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 (for convenience the present report covers also the measurements taken in July 95 and already reported).

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Magnetic circular dichroism in L_3 -resonant soft-x-ray inelastic scattering of disordered Fe-Co alloys

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We discuss the information given by dichroism measurements in soft-x-ray resonant scattering from magnetic systems with particular reference to experimental results on disordered Fe_xCo_{1-x} alloys ($x=0.05-0.9$). We show how to recover the scattering cross sections from the raw data and we introduce the emission dichroism correlating with the difference between occupied valence states with opposite spin. We obtain direct evidence of the filling of Fe majority states upon dilution and of the dominant Fe minority states at E_f (bcc structure); this is even more evident in Co due to the greater d -state occupation. The potential and limitations of this spectroscopic technique are illustrated. [S0163-1829(97)51622-5]

The study of electronic states of magnetic systems with circularly polarized x-rays is a new field whose importance is rapidly increasing due to the use of modern synchrotrons. Such studies give electronic and magnetic information of broad interest which often cannot be obtained using other methods, since core spectroscopies are sensitive to the nature of the atomic site and to the chemical species. The major role has been played so far by magnetic circular dichroism in core-level absorption¹ connecting the core levels to the empty states and giving information on the magnitude of the spin and orbital moments by means of sum rules.² In particular soft x-rays are important since they give direct access to the magnetic empty states through dipole selection rules [e.g., at the L edges of $3d$ transition metals (TM's)]. On the other hand the emission mode (dichroism in x-ray emission) connecting occupied states to a core hole has potentially a broad impact in *occupied-state spectroscopy of magnetic systems* introducing sensitivity to the site and to the chemical species. The emission mode, due to its bulk sensitivity and to the possibility of working with insulators, potentially can address problems not accessible by other methods, such as spin resolved valence photoemission. These very general perspectives motivated the efforts along this direction. The first work³ suggested theoretically that one should have a direct insight into the occupied spin resolved electronic states when a magnetic system is excited in a dichroic mode and the energy distribution of the emitted x-rays is measured by integrating over the polarization degrees of freedom of the emitted rays. This is noteworthy since, in the emission mode, the experiment strictly equivalent to the absorption dichroism and giving information on the occupied states implies the measurement of the polarization of the emitted radiation. The theoretical results of Ref. 3 on Fe show that the dichroism in emission basically mimics the energy distribution of the difference between the spin populations (hereafter referred to as "spin unbalance"). The first experimental evidence was given by the measurements on Fe excited with white beam.⁴ The first measurement with resonant excitation at the L edges of $3d$ TM's are given in Refs. 5 and 6. The

pioneering results of Refs. 5-7 demonstrate the feasibility of the experiments. However, much work remains to be done on the connection between the measurements and the properties of the magnetic materials. In particular the potential of this technique and the limits of its applicability still remain to be discussed. In the present paper we connect the measured spectra with the dichroic inelastic scattering cross sections and we discuss their dependence on the experimental parameters showing how to recover information on the *occupied* states. Moreover we show that this spectroscopic technique is particularly useful in dilute systems. As a specific case we present data on disordered Fe-Co alloys showing a promising qualitative correlation between the emission dichroism and the difference in the spin resolved local density of occupied states. In particular we give the direct spectroscopic evidence of the band filling of Fe upon alloying.

The measurements were made at ESRF-BL26 (500-1600 eV range) using a helical undulator. The scattered light was analyzed with a grating spectrograph allowing parallel acquisition in a 40 eV energy interval. The spectrograph was matched to the exit slit of the beamline monochromator with a Kirkpatrick-Baez refocusing optics. We chose ≈ 1.2 eV linewidth to analyze the scattered photons having energy ($h\nu_{out}$) and an ≈ 2.5 eV bandpass in the beam used in excitation (energy $h\nu_{in}$) centered at the absorption peak so that the counting rate integrated over the whole spectrum was above 3 counts/sec in the worst case; the background is below 10% of this value. The circular polarization is 0.8 ± 0.04 .⁷ The spectra were measured by alternating every 5 min the magnetization while keeping the incident polarization constant; during the measurements the field (≈ 2500 G) was kept on. Every two hours the phase of the undulator was changed and the spectra were measured with the opposite light polarization so that the channels were interchanged. No offset in the dichroism baseline was found so the measurements are free of instrumental asymmetries. The samples were magnetized along the surface and the light was incident 20° from the surface, with emission normal to the surface. In the same setup the absorption dichroism was obtained from

