) in a wide band system like Fe and Co than in a narrow band system like nickel (typically 2 fs). For these reasons we suggested to measure the effect in magnetic 3*d* TM alloys (Year 1 Milestone on IRRS - Scientific) and in TM metal impurities in magnetic 3*d*TM systems (Year 3 Milestone on IRRS - Scientific). The central idea is that the dynamics of the screening is dictated by the majority component. Thus Ni could reach complete screening when diluted in a wide band systems as opposite to elemental Ni where the screening is not complete within the time scale dictated by the scattering. In the Ni case the effect is expected to be particularly evident since the fully screened Ni in the intermediate case would have basically no magnetic moment. Moreover we anticipated the possible need of improvements in the detection system in our IRRS apparatus based on a multilayer defining the bandpass on the scattered beam (Year 2 Milestone on IRRS - Technical).

All the above goals have been reached successfully. We have been able to improve the sensitivity of the apparatus so that we have anticipated the milestone of year 3 and we have done measurements both on the alloys and on diluted systems. We studied the Co-Ni and the Fe-Ni systems at various compositions ($Ni_{95}Fe_5$, $Fe_{95}Ni_5$, $Fe_{98}Ni_2$, $Co_{98}Ni_2$, $Ni_{95}Co_5$, $Fe_{90}Ni_{10}$, $Co_{90}Ni_{10}$) by measuring both the majority and the minority component. All results confirm the above interpretation scheme. A particularly significant case is the magnetic circular dichroism in perpendicular geometry in Ni diluted in Fe (2% concentration, close to the impurity regime). The main message is clearly shown in figure 1, where the cases of elemental Ni with Ni impurities in Fe are compared. In the shaded area between the L_3 and L_2 edges the elemental Ni shows a non zero dichroism in agreement with our previous measurements [1] while in Ni diluted in Fe this dichroism is drastically reduced due to complete or almost complete screening of the core hole. In spite of the noise the flipping ratio gives the clear evidence of the effect (red line vs. the black line in the upper panel). The data are under analysis and we are working on a model able to give a general rationale on the behaviour vs. composition. Indeed this model has been completed and published [2]. This allowed a much more detailed analysis of the data and in particular of the RIXS data on Co metal which could not be analyzed with the previous model. Moreover tha data on the experiment on the diluted alloys have been analyzed with the new model and presented to the MMM2008 53rd Annual Conference on Magnetism and Magnetic Materials (Austin, Texas, 10-14/11/2008) (on print on JAP issue of 1 April 2009 Vol 105)

"Complete" IRRS experiments

In an integrated experiment as IRRS one renounces deliberately to obtain the whole information because one integrates over a rather wide bandpass in the outgoing energy. However this makes it easier to measure the polarization of the outgoing beam that is hardly measurable in traditional RIXS experiment. Thus a IRRS experiment with measurement of the outgoing polarization is a "complete IRRS experiment" giving access to new information. This was the milestone IRRS -Scientific of year 2 and it has been reached by exploiting also some preliminary results from the preceding LTP [HE1534]. The results are published in ref. [3] whose header is reported in figure 2. See also reference [4] for recent results in IRRS, still belonging to the previous LTP [HE1534].

RIXS (3-9/5/06, 18-24/4/07, 29/8/07-4/9/07, 16-22/7/08, 24-30/9/08)

Technical development.

