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GRAZING INCIDENCE MONOCHROMATOR FLIPPER

by

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ABSTRACT

FLIPPER is a monochromator with fixed entrance and exit beams covering the energy range from 20 eV to 500 eV used for photoemission measurements with synchrotron radiation.

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I. INTRODUCTION

The grazing incidence monochromator FLIPPER[1] was originally installed in the old synchrotron radiation laboratory at the storage ring DORIS at DESY. Because of its successful operation the monochromator was reinstalled in the new Hamburger Synchrotronstrahlungslabor HASYLAB at DORIS. The optical and mechanical principles remained unchanged. All optical components were renewed. Owing to the improvements of the optical components the useful photon energy range was extended from 250 eV to 500 eV and the photon flux behind the exit slit of the monochromator could be raised by about two orders of magnitude compared to the version operated in the old laboratory. At the same time we obtain the same high resolution and efficient suppression of higher order radiation as before.

The sample chamber behind the monochromator is well equipped for photoemission measurements. It contains a commercial double-pass cylindrical mirror analyzer (CMA) with an integrated electron gun for Auger Electron Spectroscopy. An X-ray source additionally provides Al-K_{α} radiation. Samples may be prepared either in the sample- or in a seperate preparation chamber. The complete set-up of monochromator, sample and preparation chamber is shown in Fig. 1. Synchrotron radiation induced photoemission measurements are controlled by a microprocessor which allows the application of the special photoemission techiques, applied in connection with synchrotron radiation, namely constant-initial-state (CIS) and constant-final-state (CFS) spectroscopy.

II. OPTICAL PRINCIPLE AND MECHANICS

Synchrotron radiation from DORIS is deflected twice by two mirrors before it enters the monochromator chamber. The specifications of the optical components are given in Table 1. The FLIPPER accepts 0.75 mrad of radiation horizontally and 0.25 mrad vertically. The mirror magazine with 6 plane mirrors and the rotatable grating for scanning the wavelength is shown in Fig. 2. The mirrors with the more grazing angles of incidence are used for the higher photon energies, higher orders are efficiently suppressed.

The mirror covering the desired photon energy range can be inserted into the beam in a reproducable position to deflect the radiation onto the grating. The rotation of the grating is monitored with an accuracy of 0.28 sec of arc by optically measuring the motion of a rod pushing against a lever arm connected with the grating axis. A parabolic mirror with a focal length of about 1010 mm focusses the radiation dispersed by the grating onto the exit slit. The exit slit is integrated into the sample chamber in order to allow a distance of only 75 mm to the sample resulting in a spot size of about 2 x 0.7 mm.

A photodiode can be moved into the photon beam behind the exit slit of the monochromator[2]. Two photocathodes may be used alternatively which allows a control of the aging of the cathode material under radiation. For the photon intensity measurements of the monochromator Au is used as the cathode material. The absolute photoelectric yield below 150 eV is taken from Lenth[3], above 150 eV the Au yield is extrapolated using

the theoretical relation between yield and absorption from Ref. 3 and the absorption from Ref. 4. All optical components were renewed after reconstruction except the parabolic mirror which is now coated with Pt. The range of photon energies could be extended from 250 eV to 500 eV because all reflective surfaces of the beamline are now coated with metals (see Table 1). A comparison to former measurements at the FLIPPER shows a gain in intensity by about two orders of magnitude for the whole photon energy range. From Fig. 3 it is obvious that with time the reflectivity of each mirror is reduced for higher photon energies, most drastically for mirror S1. At lower energies the reflectivity is occasionally even slightly increased. We attribute this to the coverage of the reflective surfaces with a contaminating layer of Carbon.

During the shut down of DORIS in the beginning of 1982 we put in a larger grating with a fully ruled surface and renewed all mirrors in the monochromator chamber. The mirrors (except S4 and S5) have two differently coated segments which can alternatively be used to deflect the beam. The most recent photon flux measurement is shown in Fig. 4. All mirrors except S1 are now coated with C on one of the two segments It is remarkable that for photon energies higher than 200 eV Pt shows a notably better reflectivity than Au.

III. ENERGY CALIBRATION OF THE MONOCHROMATOR AND SOME EXPERIMENTAL RESULTS

Transmission spectra of the Al-L_{III}-edge at 72.72 eV (Ref. 5) are taken with the Au photodiode for three mirrors (S2, S3, S4) in order to have known calibration points. The rest of the calibration procedure is performed by using the 4f-photoemission lines of Yb. With the grating position at 72.72 eV we take a photoemission spectrum of Yb in the 4f-region and thus effectively determine the binding energy of the 4f ($^2F_{7/2}$) bulk peak. Yb is an ideal substance for the calibration since it is easy to prepare by evaporation and has a sharp peak ($\sim$ 0.05 eV HWHM). Due to the low asymmetry of the peak it shifts only minimally when the resolution of the monochromator varies. The 4f-cross section of Yb is high enough for the whole photon energy range used at the FLIPPER. Additionally the bulk and surface emission of Yb is well separated (see Fig. 5). Thus it allows us to calibrate any position of the grating with any mirror in place by taking a photoemission spectrum without changing the analyzer resolution and the position of the Yb sample. The only parameter varied is the retarding/accelerating voltage at the entrance grids into the CMA analyzer measured by a digital voltmeter of high accuracy. A computer programme evaluates these calibrations using the grating equation. It sets up a table of photon energies for all mirrors and all grating positions and accurate values for the adjustable parameters in the grating equation. The latter serve to programme the microprocessor to control the voltage of the analyzer for the CIS-spectroscopy (synchronization of photon energy and kinetic energy of the photoelectrons). One advantage of this calibration procedure is that it relies only on one calibration point (Al-L_{III} at

72.72 eV) and would be corrected at a later time if this value should be improved.

In the following figures (Fig. 6 - Fig. 12) some experimental results at the FLIPPER are illustrated which are explained in the figure captions.

