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A Rotating Beam Splitter for the Extreme Vacuum Ultraviolet

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A rotating beam splitter for the extreme vacuum ultraviolet

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We have built a rotating-mirror beam splitter at grazing incidence which was used to measure directly (with minor corrections) the transmissivity of thin films in the photonenergy region 20 - 280 eV. The intensity fluctuations usually encountered when using electron accelerators as light sources were efficiently eliminated. The same instrument could be used to measure differential transmissivities $\Delta T/\bar{T}$ with an accuracy of a few tenth of a percent. In another arrangement a deflecting mirror which is incorporated as the photocathode into an open electron multiplier served as a very efficient beam monitor against intensity fluctuations.

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The continuous spectrum emitted by high energy accelerators in the extreme vacuum ultraviolet (synchrotron radiation¹) is presently used most successfully for the measurement of transmissivities and secondary processes on solids and gases. However, large fluctuations of the electron beam current (at DESY occasionally up to 50 % and usually not less than 10 %) and fluctuations of beam geometry constitute a limiting factor for the measuring accuracy. The usual method applied to monitoring the source intensity at the DESY laboratory is to insert an insulated sheet of metal into part of the synchrotron radiation beam in front of the monochromators. The photoemission current is amplified and serves as a reference signal. A monitor located in front of the monochromator responds linearly in first order to fluctuations of the electron-beam current only. Positional and directional fluctuations of the beam exert a different influence on the light entering the monochromator and on that hitting the monitor.

Using a monochromator² which emits a spatially fixed monoenergetic photon beam in the energy range 20-280 eV we have built a device which allows to cancel these fluctuations almost completely. The apparatus is used in three different arrangements. In one mode of operation a mirror reflecting the monochromatized beam behind the exit slit is mounted as the cathode of an open photomultiplier giving off an excellent reference signal. In another mode the monochromatized radiation is split into two equivalent beams by a rotating mirror. This mode serves either to measure transmission curves directly or to measure the difference between two transmissivities. In the latter mode the experiment is equivalent to a modulation experiment and therefore these investigations also explore the possibilities of performing modulation measurements with a pulsating synchrotron radiation source.³

Section 1 explains the principle of operation, its main part is a discussion on the proper choice of the frequency at which the beam splitter should operate. Section 2 gives instrumental details, while in Section 3 the electronics for the three individual modes of operation is described and examples of measurements are shown.

1. Principle of operation

The boundary conditions for our problem are given by the time structure of the radiation emitted by the synchrotron and by the wavelength region under investigation. Moreover, the fact that we have to put the instrumentation into high vacuum will cause problems. The time structure of the electron current I_0 is shown schematically in Fig. 1. Radiation is emitted only after the electrons exceed a certain minimum energy which occurs typically 2-3 msec after injection and depends on two parameters: maximum energy of the electrons and wavelength of the synchrotron light used for the investigation.¹ About 10 msec after injection, electrons are extracted at maximum energy for use in high-energy physics experiments. After a pause of 10 msec a new cycle is initiated, usually with a different number of orbiting electrons. This causes a fluctuation of the source intensity which can amount to as much as $\pm 50\%$ and will typically be not less than $\pm 10\%$. After monochromatization we can use a fixed beam tunable in the energy range 20-280 eV photon energy. For covering this full range of energies, our beam splitter must use only mirrors at grazing incidence.

We have designed an arrangement as shown in Fig. 2. Monochromatic radiation falls onto a gold coated mirror M_0 at a grazing angle of 4° . This mirror is mounted into a detector D_0 yielding a reference signal S_0 . The deflected beam

is split by a rotating grazing-incidence mirror M_1 (4° grazing angle) having one reflecting and one open segment. The frequency of rotation is ω_M . The proper choice of ω_M will be discussed below in detail. The beam passing the open segment is reflected by another mirror M_2 at the same angle of incidence. Both beams pass absorber films F_1 and F_2 and hit two equivalent detectors yielding signals S_1 and S_2 . Three modes of operation are possible (see also Fig. 1).