During the LTP we have further upgraded the instrumentation for high resolution RIXS in the soft x-rays. The dedicated monochromator (PoLIFEMo) has profited from a modification of the grating holder: the new one puts much less stress on the grating itself, avoiding thus to add a slope error to the optical element. The thermal contact has probably become less good, though, so before the end of the LTP we will further intervene on the grating holder by adding a water cooling facility. In fact at present the energy resolution of PoLIFEMo has much improved with respect to one year ago, but it is clearly limited by the thermal power when using both undulator sources of the beam line. The best improvements in terms of combined energy resolution have been found at the Cu L_3 edge, where we passes from 620 meV to less than 400 meV combined instrumental line width, meaning almost 2500 resolving power, which is the maximum we can expect from PoLIFEMo+AXES in the present configuration at 930 eV. Those figures are at present the best worldwide for what we know from the literature.
As promised we have installed the liquid He cryostat, that allows us to regulate the sample temperature from 26 K to 300 K. A diode temperature sensor is currently installed next to the sample, in the vacuum chamber, so that the actual sample temperature is always known with precision.
Instead of the modifications to the entrance slit of AXES promised in the proposal, we have decided to rather built a refocusing stage to improve the optical coupling between the beam line and the spectrometer. In fact having a smaller spot on the sample can help much in increasing the count rate with no consequences on the energy resolution. The design of the optics and of the mechanics has been done and we foresee to install the new spherical mirror, placed after POLIFEMo and just before AXES, in the first half of 2008. Mainly for the low photon energies (450-650 eV) we expect to gain up to a factor of 4 in intensity. In addition the new optics will allow to insert an I_0 detection system after the PoLIFEMo exit slit, thus allowing to measure better XAS spectra in situ.

These goals have been reached in the first semester of 2008. We have installed and commissioned the refocusing mirror before the sample with a gain in intensity of a factor of 3 at the Oxygen edge i.e. only slightly less than expected. Moreover the combination of a new CCD detector (1048×1048 pixel, liquid N_2 cooled, 13.5 micron pixel size) with the new optics before the sample allows to play with the trade-off between intensity and resolution with excellent results. It is possible to work at the Oxygen edge with a combined resolution of 140 meV and at Cu L_3 around 320 meV. This makes the instruments competitive in spite of the availability of the SAXES spectrometer at the ADRESS beam line of the Swiss Light Source (developed jointly by our group, the SLS staff and M. Grioni group at the EPFL). At present AXES is the second best in the world after the Swiss instrument. New procedures for passing from the CCD images to the high resolution spectra, in case of very low intensity, have been developed too.

dd excitations of early 3d metal oxides

Following our previous work on Cuprates [5,6], NiO [7], MnO [8,9] and CoO [10] we have extended to Ti [HE1984,11] and V [12] our high resolution RIXS measurements. In particular some time was dedicated to optimise the energy resolution at the V edge, also in view of two experiments by external users held in 2006 [HE2227, HE2093]. Despite a low count rate the spectra measured on VO_2 during the tests turned out to be very interesting and have already been published in ref. [12], see Figure 3.

A big step forward has been done in the fitting of the spectra with a theory based on a non dispersing excitonic local (single ion) model. This procedure introduced by the Milano group has been further improved in collaboration with M. Haverkort (Max Plank, Stuttgart). The good fittings obtained in Cuprates allow to clarify the positions of the different contributions due to excitation of the Cu 3*d* hole from the x^2-y^2 ground state to different excited states.

Mid-infrared excitations in layered cuprates

Following our pioneering work on *dd* excitations in cuprates [5,6,13], we have devoted much beam time and efforts to the study of low energy excitations in cuprates, in particular the $La_{2-x}Sr_xCuO_4$ family, the $Nd_{2-x}Ce_xCuO_4$ family, and $CaCuO_2$. Thanks to the improved energy resolution and to a careful determination of the zero-energy loss point, we could find that in all those compounds an important spectra weight lays between 200 meV and 600 meV. This is an energy scale typical of magnetic excitations in cuprates, as studied in the past with optical spectroscopies. In optical absorption they fall in the mid infrared region and are thus often known as mid-IR excitations: the assignment to multi-magnon excitations has been the object of an important debate [14]. For the first time we have been able to detect those excitations using RIXS at the Cu L_3 edge. The data set collected in the last two runs is still under analysis, but we can anticipate here that we could find an interesting dependence on the sample doping, but not on its temperature. See figure 4 for some significant examples. In consideration of the difficulty of the experiment, where energy resolution is still a limiting factor, the data collected until now are still to be considered preliminary and further work is needed to fully characterize the mid-IR band in various cuprates.