Acknowledgement:

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Table 1 : Optical components of FLIPPER

Dimensions (length, width, height) (mm)	Material	Angle of incidence	Coating 80/81	Coating 82	Manufacturer	Remarks
440 x 20 x 60	Cu	86°	Canigen		Zeiss	beam deflection horizontal
160 x 40 x 35	Zerodur	85.75°	Au		Zeiss	beam deflection vertical
100 x 88 x 25	glass BK 7	86°	Pt	Pt/Au	Zeiss	mirror 1
100 x 88 x 25	glass BK 7	82.5°	C	C /Au	Zeiss	mirror 2
60 x 88 x 25	glass BK 7	78°	Au	C /Au	Zeiss	mirror 3
60 x 88 x 25	glass BK 7	70°	C	C	Zeiss	mirror 4
60 x 88 x 25	glass BK 7	57°	C	C	Zeiss	mirror 5
60 x 88 x 25	glass BK 7	40°	W	C /Au	Zeiss	mirror 6
50 x 30 on blanc: 60 x 40 x 15	glass	variable	Au	-	Astron	plane gratings 1200 lines/mm, 1.5° blaze
65 x 40 x 15	glass	variable	-	Au	Astron	
120 x 40 x 15	glass	84.3°	Pt		Optical Surf. Ltd.	parabolic focussing mirror

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Figure Captions:

fig. 1 Set-up of the FLIPPER monochromator, sample and preparation chamber (schematically)
SR synchrotron radiation, M mirror, G grating,
PM parabolic mirror, ES exit slit, X X-ray source,
CMA double pass cylindrical mirror analyzer,
S sample, MA manipulator, PC preparation chamber,
TS transfer system.

fig. 2 FLIPPER mirror magazine with 6 plane mirrors and the plane grating (SR synchrotron radiation).

fig. 3 Photon flux of the monochromator FLIPPER after re-installation at HASYLAB (Sept. 1980), after three months measurements (Nov. 1980) (nearly identical except mirror S1) and one year later (Oct. 1981). The given intensities and resolutions are valid for a 200 μm exit slit. The resolution can be improved by reducing the slit width to 1/3 - 1/4.

fig. 4 Photon flux in June 1982 with the same parameters as in fig. 3 (partially renewed elements, see text).

fig. 5 Photoemission spectra of Yb in the valence band (VB)-region at different photon energies showing the bulk (b) and the surface (s) 4f-doubletts.
Surface shifts on RE metals are part of the programme performed at the FLIPPER [6,7,8].

fig. 6 High resolution energy distribution curves (EDC's) on clean Na-metal
The spin-orbit splitting of the 2p-levels is 140 meV.
The fits are calculated by using Doniach-Sunjic-line-shapes for the bulk and surface emission including background and convolution with the instrumental resolution (from ref. 9,10).

fig. 7 EDC's of the 5d-valence band of Au with photon energies above the Carbon-K-edge. The doublet structure can be assoziated with the $5d_{5/2}$ and $5d_{3/2}$ states, respectively (from ref. 14).

fig. 8 $5d_{5/2} : 5d_{3/2}$ - intensity ratio of solid Au (compare fig. 7),
- o Johansson et al. (ref. 11),
- • our data.

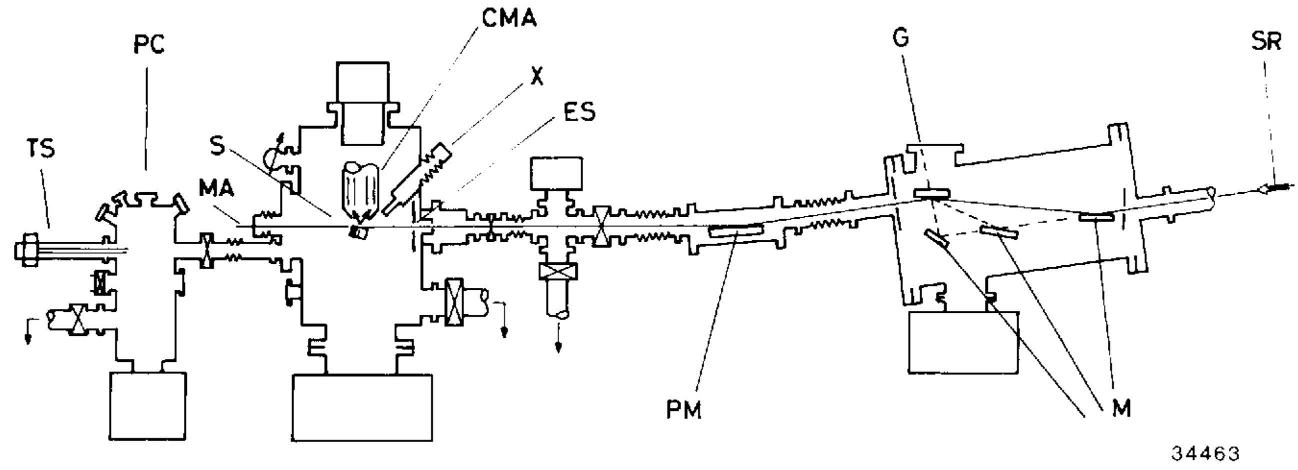
The modulations that extend over the entire energy range have been discussed controversary in the literature [11,12]. The correspondence to features of the inner shell cross-sections (broken curve, from ref. 13) favors an interpretation in terms of intershell interactions. The $4f_{7/2} : 4f_{5/2}$ - branching ratio (x) is obviously not affected (from ref. 14).

fig. 9 Absorption yield and VB-intensity (CIS) of Ti in the region of the $L_{II,III}$ - edge.
The 3d-resonance shows the 2p-3d intershell interaction [14].

fig. 10 Photoemission measurements of Eu exposed to 1 L O_2 showing a Constant-Final-State (CFS) spectrum and two 4f resonance curves in the region of the 4d → 4f excitation. The different lineshapes of the CIS spectra allows the identification of the valence structure to the final states $4f^5$ (Eu^{3+}) and $4f^6$(Eu^{2+}). From this it could be shown that oxidized Eu metal contains divalent and trivalent Eu atoms [15].