(1) The rotating mirror M_1 is stopped at the reflecting position. Then the ratio of the averaged signals \bar{S}_1 and \bar{S}_0 is recorded. In measuring \bar{S}_1/\bar{S}_0 with and without an absorber, transmissivities are obtained.

(2) The signal \bar{S}_1/\bar{S}_2 with an absorber in the upper beam No. 1 gives directly the transmissivity^u T .

(3) The signal $2(\overline{S_2 - S_1})/(\overline{S_2 + S_1})$ is proportional to $\Delta T/\bar{T}$, a quantity giving useful information on small differences in transmissivities. In this mode of operation a signal S_+ is formed by superposition of S_1 and S_2 . The signal which is in phase with the rotation of the beam splitter (at frequency ω_M) is proportional to ΔT , while the signal at $2\omega_M$ is proportional to \bar{T} (see Fig. 1 and Ref. 5).

Since the signals are measured using lock-in amplifiers, the question arises as to which frequency is best suited for operation of the beam splitter. This is the general problem of two-frequency modulation. There is one given frequency (50 cps) determined by the time structure of the synchrotron, and we need to make a good choice for the second frequency. With the rotating mirror used in our arrangement we cannot use much higher frequencies of revolution than 50 cps. On the other hand very low frequencies as, e.g., 1 cps give too large contributions due to $1/f$ noise.

Several arguments show that synchronous beam splitting at a repetition rate of $\omega_M = 25$ cps is the best choice. The phase is adjusted such that alternative light pulses are directed into the upper and lower beams. The critical part of the motion, the switching from the mirror to the open segment section, occurs while there is no synchrotron light emission (Fig. 1).

The choice of $\omega_M = 25$ cps can be justified especially with respect to the needs of mode (3) where a small signal at ω_M ($\nu\Delta T$) has to be detected in the presence of a large signal at $\omega_0 = 50$ cps (\sqrt{V}). Lock-in amplifiers using wide-band passive filters pass a signal frequency of ω_0 also at reference frequencies $\omega_0/(2\nu+1)$ (odd subharmonics) of a relative amplitude⁶ $1/(2\nu+1)$. Therefore 16.66 cps, 10 cps, 7.1 cps ... and frequencies near these have to be avoided. On the other hand, harmonics of 50 cps at 100, 150, 200 ... cps must not be used. Synchronous operation at $\omega_0/2 = 25$ cps appears to be the best choice. One word of caution should be said: A proper balance⁷ of the positive and negative cycles of a lock-in amplifier is necessary in order to avoid artificial generation of a coherent signal at $\omega_0/2$. When sufficiently small, it will be possible to subtract this signal in the final result.

2. Technical details (see Fig. 2)

The first mirror M_0 is incorporated into an open magnetic photomultiplier (Bendix Type M 306), in which the mesh screen opposite to the cathode is removed and some other minor changes are made. The light beam hits the cathode which is placed about 5 cm behind the exit slit at a position near to the dynode strip. The beam diverges from a focal size of 100 μm (vertically) x 300 μm (horizontally) to a size 3 mm x 10 mm at the position of the detectors D_1 and D_2 . The mirrors M_0 , M_1 , and M_2 are made of glass coated with gold.

The mirror on the rotating disc is counter-balanced in order to avoid vibrations. The 25-Watt synchronous motor (Papst model HSZ 14.50-4) driving the beam splitter is encapsulated into a small housing in order to allow for free adjustments to be made inside the vacuum receptacle. The motor-housing is water-cooled by flexible tubings. The rotational motion is transmitted using a magnetic feed-through. The motor locks into several different stable phase positions. The proper position is adjusted on a try-and-error basis by watching the signals S_1 and S_2 on an oscilloscope.

The angles of incidence onto the mirrors M_1 and M_2 are adjusted in order to equalize the reflectivities. Imperfections in this adjustment together with asymmetries in the two detectors (Johnston, type MM1 equipped with external cathodes in the most recent experiments) are determined by measuring a correction curve. This correction curve is the spectrum as obtained by measuring \bar{S}_1/\bar{S}_2 without putting samples into the beams. For optimum adjustment this curve varied by 25 % in the range 20-280 eV. There is room for two pairs of samples on a rotatable sampleholder. The samples in the upper and lower beams can be interchanged. This allows for an independent measurement for determining asymmetries in the two light paths.