the sample drain current. To avoid artifacts due to magnetic stray fields a high-voltage extracting electrode faces the sample (field ≈ 2 kV/cm). Absorption dichroism was measured at various magnetizing fields showing that in the scattering measurements the samples are at saturation within 1–2%. The Fe-Co samples (600 Å thick) were prepared *ex situ* by co-evaporation onto Si substrates and capped with 20 Å Au. The Fe samples were prepared in the same way. The 20% Fe and 5% Fe samples are bcc and fcc, respectively, as determined by x-ray diffraction.

We define the measured dichroism as $(I^+ - I^-)$ with the normalization $(I_{\max}^+ + I_{\max}^-)/2 = 100$ where $I(h\nu_{\text{in}}, h\nu_{\text{out}})$ is the intensity of the measured spectra and we define the scattering dichroism as $(\sigma^+ - \sigma^-)$ with the normalization $(\sigma_{\max}^+ + \sigma_{\max}^-)/2 = 100$ where σ is the inelastic scattering cross section of the transition from the ground state to a final state with a valence d hole. It is important to realize that in general the measured dichroism cannot be interpreted directly as a scattering dichroism as one might think naively. For a given sample each measurement gives a different measured dichroism depending on experimental conditions such as dilution and sample thickness while the scattering dichroism, being an intrinsic property of the material (given a scattering angle), must always be the same. This is easily understood in a model in which self-absorption is neglected. By considering an elemental sample the intensity I of the scattered radiation normalized to the incident one is given by

$$I = \int_0^{s_{\max}} \sigma \exp[-s\alpha_{\text{in}}] ds = [\sigma/\alpha_{\text{in}}] \{1 - \exp[-s_{\max}\alpha_{\text{in}}]\}, \quad (1)$$

where σ is the inelastic scattering cross section, s is the coordinate along the absorption path, α_{in} is the total absorption coefficient at the energy $h\nu_{\text{in}}$ and $s_{\max} = Z/\sin\beta$ (β is the incidence angle with respect to the surface and Z is the sample thickness). With thick samples ($s_{\max} \rightarrow \infty$) one measures $[\sigma/\alpha_{\text{in}}]$ while with thin samples the development of the exponential gives I proportional to σ . Formula (1) leads to a very strong thickness dependence of the measured dichroism, since α_{in} is dichroic in itself.⁸ Only for thin samples does one measure the inelastic scattering dichroism $(\sigma^+ - \sigma^-)$. In a dilute system α_{in} is basically independent of the polarization since it is dominated by the nonresonant absorption by the majority species; thus the measured dichroism of a minority species at high thickness is due to σ in contrast to concentrated systems. Hence, the connection between the scattering dichroism and the measured dichroism is strongly dependent on the dilution and thickness. The self-absorption of the emitted photons exiting from the sample is not dichroic due to the emission normal to the magnetization and this is the reason for choosing this geometry. This simplifies considerably the treatment of self-absorption which will be discussed in detail elsewhere.⁹ In what follows we recover the cross sections by considering also self-absorption. The whole procedure requires the knowledge of the absorption coefficients and of the absorption dichroism. It is important to measure the absorption dichroism with the same setup as in the scattering thus reducing systematic errors and making the knowledge of the absolute circular polarization rate less critical. Our measurements of absorption dichroism are in

complete agreement with the results of Chen *et al.*¹⁰ In order to keep the correction to the data below 15%, formula (1) shows that in elemental Fe the sample must be thinner than ≈ 50 Å with an absorption of about 25% of the light in our geometry. In an alloy a resonant absorption of this order by one component ensures that the recovery of the scattering cross sections is not subject to heavy data handling. Outside these conditions the use of circular dichroism in soft-x-ray scattering, with resonant excitation, becomes more difficult and in many cases probably impossible.