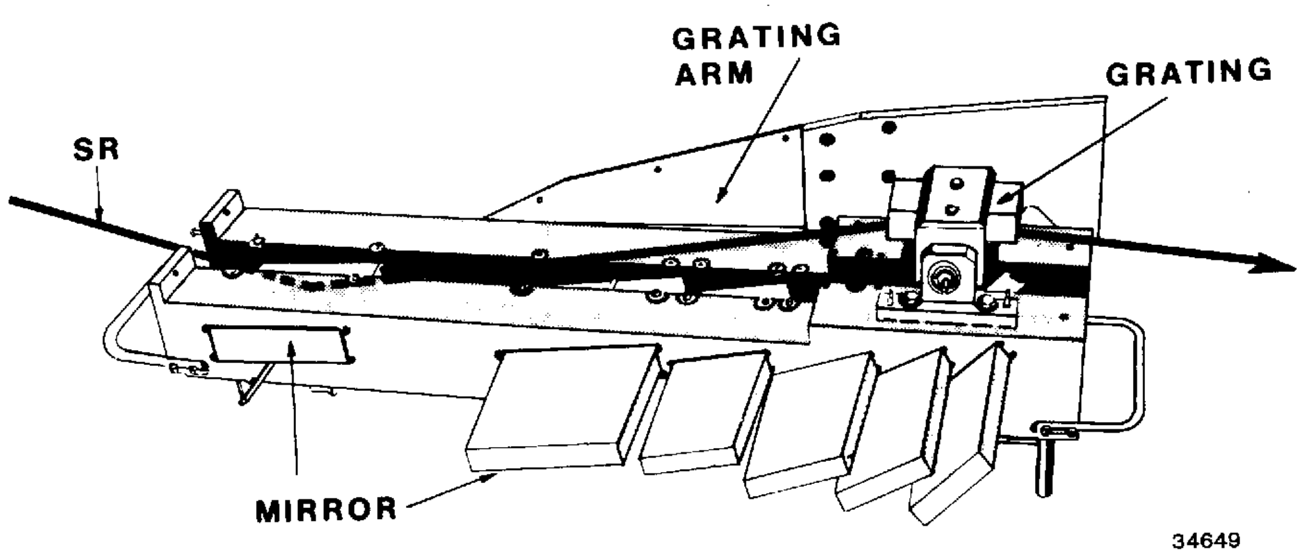
fig. 11 XPS spectrum of LiF -(100) measured at the FLIPPER with Al - K_α radiation [16].

fig. 12 $N_{4,5}$ Auger spectrum of Gd in 1st and negative 2nd derivative (energy of primary electrons: 3 keV) (from ref. 17).