3. Modes of operation, examples

Mode (1) / \bar{S}_1/\bar{S}_0

In this mode the rotating mirror M_1 is fixed. Frequently an external chopper in front of the monochromator operated at 25 cps is used cutting out every second light pulse in order to avoid line-frequency disturbances. The output signals of the two lock-in amplifiers (Ithaco model 353) are electronically divided (Fig. 3a). The fact that both signals are generated by the same light beam gives ideal stability against fluctuations of the beam.

Figure 4 shows a simultaneous record of three spectra: a) is the non-normalized signal of the Al_2O_3 coated detector D_1 . Large fluctuations almost hide the exciton structure at the onset of the Al $L_{2,3}$ transitions at 77 eV and 80 eV, b) is the older type of normalization where the signal \bar{S}_1 is divided by a signal derived from an insulated sheet of metal in front of the monochromator. This monitor is hit by part of the undispersed synchrotron radiation beam. The fluctuations are reduced but still appreciable. Finally Fig. 4c) shows the spectrum of \bar{S}_1/\bar{S}_0 . Now the structure at the $L_{2,3}$ edge is undisturbed. The spectrum is smoothly distorted by the characteristics of the reference cathode. (The origin of such structures in the photoelectric yield was discussed in another paper.⁸) The fluctuations originating from the synchrotron are barely detectable in this record. Take, e.g., the fluctuations at 142 eV which is ~40 % in Fig. 4a) about 25 % Fig. 4b) but only in the order of magnitude of the statistical noise in Fig. 4c).

Mode (2): \bar{S}_1/\bar{S}_2

The signals S_1 and S_2 are detected by two lock-in amplifiers tuned to 25 cps with a phase difference of π (Figs. 1) and 3b)). There exist ways of operating the synchrotron which interfere adversely with this mode (and also with mode (3)). This occurs when the high-energy beams are switched alternately to different high-energy experiments. For most of the runs this is not a serious problem. Mode (2) is the one most frequently used with the present instrument. We were able to put, e.g. a carbon substrate into one beam and an equivalent substrate covered with an alloy sample into the other beam. The influence of the substrate cancels and the transmissivity of the alloy is obtained directly (aside from a slowly varying correction curve mentioned above). Examples of measurements using this method on Cu/Ni alloys are given in Ref. 9.

$$\text{Mode (3)} / 2 \cdot \overline{(S_1 - S_2)} / \overline{(S_1 + S_2)}$$

The electronic arrangement is shown in Fig. 3c). The main signal path is drawn in fat lines. The anodes of the detectors D_1 and D_2 are connected yielding a signal S_+ (see Fig. 1). Then the detectors D_1 and D_2 can be considered to be one single detector with the advantage that the amplification of the two parts can be regulated individually by using different power supplies. The arrangement is equivalent to the one described in Ref. 5. $\overline{S_1 - S_2}$ is derived from the 25 cps component of S_+ (lock-in L_1) and $\overline{S_1 + S_2}$ is derived from the 50 cps component of S_+ (lock-in L_2).

Since we are looking for small signals $\overline{S_1 - S_2}$ in the order of $10^{-2} - 10^{-3}$ of $\overline{S_1 + S_2}$, we will be bothered by the 25-cps noise component in synchrotron emission. This random signal needs to be subtracted from the genuine 25 cps signals. In order to achieve this purpose, we derive a signal of equal amplitude from detector D_0 containing only the random 25 cps noise, and we then feed it into the differential input of lock-in L_1 . This compensation signal is kept at the appropriate level by a servo loop which regulates the power supply of D_0 . The criterion is, to make the difference signal $S_+ - S_0$ measured at 50 cps by lock-in L_3 zero. The necessity of using this compensation depends very much on the conditions of operation of the synchrotron. In some runs fluctuations could be reduced by a factor of as much as 1/5 by switching on this circuit, while at other times it was barely helpful.

