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PHOTOEMISSION INVESTIGATION OF INNER SHELLS

### WITH SYNCHROTRON RADIATION

by

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#### PHOTOEMISSION INVESTIGATION OF INNER SHELLS WITH SYNCHROTRON

RADIATION

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While many of the concepts discussed in this paper apply equally well to the solid and to the gaseous state of matter, this paper deals only with inner shells of atoms in solids or at the surfaces of solids. The properties of synchrotron radiation (SR) by which it extends the information obtainable on inner shell states with the important classical sources (He I: 21.2 eV, He II: 40.8 eV, Mg  $K_{\alpha}$ : 1254 eV, Al  $K_{\alpha}$  1487 eV) are (see e.g. Kunz ed., 1979 and references therein):

1. Tunability (any photon energy between 1 eV and 100 keV is in principle available),

2. linear and elliptical polarization,

3. pulsed time structure.

Tunability is by far the most important property, while up to now the polarization and time structure effects in connection with inner shells have only been marginally exploited.

Tunability depends on the monochromators available (Gudat and Kunz, 1979; Wuilleumier and Farge, 1978; Ederer and West, 1980). Monochromators for the 5 - 40 eV range dedicated to PE experiments exist in practically every SR laboratory. Other instruments which are still so few that they are known individually by the SR community operate up to 150, 200, 300, or even roughly 1000 eV. The region above 300 eV, however, is not yet covered in a very satisfactory way. The problems there are connected with monochromator efficiency, resolution and stray light. One single experiment at a fixed photon energy of 8 keV was performed with a low efficiency monochromator (Lindau et al., 1974).

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From these remarks it is evident that mainly electrons from inner shells with binding energies up to 200 eV have been the objects of investigation. All these levels were accessible also before with Mg  $K_{\alpha}$  and Al  $K_{\alpha}$  radiation. The following qualities, however, are added with tunability:

1. The partial atomic excitation cross section can in principle be determined and varied (see e.g. Manson, 1979).

2. The integral cross section and especially the differential (angle resolved) cross section fluctuates with excitation energy, when the atoms are located in crystals or at surfaces due to the nearest neighbor back scattering. This is exploited for structural information (e.g. Kevan et al., 1978; Li and Tong, 1979).

3. The kinetic energy of the emitted electrons  $E_{kin} = \hbar\omega - E_B - \emptyset$  is variable from near zero at threshold to high energy where  $E_B$  is the binding energy and  $\emptyset$  the work function. This implies a variation of the escape depth of the electrons which is in the order of 50 Å at threshold decreasing to ~4 Å at  $E_{kin} = 70$  eV and increasing again to ~50 Å at  $E_{kin}$  1.5 keV ("universal curve", see e.g. Lindau and Spicer, 1974). The variation of the escape depth can be exploited for a variation of surface sensitivity. Unfortunately, the values of the escape depths are still ill defined for individual substances.

4. By varying the photon energy it is hoped to bridge the gap (at least partly) between the sudden and the adiabatic creation of hole states. This implies that long range shielding (observable as intrinsic plasmon excitation (Flodström et al., 1977) and short range shielding (observable as line shape asymmetry (Doniach and Sunjic, 1970, Himpsel et al., 1980) could be influenced.

5. Quasibound states of the excited photoelectron near threshold, e.g. excitons (Lapeyre et al., 1974, Iwan and Kunz, 1978), shape resonances and giant resonances (Lenth et al., 1978) can be tuned in and investigated systematically.

6. The interaction of different excitation channels depends on the excitation energy. Especially the effects of the opening of new channels at threshold on the channels already open can be studied (Kotani and Toyozawa, 1979).

Points 1 to 3 provide information within the one electron picture, while points 4 to 6 are an attempt to systematize the relevant many body effects. The references given cite papers where typical examples or the theoretical background can be found. In this context it was impossible to provide a complete listing even of the most recent work alone.

7. The polarization is mainly exploited in the investigation of valence band electrons, where the band structure scheme applies and symmetry selection rules are easily deduced.

Some very interesting recent results are,

however, available (see e.g. Shirley, 1980). Other effects of the polarization have been observed in the case of photoemission from inner shells of adsorbate molecules, especially when angular resolved photoemission is used (see e.g. Smith et al., 1976). Further, anisotropy in photoemission relative to the direction of the axis of linear polarization is observed by Davis et al. (1980) similar to the effects in atoms.

#### MODES OF SR SPECTROSCOPY

The modes in which photoemission spectra are obtainable with SR (Lapeyre et al., 1977; Kunz, 1978) are extended compared to those with classical line sources. There only EDC's (energy distribution curves) at a few photon energies are measured. Take as a typical EDC (Fig. 1, from Eberhardt 1978) that of Al with features showing the Fermi edge at  $E_F$ , a valence band(VB),volume (p) and surface ( $p_s$ ) satellite plasma lines, core levels like 2p and 2s levels with the low energy shake-up asymmetry (as) characteristic for metals, Auger structures (Auger) and low energy scattered electrons (sc).