Indeed further work on cuprates has been done both at the Cu L_3 edge and at oxygen K edge. At the Cu L_3 we have demonstrated for the first time that in the Mid-Infrared region a magnetic contribution is seen with a strong dispersion with the momentum transferred along the basal planes of the cuprates which are parent compounds of the high Tc superconductors (in particular in LCO and CCO). In collaboration with the theorists (J van den Brink, L. Ament and F. Forte) we have demonstrated that bimagnons largely contribute to this excitations [15,16]. Moreover in the doped systems as LSCO a dispersion is still seen with a nonzero excitation at $k = 0$; this is a delicate point still under investigation. Moreover we have seen the counterpart of the effect at the oxygen K edge where the selection rules are different. In particular spin-flip is not allowed because of the absence of spin-orbit in the intermediate state of scattering. The oxygen data are at present under analysis.

In our opinion it is very important having demonstrated that magnons can be seen with RIXS. In fact this is possible on thin films and in the near future also in monolayers whereas the traditional neutron work require very massive samples. This expands drastically the set of materials that can be investigated and paves the ways to the study of spin waves at interfaces. Needless to say this new approach will cross fertilize with neutrons.

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FIGURES

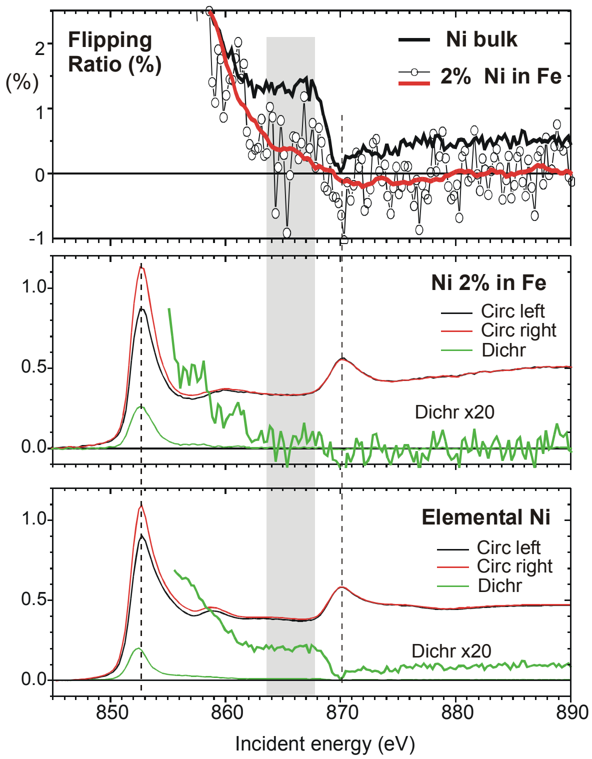

FIGURE 1. MCD in IRRS: results for Ni in different environments. The perpendicular geometry MCD signal vanishes, in the shaded energy region, when Ni atoms are surrounded by Fe, indicating a faster core hole screening process in the intermediate state.

PHYSICAL REVIEW B **75**, 184408 (2007)

Resonant inelastic x-ray scattering from magnetic systems with angular resolutio and polarization analysis of the scattered beam: Results on metallic Co, Fe, and Co ferrite at the $L_{3,2}$ edges