CMA
PC
X
G
SR
ES
TS
S
MA
PM
M
34463

Fig 1



GRATING
ARM
GRATING
SR
MIRROR
34649

Fig 2

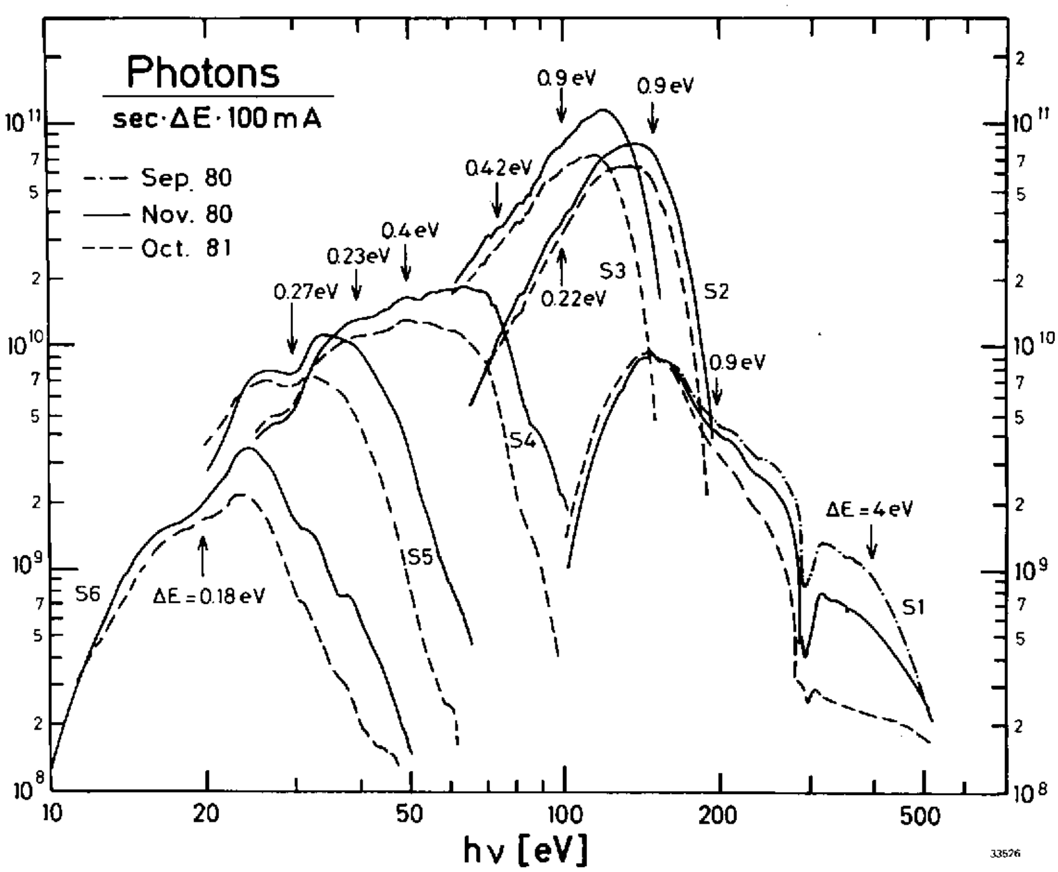


Fig 3

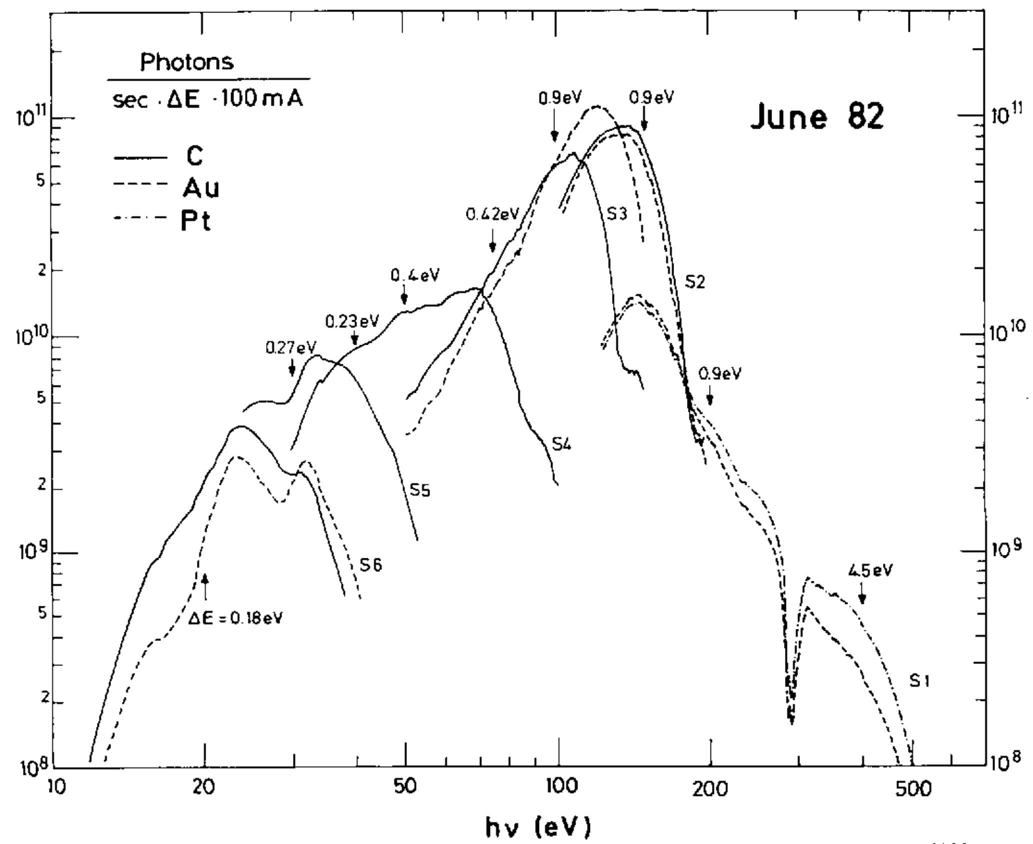


Fig 4

34332

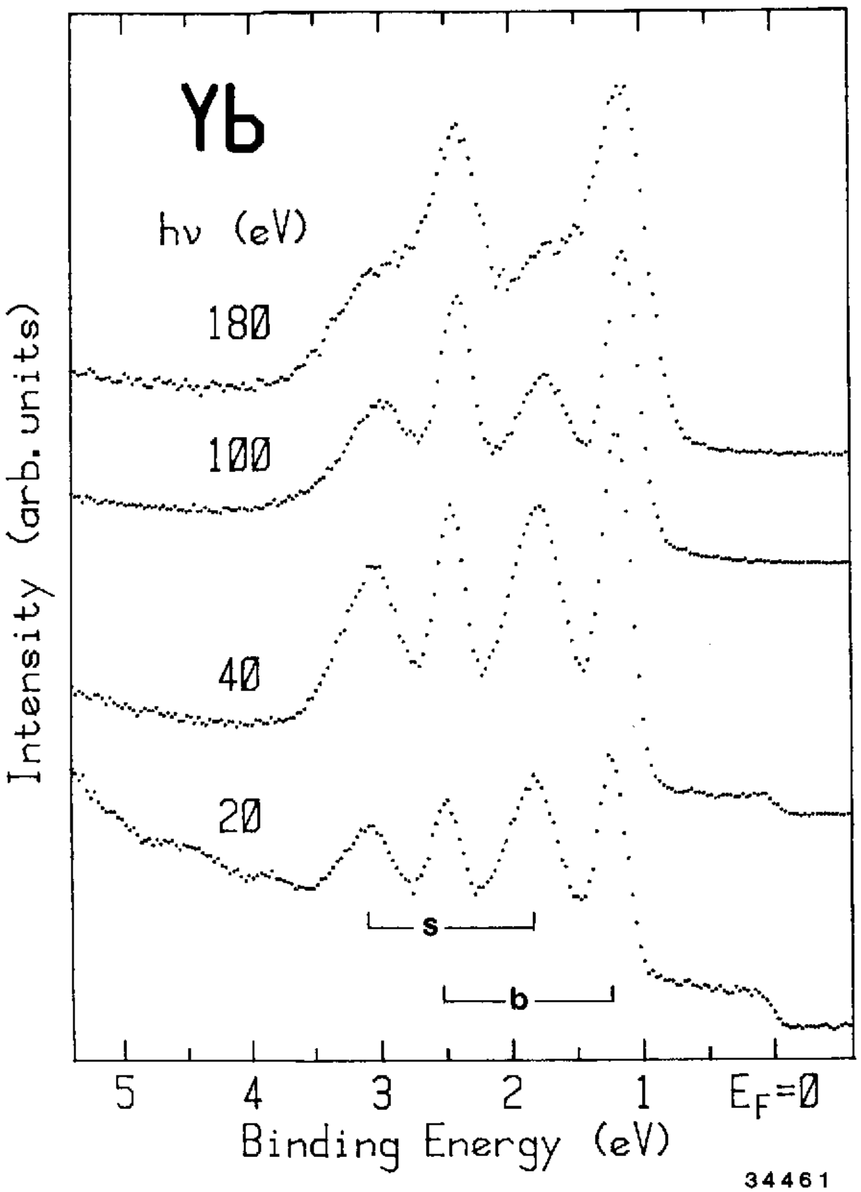

Yb
hν (eV)
180
100
40
20
s
b
Intensity (arb. units)
5
4
3
2
1
E_F=0
Binding Energy (eV)
34461