Fig. 1. EDC, the asymmetry (as) is marked schematically only.

A series of such spectra at different densely spaced photon energies as it is obtainable with SR allows to follow the intensity and shape changes of all these features as a function of  $\hbar \omega$ . In order to monitor such changes continuously while taking the data, two new spectroscopies were developed: constant final state (CFS) spectroscopy and constant initial state(CIS) spectroscopy. For CFS spectroscopy an energy interval at an energy  $E_{kin}$  of the electron analyzer is fixed and  $\hbar\omega$  of the monochromator is varied. Typically, Auger structures or low energy scattered electrons are monitored this way as a function of photon energy.

a) Monitoring the low energy scattered electrons, like yield spectroscopy, where all the electrons are collected, usually reproduces the absorption coefficient (Gudat and Kunz, 1972).

b) The Auger yield monitors the partial excitation crosssection of those inner levels for which the hole state is decaying via this channel. Modifications can occur usually near threshold due to changing partitioning with competing decay channels. Usually, these partial excitation spectra are characteristic of the surface layer of a sample due to the small escape depth at typical Auger energies.

c) As is easily verified, all CFS spectra must contain a contribution which includes the features of an EDC with the exception of the Auger structure, i.e. CFS spectra monitor core and valence states and the satellite features.

For obtaining CIS spectra a synchronous variation of  $\hbar\omega$  and  $E_{kin}$  is required so that  $E_{B} = \hbar\omega - E_{kin} - \phi$  is kept constant.  $E_{B}$  is tuned to certain features like e.g. d state satellite structures , or core levels.

This spectroscopy is well suited to study partial cross-section variations of core or well defined valence band features. The interaction of different excitation channels are favorably investigated this way (Allen et al., 1978).

This CIS technique requires considerable effort in instrumentation. It depends on a good calibration of the monochromator and in its advanced state involves a microprocessor for coupling monochromator and electron analyser. For those features which are superimposed on a background in the EDC's a background subtraction involves at least running one other spectrum just next to the feature under investigation. The technique is not yet fully developed and available only with a few instruments. It is, nevertheless, probably one of the most promising recent achievements in photoelectron spectroscopy (Lapeyre et al., 1977).

Both CFS and CIS spectroscopies depend on an accurate normalisation to the photon flux for a quantitative evaluation. This is not easy to obtain and up to now one of the weakest points in many investigations. For several physical conclusions one can do without such quantitative spectra. The knowledge of all structural features in the primary spectrum, however, is indispensable. Such structures "shine through" into the CFS and CIS spectra and can be confusing. The following examples are only a small selection from what has recently been achieved with SR.

#### SURFACE EFFECTS ON CLEAN SURFACES

Since the surface atoms experience a potential different from the bulk, because of a lower coordination number, their core levels are expected to be shifted if only initial-state effects are considered. Different relaxation around the core hole at the surface and in the bulk is one of the possible complications. From appearancepotential spectroscopy lower binding energies were claimed for Ni surface atoms by Houston et al. (1973), but this result was challenged by Webb and Williams (1974). Figure 2 shows an attempt by Eberhardt et al. (1979) to see such an effect on an Al(100) surface. Tuning the kinetic energy of 2p emission from roughly 5 eV to roughly 40 eV with respect to  $E_F$  should decrease the escape depth from 25 Å to about 4 Å. No shift within  $\pm$  40 meV was observed. The broadening of the 2p structure in Fig. 2 is a genuine surface effect. Its origin is not yet completely clarified.



Fig. 2. Broadening of Al 2p levels from surface atoms.

Fig. 3. Changes of the W(110) 4f<sub>7/2</sub> spectra with polarization and contamination.





Fig. 5. Shifts in the position of the LIII line of Fig. 4 as a function of doping and photon energy (see Eberhardt et al., 1978).

## Fig. 4. Experimental observation of the shifts in the $2p (L_{III}-L_{1I})$ core lines of silicon with doping. a) $n=10^{14}$ cm<sup>-3</sup> b) 8 x 10<sup>19</sup> cm<sup>-3</sup> c) $p = 1.6 \times 10^{20}$ cm<sup>-3</sup>

XPS of Au 4f by Citrin et al. (1978a) showed an enhanced shoulder at lower binding energy which was attributed to a surface peak. Then measurements by Duc et al. (1970) with SR clearly demonstrated such a surface peak in the W(110)  $4f_{7/2}$  spectra as shown in Fig. 3. The peak shifted to 0.3 eV lower binding energy. It is enhanced (curva a) with p-polarized light compared to s-polarized light (curve b) similar to the classical surface photoemission for free conduction electrons. The peak diminishes on adsorption of H<sub>2</sub> and O<sub>2</sub> (curves c and d). Van der Veen et al. (1980) found a similar behavior with SR also for the Ir 4f levels. Moreover, they could demonstrate different shifts at crystallographically different surfaces.