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We present experimental results on resonant inelastic x-ray scattering (RIXS) in perpendicular geometry excited with circularly polarized x rays at the Co $L_{3,2}$ edges in Co ferrite ($CoFe_2O_4$) and Co metal and at the Fe $L_{3,2}$ edges in Fe metal. In this geometry with magnetic samples the incident x-ray beam is perpendicular to the magnetization and the magnetic dichroism is only present in RIXS. We study the 3s-RIXS channel terminating in an inner shell 3s hole and extra 3d electron ($2p^6 3d^n \rightarrow 2p^5 3d^{n+1} \rightarrow 2p^6 3s^1 3d^{n+1}$). The work is based on the measurement of the spectra vs incident photon energy and integrated over the scattered photon energy. We present data resolved in linear polarization of the scattered beam for scattering at 45° and data summed over the outgoing polarizations and taken vs the scattering angle φ. The results show common features in both metals and ionic system: the dichroism scales as $\sin 2\varphi$ while the spectral shape remains constant. The dichroism is maximum when the outgoing linear polarization is in the scattering plane and zero when the outgoing linear polarization is perpendicular to the scattering plane. The presence of the dichroism is due to the breaking of the mirror symmetry of the geometry consisting of the helicity vector of the incident radiation, the sample magnetization, and the linear polarization of the scattered beam. This is expected, in general, when the scattering occurs without rearrangement in the intermediate state. It is noteworthy that this occurs also in the metallic systems, where the scattering takes place with rearrangement due to a spin-dependent screening of the intermediate state core hole. This shows that this rearrangement does not require a more complex vector system in its description. The results show the relation between the selection of the scattering angle and the measurement of the polarization of the scattered radiation. We also present results from a geometry where the dichroism is absent in the emission integrated over outgoing polarization but where it is observed when this polarization is selected.

FIGURE 2

PHYSICAL REVIEW B **76**, 125105 (2007)

Neutral 3*d* excitations in insulating VO_2 as seen with resonant inelastic x-ray scattering at the $V\,L_{3,2}$ edges

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We present resonant inelastic x-ray scattering (RIXS) results on insulating monoclinic VO_2 with excitation across the $L_{2,3}$ edges of vanadium. The spectra measured near threshold in the L_3 region and in the L_2 region show a Raman behavior with a clear peak around 0.9 eV transferred energy. The analysis of the data allows the neutral excitation spectrum of the system up to about 2–2.5 eV to be determined. These excitations are due to *dd* transitions, whose main contributions are discussed. The possibility of $\{d^1;d^1 \rightarrow d^2;d^0\}$ charge fluctuations within a dimer in RIXS is also discussed. The results are compared with available information on optical constants and on electron energy loss spectroscopy. Due to the low symmetry of the system, there is a similarity between the Raman spectrum obtained with RIXS and the optical absorption spectrum.