Fig 5

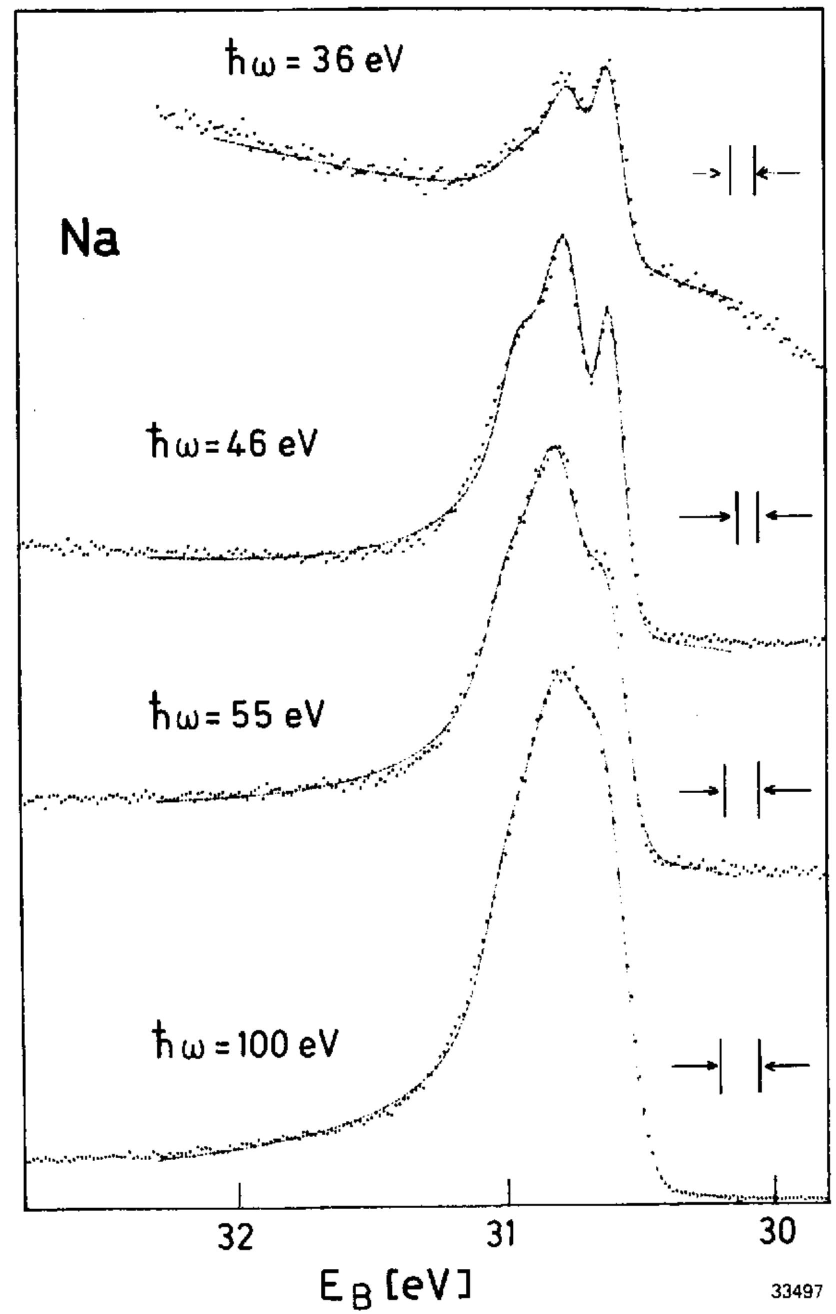

ħω = 36 eV
Na
ħω = 46 eV
ħω = 55 eV
ħω = 100 eV
32
31
30
E_B [eV]
33497

Fig 6

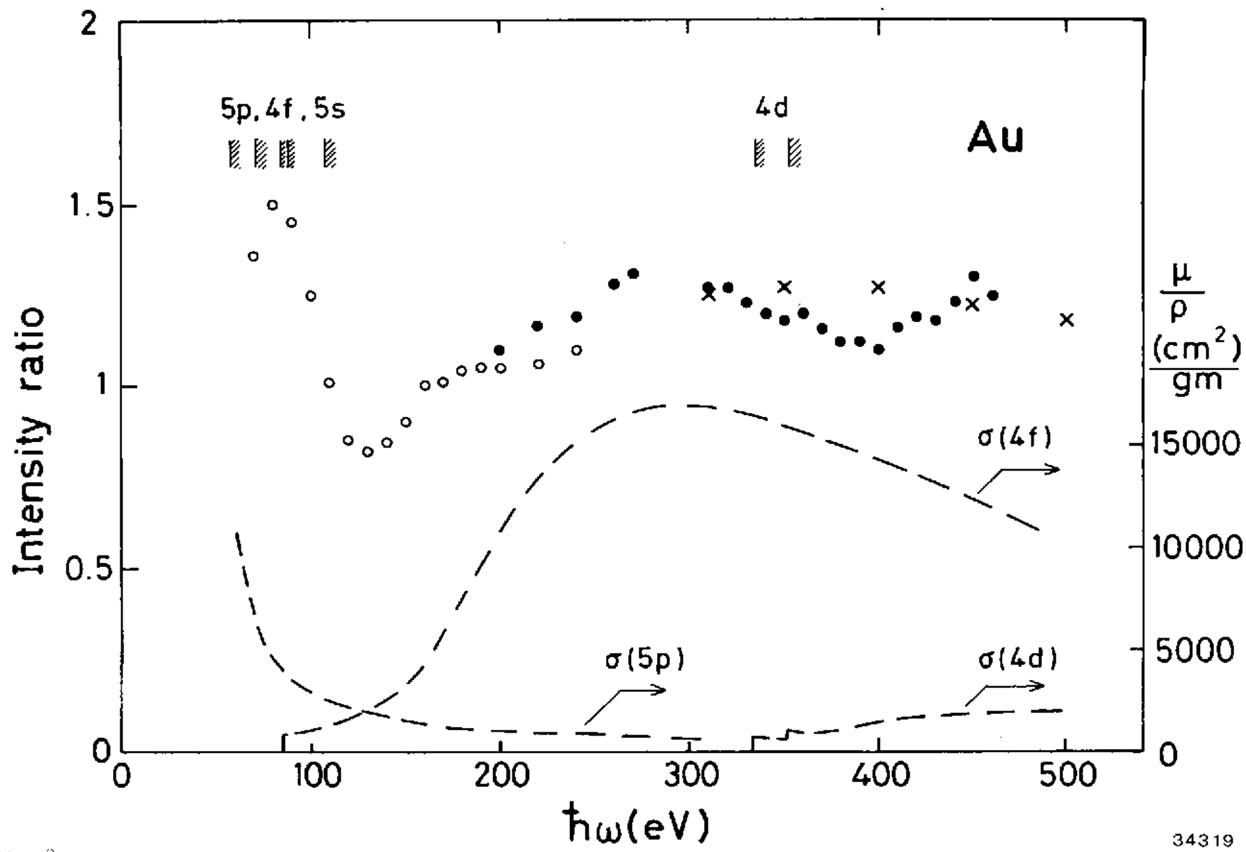

5p, 4f, 5s
4d
Au
Intensity ratio
μ/ρ (cm²/gm)