The varying escape depth with  $E_{kin}$  is also exploited in the investigation of semiconductor surfaces (Eberhardt et al., 1978). Fig. 4 shows as an example binding energy shifts of the Si 2p level on highly p and n-doped samples. Fig. 5 gives depth profiles extracted from these data. Investigations on semiconductors include also Schottky barrier formation (e.g. Chye et al., 1978).

#### SURFACES WITH ADSORBATES

The photoemission from a core level of an atom or molecule adsorbed on a surface is pronouncedly modulated. This modulation originates from a diffraction of the emitted electron wave from the substrate atoms. In this respect, photoemission is closely related to LEED (see e.g. Liebsch, 1974). We mention especially the normal photoemission measurements by Kevan et al. (1978) (see also Shirley, 1980) on the system c (2x2) Se or Ni(100) together with calculations

of peak positions for different adsorption geometries by Tong and Li (1979). The data suggestively favor the hollow site of adsorption of the Se atoms.

It should be mentioned here that this technique is not exhausted with using the normal emission configuration. The interpretation of all these data, quite similar to the situation with the equivalent LEED techniques, depends on the availability of reliable model calculations. There are some advantages over LEED, the most important being lower destruction of adsorbates, no long-range order is required and low coverages are accessible to investigation under favorable circumstances.

Angular integrated photoemission which is equivalent to the socalled "surface extended absorption fine structure" (SEXAFS, see Citrin et al., 1978b) was studied by Margaritondo and Stoffel (1978). This technique gives nearest neighbor distances rather than binding site geometries.

#### RESONANT PHOTOEMISSION

For several years now the photon energy dependence of the differential cross-sections is used in photoemission with synchrotron radiation in order to disentangle the contributions from different atomic states to the valence band of compound solids (see e.g. Eastman and Freeouf, 1975). Recently, it was found that under favorable circumstances there is an even more powerful method available. This applies especially to those materials which contain atoms with partially filled shells like e.g. rare earth metals and transition metals. It is well known that transitions like e.g. the 4d excitations from the rare earths lead to giant inner well resonances in the absorption cross-section. The reason is a range of quasi bound states above the ionization potential in the inner well region of the combined attractive Coulomb and repulsive centrifugal potential (Dehmer et al., 1971). This is depicted in Fig. 6. These excitations can decay by mechanism A (tunneling of the electron into a free propagating wave state). This mechanism is considered by theorists as being responsible for the broadening of the resonance lines. The state, however, can also release its stored energy again completely in a recombination decay of the electron and the hole and transfer it to a one electron ionization from a state with lower binding energy, e.g. 4f state (process B).

It is interesting to note that the released energy is exactly equal to the stored energy independent of photon energy (unless shake-up occurs). Thus, the ionized electron in the photoemission spectrum is indistinguishable from an electron directly excited from the 4f state. If process B is not negligible, an enhancement of the 4f photoemission line in the region of the giant 4d resonance must be observable. Such a stealing of oscillator strength was observed with Xe gas (West et al., 1976), a case where one could



Fig. 6. Inner well resonances and resonance decay (schematic).

Fig. 7. (a) Yield spectrum of Gd<sub>0.67</sub> La<sub>0.33</sub> P<sub>5</sub>0<sub>14</sub>.
(b) Photoelectron EDC's of the valence-band region.

only marginally call the 4d absorption peak a resonant phenomenon. It turns out that the effect is much stronger in the rare earths (Lenth et al., 1978).

Figure 7 shows as an example yield and EDC curves of  $Gd_{0.67}$ La<sub>0.33</sub> P<sub>5014</sub> a laser material. The yield spectrum displays the La and Gd giant resonances at 118 and 148 eV, respectively. The EDC's taken outside the resonances show structure in the valence band which is based mainly on the oxygen 2p states. Nothing is indicat-

ing which of these structures could be ascribed to the 4f state. In the EDC No. 6, however, one peak is increased by an order of magnitude. This is clearly the resonance transfer of excitation energy to the 4f states at the Gd atoms quoted above. Nothing alike happens in the EDC No. 2 where the resonance is excited at the La atom. The energy transfer obviously is a localized process. Here the La 5s and 5p states are enhanced.