FIGURE 3

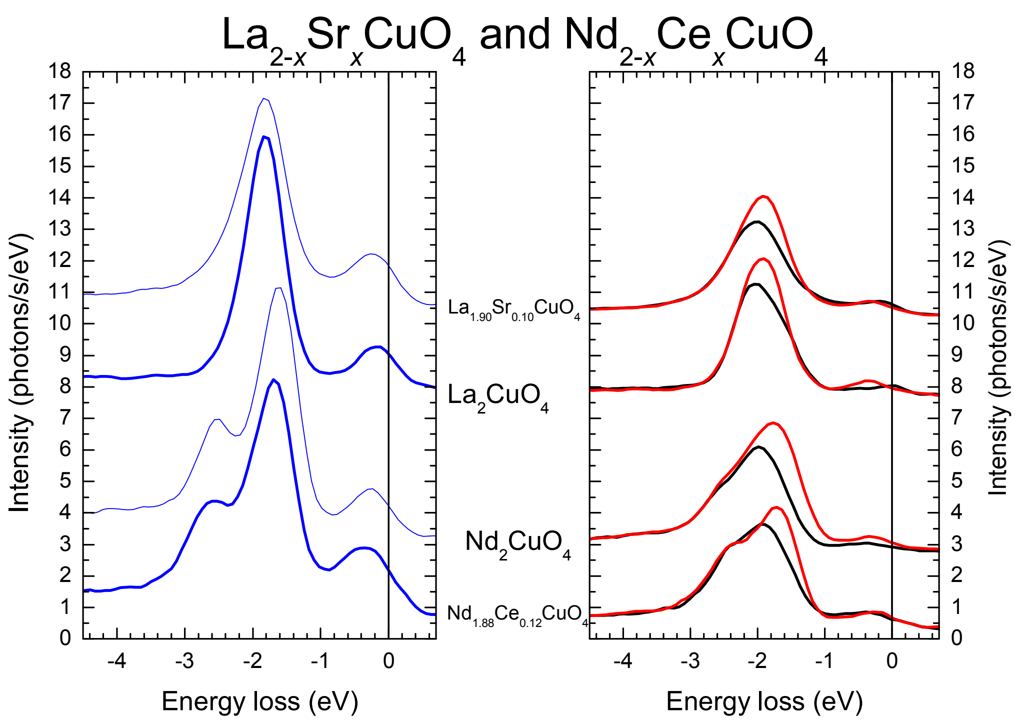

FIGURE 4: an example of the Cu L_3 RIXS spectra measured with 0.4 eV combined line width on some cuprates. The arrows indicate the low energy excitations, corresponding to the mid IR absorption peaks detected in optical absorption. The spectra are sensitive to the incident photon polarization. Left panel: grazing incidence, polarization perpendicular tot he scattering plane. Right panel: normal incidence, polarization perpendicular (black) or parallel (red) to the scattering plane.

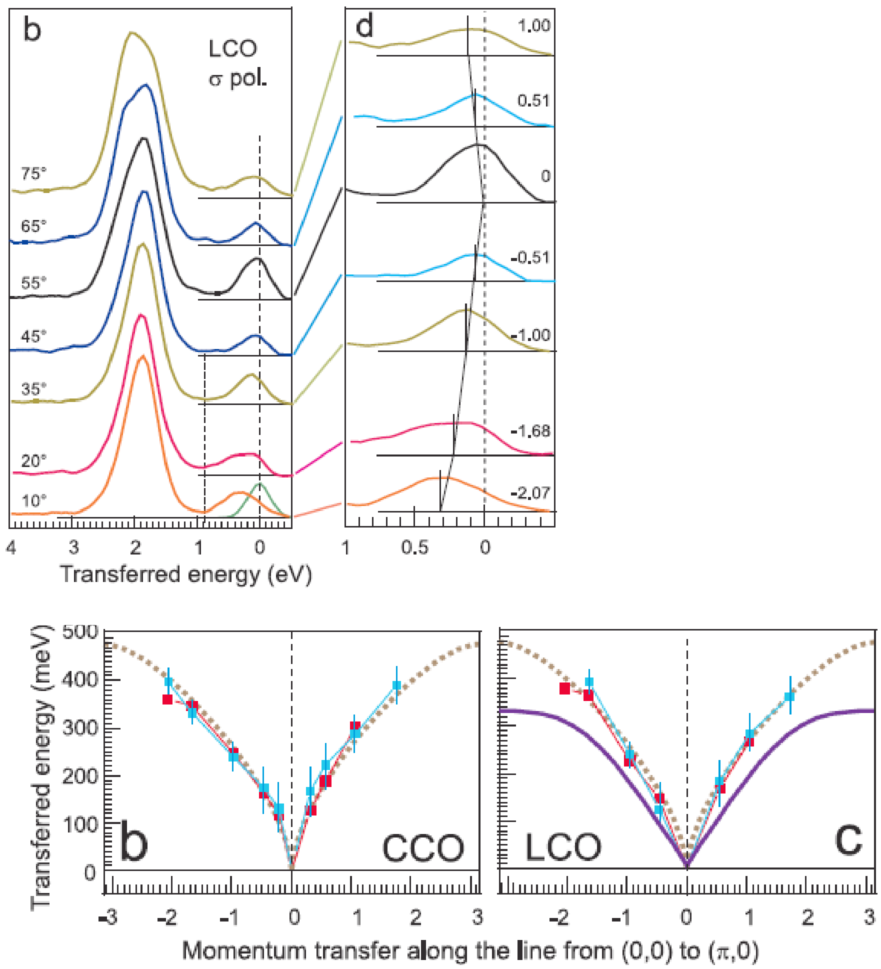

FIGURE 5: LCO raw data and dispersion of the mid-IR feature assigned to magnon (single and double) excitations. [arXiv:0807:1140v1, (2008)]