Having clarified the above points it is interesting to apply the method to the Fe-Co alloys. We have studied a variety of compositions covering most of the important cases in Fe-Co magnetism. Although we emphasize mostly the minority component, we also present information on the majority component in spite of the rather heavy data handling. In this case the use of data on the majority component is justified by the availability of very accurate absorption dichroism results to be used in formula (1). The measured spectra and the measured dichroism of Fe in Fe-Co alloys with Fe- L_3 excitation are summarized in Fig. 1(A), while Fig. 1(B) gives the scattering cross sections corresponding to 100% polarization. The corresponding L_3 absorption dichroism is given in Fig. 2(A) [with the normalization $(\alpha_{\max}^+ + \alpha_{\max}^-)/2 = 100$] and can be compared with the scattering cross sections. As expected the data treatment has a large effect in the concentrated systems and little effect at extreme dilution where most of the correction is trivial and comes from the polarization of the incident beam.

The main goal of the present paper is to investigate how far these scattering results can be used to understand the occupied states involved in the deexcitation transition of the intermediate core hole state in the spirit of the initial theoretical suggestion of Ref. 4. To this end we can take advantage of the positional disorder of the samples. When \mathbf{k} is a good quantum number the cross section in general cannot be factorized into an absorption coefficient and a fluorescence yield (i.e., $\sigma = \alpha Y$) due to the restrictions coming from \mathbf{k} conservation as shown in Ref. 11. Here the factorization should be possible since \mathbf{k} is not a good quantum number due to the random occupation of the lattice sites by Co and Fe. On the other hand, another obstacle to the factorization could be due to multiplet effects in the intermediate excited states giving rise to different scattering spectra at different $h\nu_{\text{in}}$. We have clarified this point in a separate experiment on Co- L_3 and we have demonstrated that multiplet effects are not important in measurements with the present bandpass of the incident beam because the scattering cross sections do not depend on $h\nu_{\text{in}}$. Thus we factorize the cross section [i.e., $\sigma = \alpha(h\nu_{\text{in}})Y(h\nu_{\text{out}})$] and we define the "emission dichroism" $(Y^+ - Y^-)$ [with the normalization $(Y_{\max}^+ + Y_{\max}^-)/2 = 100$]. By definition the emission dichroism gives a value referring to the same number of core holes in the two polarization cases removing the effect of the absorption channel in the cross section. The meaning of this definition is very delicate. According to the cross section concept and to the theory of Ref. 3 the sensitivity of the emission dichroism to the occupied states comes from the decay rate $e(h\nu_{\text{out}})$ of the intermediate core hole which reflects the number of final states available to the transition. Thus a measurement integrated over the polarization degrees of freedom of the scat-

tered beam and aiming to emphasize the occupied-state properties must give information on the decay rate and not on the total number of intermediate core hole states. In fact this last case would be a measurement of the absorption dichroism. In a hypothetical system having only the radiative decay channel from the valence band to the intermediate-state core hole the measured spectra integrated over all angles would give necessarily the total number of intermediate holes so that by definition $\int Y d(h\nu_{\text{out}}) = 1$ generating an emission dichroism with the constraint of zero average. This would give a severe deformation of the emission dichroism with respect to the spin unbalance, i.e., with respect to the desired result and would certainly limit the usefulness of the emission measurements. However, in reality the situation is more favorable since other decay channels are also present. In this case the emission dichroism becomes proportional to the ratio $[e/(A+B)]$ where e has the polarization dependence we are looking for, A is the Auger decay rate with valence electrons, and B is the rate of all other inner-shell processes leading to core hole annihilation. The Auger rate A is also polarization-dependent while B is not, since it describes decay of closed shells integrated over all angles and polarization degrees of freedom. Thus the measurements of emission dichroism have the desired property (the proportionality to e) and no longer have the constraint of zero average. Nevertheless a correction is present due to the polarization dependence of A . In general this distortion can be a serious limitation mainly if $A \gg B$ and this general problem will be addressed elsewhere. In the present case the situation is much more favorable since the distortion is limited, A being about 30% of the denominator $(A+B)$ as demonstrated in Ref. 12. Thus we expect that the major features of the occupied spin unbalance are still present in the results. On top of that the results can be modified by angular effects caused by the symmetry breaking due to the detection of the scattered photons at an angle. We show below on a pure experimental basis that the Fe-Co results can be interpreted qualitatively without discussing these angular effects. However, a theoretical work on the angular effects would be very useful in this connection.