σ(4f)
σ(5p)
σ(4d)
ħω(eV)
0
0.5
1
1.5
2
100
200
300
400
500
5000
10000
15000
34319

Fig 8

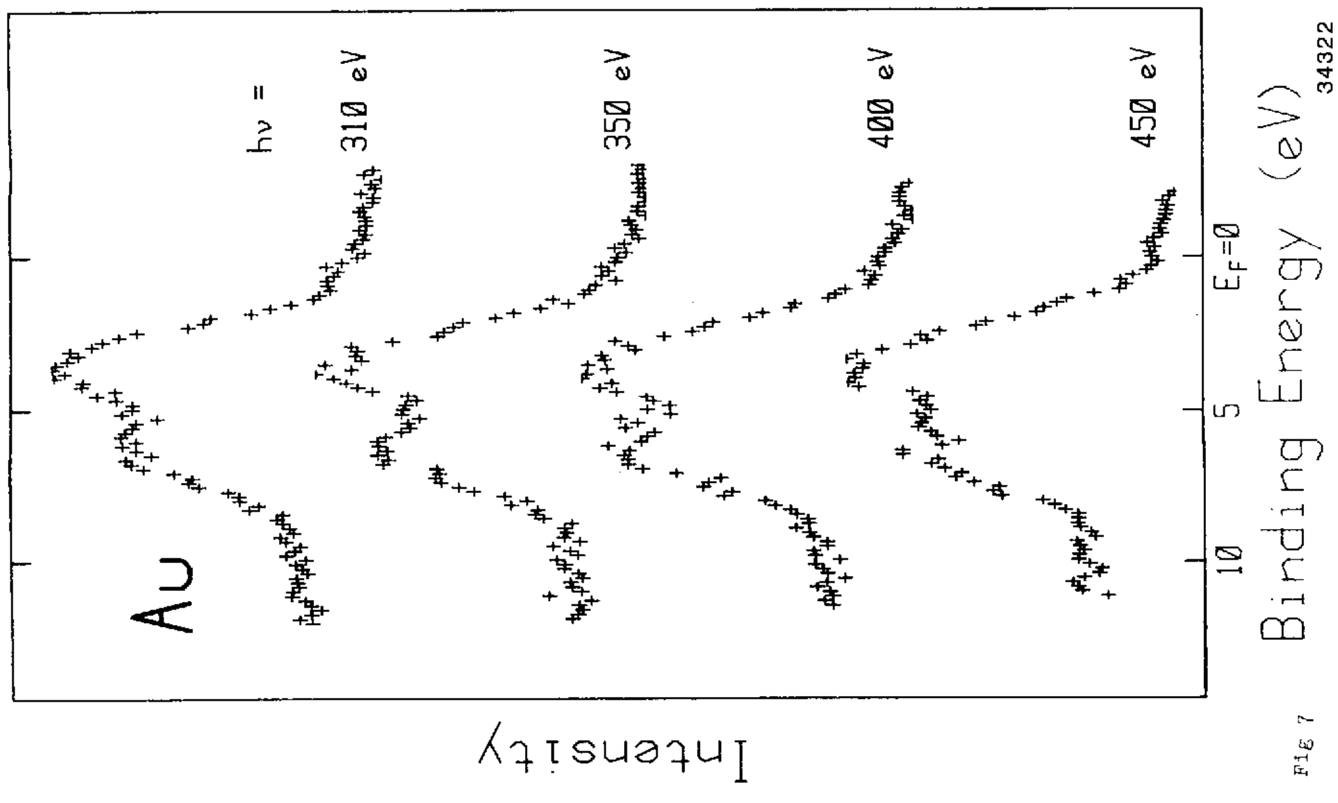

Au
hν =
310 eV
350 eV
400 eV
450 eV
E_F=0
5
10
Binding Energy (eV)
Intensity
34322