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Rationale for femtosecond magnetism explored with x-ray core-hole excitation

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In magnetic metals the core-hole excitation by x rays leads to a sudden creation of an impurity that is screened in a spin-dependent way. A scattering experiment in the L_3 region terminating with a 3*s* hole has already been shown to give information on the time scale of this screening process. Here we present a linear-rate model for the analysis of such experiments and apply this to Ni, Co, and Fe metals. This model yields quantitative results that could not be obtained before using an empirical model. The screening time constant in Ni (most likely ~1.5 fs) is definitely longer than in Fe, which presumably is caused by narrow-band effects. The screening time is 0.43 fs in Co, while 0.18 fs is an upper limit in Fe metal. In agreement with the experiment, the model clarifies the variation in the dichroism due to the competition between the core-hole decay and the time it takes to accumulate the screening charge. Finally, the perspectives of this approach are briefly discussed including the importance of future experiments on diluted magnetic impurities.

FIGURE 6

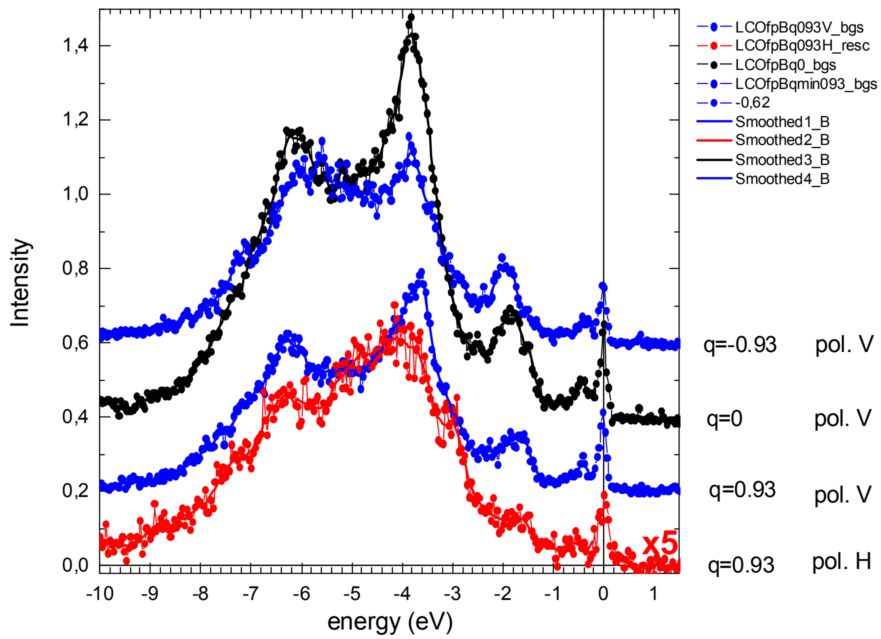

FIGURE 7: An example of O K edge RIXS spectra of LCO, at different values of the momentum trasferred parallel to the ab plane. The data analysis is under way. The spectra are dominated by fluorescence from the O 2p band, but below 2 eV RIXS features appear, having both crystal field and magnetic origin.

Effect of the chemical pressure on bimagnons in antiferromagnetic insulators: $CaCuO_2$ and $BaCuO_2$ studied with Cu-L_3 Resonant Inelastic X-ray Scattering

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Abstract. Substitution of Ca with Ba in $CaCuO_2$ increases the lattice parameter which in turn modifies all interactions between the constituent ions. Here we study the effect on the bimagnon propagating in the Cu-O planes as seen with Resonant Inelastic X-ray Scattering at the Cu-L_3 peak. The bimagnon energy is reduced by a factor of 0.67 ± 0.06 while its spectral weight decreases at least by a factor of 0.5. This scaling is understood from the dependence on superexchange, which is linear in the former case and quadratic in the second. Moreover the scaling of the bimagnon energy can be understood from the distance dependence of the transfer integrals.

FIGURE 8: a short article to be published in Eur. Phys. J. Special Topics.