In Fig. 1 we see that the scattering dichroism is smaller than the absorption dichroism so that the emission process has in some sense a countereffect with respect to the absorption. This behavior is understandable since the empty valence states are dominated by the minority spin and the occupied states by the majority one. In Fig. 2(B) we give the Fe emission dichroism obtained by factorizing the spectra of Fig. 1 as explained above. The corresponding Co results are given in Fig. 2(C). When Fe is a minority component in the bcc Fe-Co alloys, the calculations¹³ show a filling of the majority Fe spin states so that the Fermi-level region is principally of minority spin. This filling should give rise to the change of sign in Fe emission dichroism near E_F in Fe₂₀Co₈₀. The effect is clearly observed [Fig. 2(B)]. Since there are no majority states at low binding energies, any deformation with respect to the spin unbalance will only change the amplitude of the emission dichroism near E_F . Consequently the inversion will still be seen. Cobalt, having formally one more electron, should show a similar inversion over a broader range of compositions as in the calculations.¹⁵ In fact the inversion is seen in the Co emission dichroism both for Fe₂₀Co₈₀ and for Fe₃₀Co₇₀. The more dilute

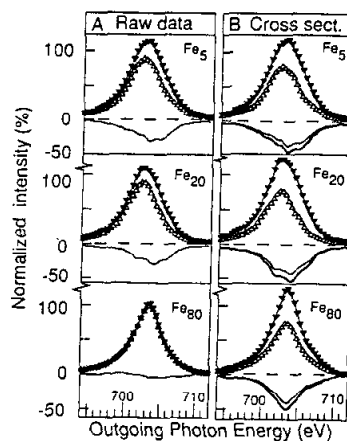


FIG. 1. (A) Measured spectra of scattered photons with L_3 excitation and measured dichroism (thin line) from Fe-Co with different compositions. (B) The scattering cross sections and the scattering dichroism (heavy lines with 100% incident polarization and thin line without polarization correction).

Fe₅Co₉₅ is fcc and no sign inversion of Fe dichroism is seen in the Fermi-level region where the dichroism is zero within the errors in a ≈ 1.5 eV wide region. Although we know of no published calculations of spin resolved partial density of states in this structure, the result is not unreasonable. In the fcc and in the hcp structures, the inversion region with formally eight electrons is expected to be seen in a 1–1.5 eV

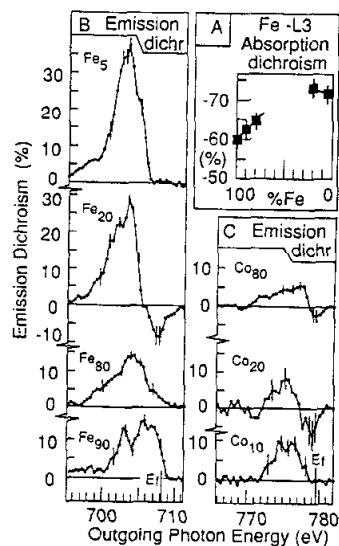


FIG. 2. (A) Fe- L_3 absorption dichroism with 100% incident polarization. (B) Fe- L_3 emission dichroism in Fe-Co with different compositions (C) Co emission dichroism in the same systems. The error bars are given at selected points for readability.

wide region so that it is not surprising that this effect is drastically reduced in Fe which locally has less d charge, although the dilution in Co can increase it with respect to elemental Fe.¹⁴ In Fe₉₀Co₁₀ the disorder induced by Co relaxes the k -selection rule effects so that the Fe emission dichroism is expected to correlate with the scattering dichroism from elemental Fe measured with white beam excitation, i.e., in conditions in which the k -selection rule is not important; this is the case and is shown in particular by the dip at mid-spectrum. Note that a similar feature has been attributed in the model calculations of Ref. 3 to the spin unbalance vs energy. Moreover the trend of the emission dichroism is in qualitative agreement with the composition dependence of the magnetic moments and in particular with the increase of the Fe magnetic moment upon dilution in the bcc structure and with the smaller magnetic moment for Co.¹⁵