This resonant photoemission process is very pronounced and easy to observe when a tunable synchrotron radiation light source is available. The investigation of rare earth compounds rather than pure metals was very useful for establishing this effect. In the meantime, the effect was observed with rare earth metals Ce by Johansson et al. (1978) and Kalkoffen (1978), Yb by Alvarado et al. (1979) and Johansson et al. (1980). A good demonstration of the potential of CIS spectroscopy in this context was published by Allen et al. (1978) on the problem of mixed valence surface states on Sm metal.

Figure 8 shows a plot of the intensity of the Gd 4f peak in Gd metal as a function of photon energy by Gerken et al. (1980). The resonance profile is almost perfectly fitted by a Fano resonance profile over a wide photon energy region. As was demonstrated in detail by these authors, the yet unidentified large peak at about 135 eV in the electron excited Auger spectrum Fig. 9 is clearly due to this elastic decay channel of the giant resonance peak. Such a



Fig. 8.

Intensity of the 4f photoemission peak (X) for different photon energies.

decay mechanism was held responsible for this peak in the Auger spectra of Gd already by Dufour et al. (1976). Since electron excitation corresponds roughly to white light excitation, ordinary photoemission structures are smeared out and are therefore unobservable in Auger spectra. Resonances like the one in Gd, however, decay into electrons of well defined kinetic energies. In this case, the peak in the Auger spectrum at 135.0 eV (Fig. 9) is shifted relative to the resonance peak at 148.4 eV (Fig. 8) by the sum of the work function of the analyser (roughly 5 eV) and the mean binding energy of the 4f levels in Gd (8.5 eV). A detailed analysis of the two types of spectra of Gd by Gerken et al. (1980) and Gerken (1979)





Fig. 9. N<sub>4,5</sub> Auger spectrum of Fig. Gd.

Fig. 10. Normal-emission EDC's of Ni(111) and Ni(110) taken in (67 eV) and off resonance (55 eV) with the 3p excitations. The multielectron shake-up structures are marked by arrows.

served to explain several of the finer structures in the Auger spectra of Gd metal and oxidized Gd. It is clearly demonstrated that these Auger structures are a general feature of the rare earths. Many other materials will contain related structures in their Auger spectra, especially, if excitations to bound or quasibound states occur.

In 1977 Guillot et al. observed a very pronounced resonance of the "6 eV peak" in Nickel which subsequently aroused considerable interest and is obviously a peak not corresponding to a band feature in the Ni valence band, but rather a shake-up structure of a not yet undisputedly clarified configuration. The atomic nature of such peaks was well proven in a series of experiments on metal phtalocyanines (see Iwan et al., 1980, and refs. therein) where the isolated metals produce resonating d band satellites.

Such theoretically more complicated resonances demonstrate that resonance photoemission is a wide field. Most of the references on work relevant for this phenomenon and its relation to phenomena in elements adjacent to Ni are given in a recent paper by Martensson and Johansson (1980). Fig. 10 shows a result by Eberhardt and Plummer (1979) from which one can see that not only the 6 eV peak undergoes enhancement near the threshold of the Ni 3p excitation, but a whole series of peaks at higher "binding energies". They are attributed to shakeup processes. Further, it was demonstrated by Barth et al. (1979) that different parts of the Ni d band are coupled to the 3p excitation differently.

#### INTERCHANNEL INTERACTION

The resonance phenomena discussed up to now are only a prominent example of a more general phenomenon, the interchannel interaction. Even in metals with closed shells as e.g. Au, a breakdown of the one electron model of independent inner shells with separate uncorrelated excitation cross-sections is observed. Barth (1979) has collected data on the  $\sigma(5d_5/2)/\sigma(5d_3/2)$  ratio as shown in Fig. 11. This ratio should be 1.5 if the d peaks superimposed on the Au valence band are interpreted this way with respect to their atomic origin. The deviations have been attributed before to solid state and other phenomena. Barth (1979) correlates the observed strong fluctuations with the threshold of the 5p and 5s shell excitations which is quite suggestive from Fig. 11. The experimental results were confirmed by Barth et al. (1980) and extended to Cu.

The observation of an intensity ratio is experimentally simpler than the measurement of absolute intensities. It is however in many cases not the information required. Reliable absolute measurements will probably reveal even more phenomena in this direction.

In conclusion, one can state that the general trend in photoemission spectroscopy is directed towards taking not only peak positions but also peak intensities serious. Considerable physical insight can obviously be gained from such results. The experimental obstacles are, however, not easy to overcome. Mentioning only a few: accurate monitoring of photon flux is required, energy dependent transmissivity of electron analysers must be known and the escape depth of photoelectrons as a function of  $E_{kin}$  must be determined for individual materials.

![](_page_13_Figure_0.jpeg)

Fig. 11. Ratio of the two spin orbit 5d partners in the Au valence band.

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