Fig 7

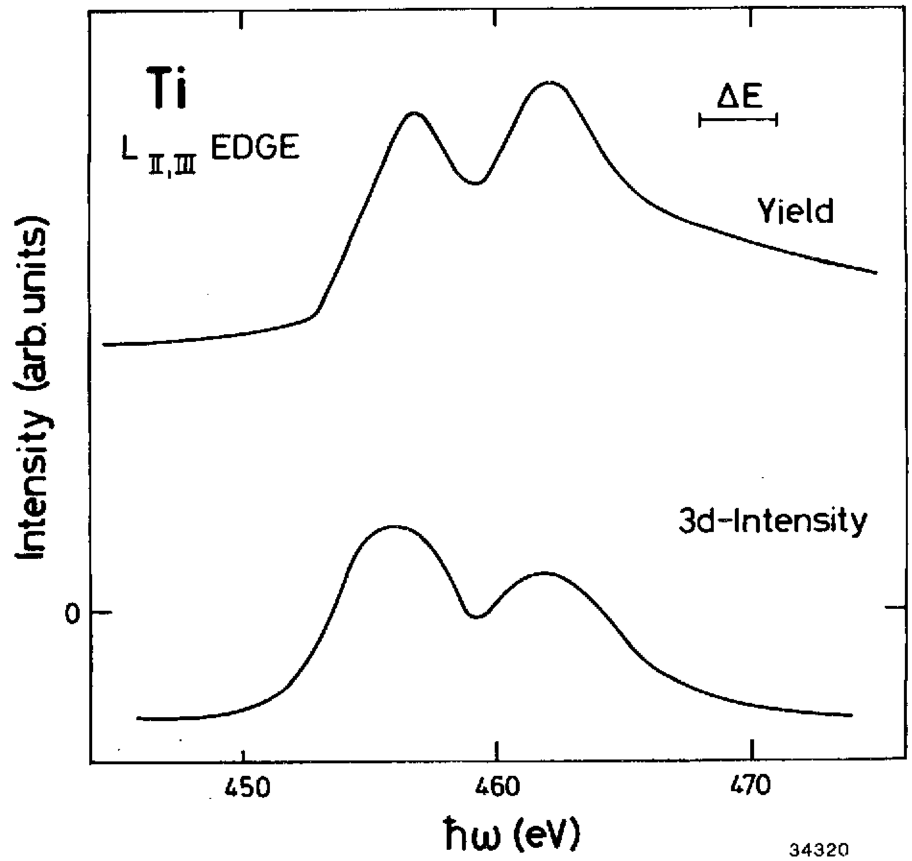


Fig 9

34320

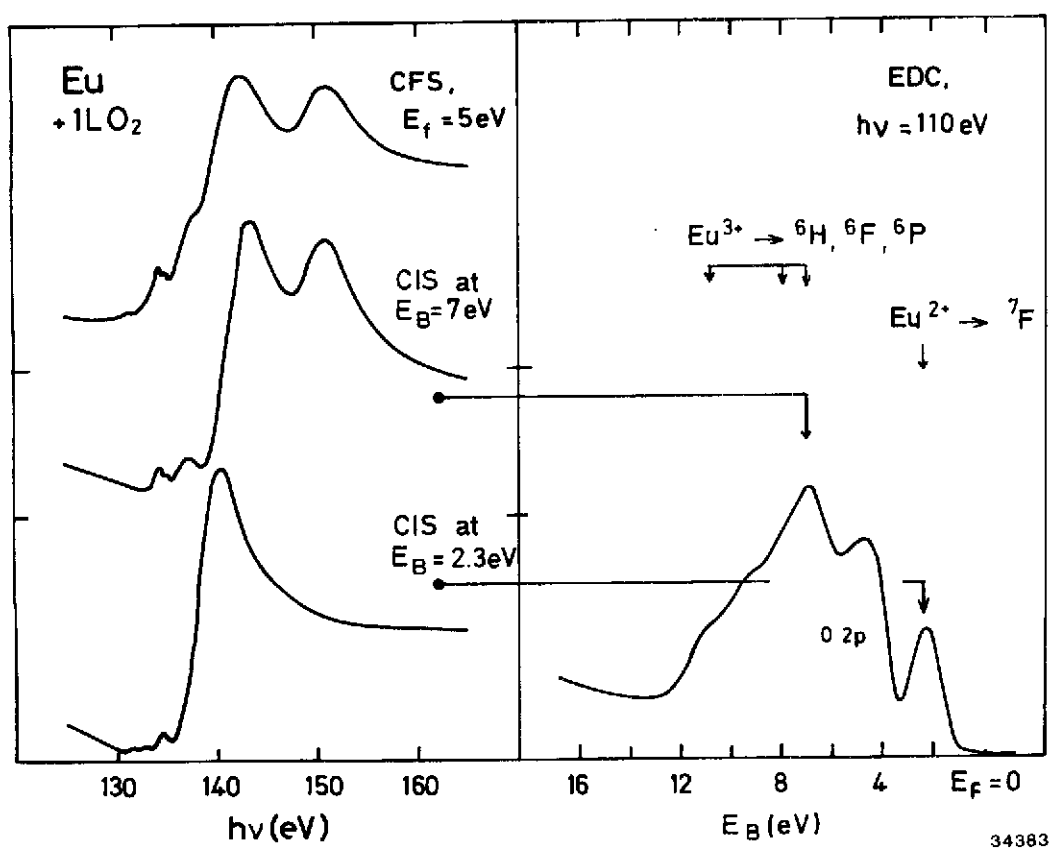
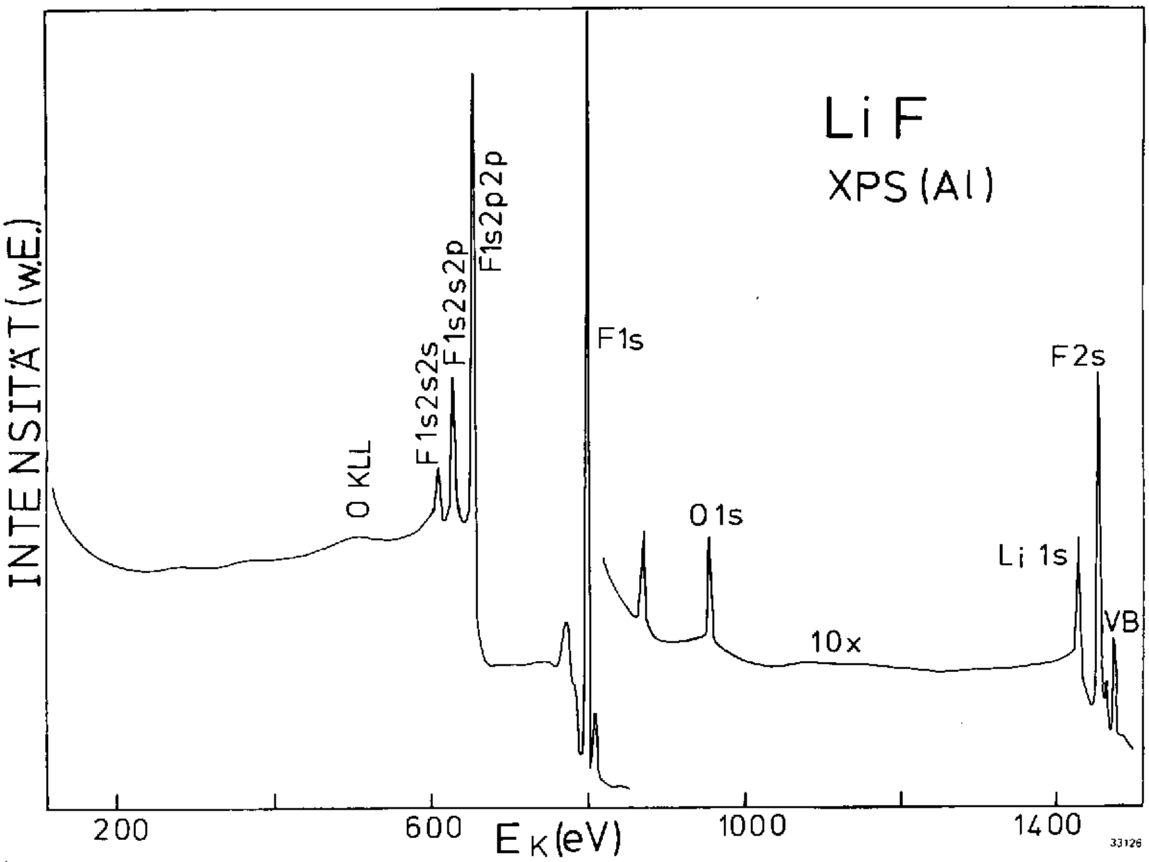