In summary we have discussed the perspectives and limitations of dichroism measurements in resonant soft-x-ray in-

elastic scattering and we have shown that this approach is particularly convenient in the study of dilute systems. We have presented a promising correlation between the emission dichroism and the unbalance of the spin resolved occupied electronic states of magnetic materials on the basis of measurements on disordered Fe-Co alloys. Dichroism in resonant soft-x-ray inelastic scattering is sensitive to the bulk, to the nature of the site and to the chemical species and can also be easily applied to insulators.

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¹ *Spin-Orbit Influenced Spectroscopies of Magnetic Solids*, edited by H. Ebert and G. Schütz (Springer-Verlag, Berlin, 1996).

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⁴ C. F. Hague *et al.*, Phys. Rev. B **48**, 3560 (1993).

⁵ L.-C. Duda *et al.*, Phys. Rev. B **50**, 16 758 (1994).

⁶ C. F. Hague *et al.*, Phys. Rev. B **51**, 1370 (1995).

⁷ N. B. Brookes and U. Heinzmann (private communication).

⁸ This strong thickness dependence has been measured on Fe [L.

Braicovich *et al.* (unpublished)].

⁹ L. Braicovich *et al.* (unpublished).

¹⁰ C. T. Chen *et al.*, Phys. Rev. Lett. **75**, 152 (1995).

¹¹ Y. Ma, Phys. Rev. B **49**, 5799 (1994).

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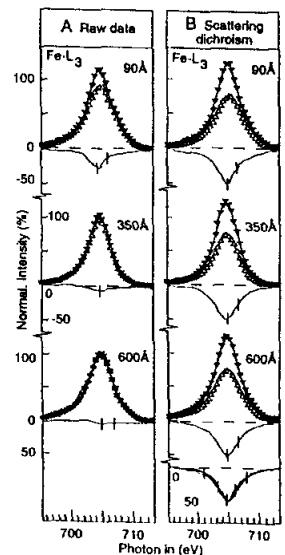
¹³ R. Richter and H. Eschrig, J. Phys. F **18**, 1813 (1988).

¹⁴ Preliminary calculations on fcc Fe₃Co₅ by B. Johansson *et al.* (unpublished) confirm this argument.

¹⁵ See S. Pizzini *et al.*, Phys. Rev. B **50**, 3779 (1994), on the trend of the magnetic moments.

Other results not included in the above publication

Besides the result given above there is another important piece of information which complements the discussion of the dilution effects in dichroism measurements in soft X-ray emission spectroscopy with resonant excitation. The formula given above shows also that the measured dichroism must depend on the thickness of an elemental sample, being all other parameters constant. In fact at low thickness the system is dilute and at high thickness it is concentrated. This clarifies that the method is not appropriate to concentrated samples since the data handling is too heavy, unless one works with very thin semitransparent samples. This idea has been verified with the measurements vs. thickness on magnetic Fe as shown here (panel A and paper in preparation). Very different raw data are obtained vs. thickness from the same material and this is a problem in the use of this approach. The accuracy of the data handling is shown by the fact that the cross sections one obtains (panel B) are always the same being an intrinsic property of the sample. This result defines clearly the applicability range of this resonant spectroscopy and it is not given to suggest an application to concentrated samples but to show the internal consistency of the argument.



the sample drain current. To avoid artifacts due to magnetic stray fields a high-voltage extracting electrode faces the sample (field ≈ 2 kV/cm). Absorption dichroism was measured at various magnetizing fields showing that in the scattering measurements the samples are at saturation within 1–2%. The Fe-Co samples (600 Å thick) were prepared *ex situ* by co-evaporation onto Si substrates and capped with 20 Å Au. The Fe samples were prepared in the same way. The 20% Fe and 5% Fe samples are bcc and fcc, respectively, as determined by x-ray diffraction.

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