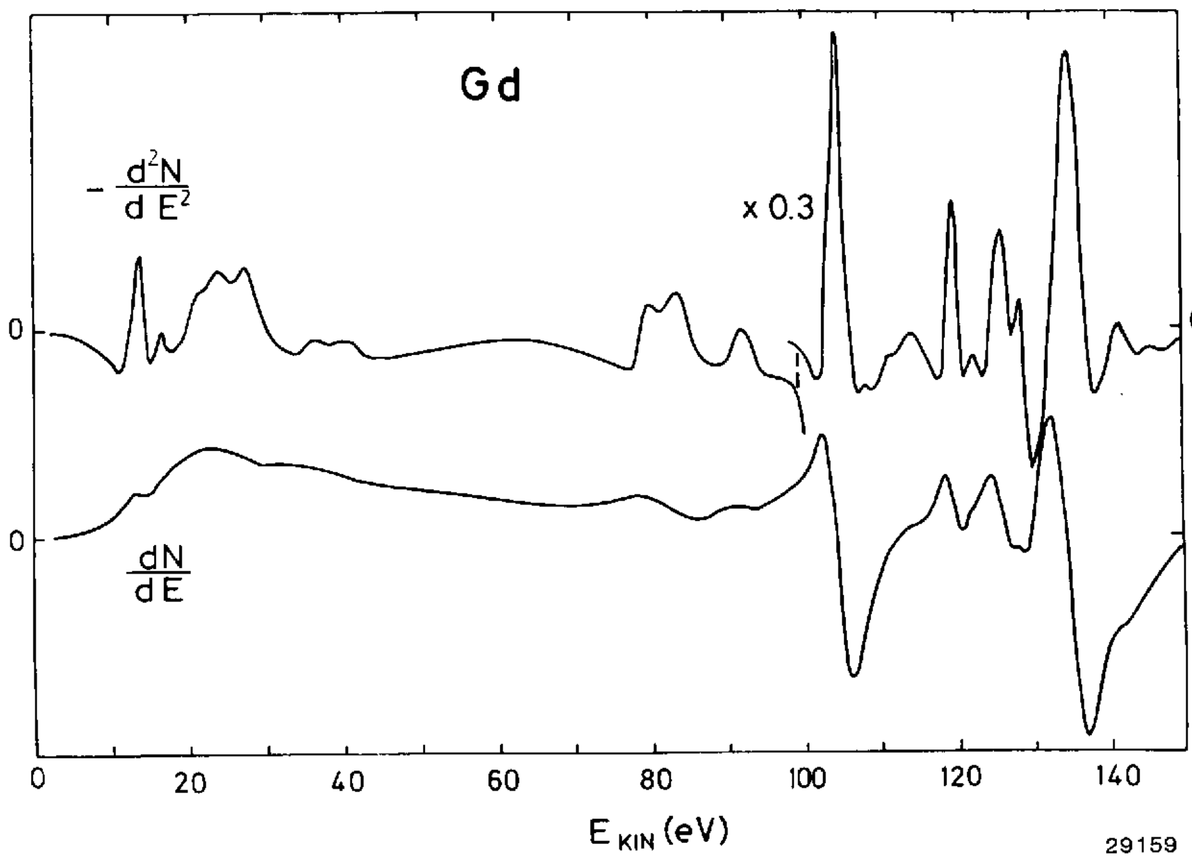
Fig 10

34383



Li F
XPS (Al)
INTENSITÄT (w.E.)
O KLL
F1s2s2s
F1s2s2p
F1s2p2p
F1s
O1s
10x
Li 1s
F2s
VB
200
600
1000
1400
E K (eV)
33126

Fig 11



Gd
$-\frac{d^2N}{dE^2}$
x 0.3
0
0
$\frac{dN}{dE}$
0
0
0
20
40
60
80
100
120
140
E KIN (eV)
29159

Fig 12