The main limiting factor of method (3) is photon statistics. With 10^6 detected photons/sec and a time constant of 1 sec noise on signals S_1 or S_2 is in the order of 10^{-3} . Due to the absorption in the films, the number of detected photons is usually even less. In some instances we had to use averaging time constants of up to 10 sec. The lowest noise level obtained for $\Delta T / \bar{T}$ was in the order of

10^{-3} . The method ought to work with a much better accuracy and without the need for a special compensation technique when using the continuous radiation from an electron storage ring.

Figure 5 shows the uncovering of the Au $N_{6,7}$ edges in absorption as an example of measurements using this method.¹⁰ Since the onset of these transitions is weak due to the shift of oscillator strength away from the edge, and since it is superposed onto the slope of decreasing d-electron absorption it was not resolved in the early experiments.¹¹ By now we are able to identify these edges in measurements performed with all three modes described here. The result shown is obtained with a 155 Å thick Au film placing a carbon film of appropriate thickness (~ 1000 Å) in the reference channel. The absorption coefficient of carbon decreases with increasing photon energy similarly to Au but exhibits no structure. The two free parameters: film thickness and gain of detector D_2 serve to approximately match the signal amplitude and the average slope of the Au spectrum in this range. The $\Delta T/\bar{T}$ spectrum shows the $N_{6,7}$ edges superimposed on a slightly rising background. Our best values¹⁰ for the position of the N_6 edge is 87.6 ± 0.2 eV and for the N_7 edge is 84.0 ± 0.2 eV.

Although several features of the instrumentation described here are consequences of using a pulsating (synchrotron-) radiation source this two-beam technique is applicable with other sources in the grazing-incidence vacuum ultraviolet region as well. The advantages of this technique will be demonstrated in further publications on the absorption spectra of alloys and metals.

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+) Now at Hahn-Meitner-Institut für Kernforschung, Berlin-Wannsee

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Figure Captions

- Fig. 1: Time-structure signals. I_0 = electron current in the accelerator, S_0 = signal from detector D_0 (Fig. 2), S_1 and S_2 = signals from detectors D_1 and D_2 when the rotating mirror M_1 is operated at 25 cps, S_+ superposition of signals S_1 and S_2 .
- Fig. 2: Grazing-incidence beam splitter. Monochromatized radiation is reflected by a mirror M_0 , which simultaneously serves as the cathode of an open photomultiplier D_0 . Radiation either falls onto the rotating mirror M_1 (B = balancing weights) and continues on a path designated "beam 1", or it passes the open segment of the rotating mirror holder, is then reflected by the static mirror M_2 , and continues as "beam 2". The two beams pass two interchangeable filters F_1 , F_2 and hit two detectors D_1 and D_2 .
- Fig. 3: Electronics (see text): a) mode (1) is primarily used to obtain well monitored spectra with and without filters, b) mode (2) is used to obtain the ratio of the transmissivities of two filters, c) mode (3) is used to obtain the normalized differential transmissivities of two filters. The thin-line signal path and the double-line feed-back path are used in order to compensate fluctuations of the source at the detection frequency of L_1 (25 cps). L_1 , L_2 , L_3 = lock-in amplifiers, R = ratio amplifier.
- Fig. 4: Original spectra registered simultaneously with and without reference monitors: a) Signal as detected by detector D_1 (Fig. 2) equipped with an Al_2O_3 -coated cathode. The details of the spectrum are covered by the fluctuations of the source. b) The same spectrum

divided by a reference signal obtained from a cathode situated next to the entrance aperture of the monochromator. This monitor uses undispersed "white" radiation from a different portion of the incoming beam. c) The signal of detector D_1 divided by the signal of the internal reference detector D_0 (Fig. 2). The spectrum is smoothly distorted with respect to spectra a) and b) because of the wavelength dependent signal D_0 . (The exciton peaks due to the $L_{2,3}$ transitions of Al in Al_2O_3 are recognized around 80 eV.)

Fig. 5: Observation of the Au N_6 and N_7 absorption edges using the technique shown in Fig. 3c) with a 155 Å thick Au film in one beam and a ~1000 Å thick C film in the reference beam. Curve 1: 3 sec time constant, curve 2: 1 sec time constant.

Fig. 1

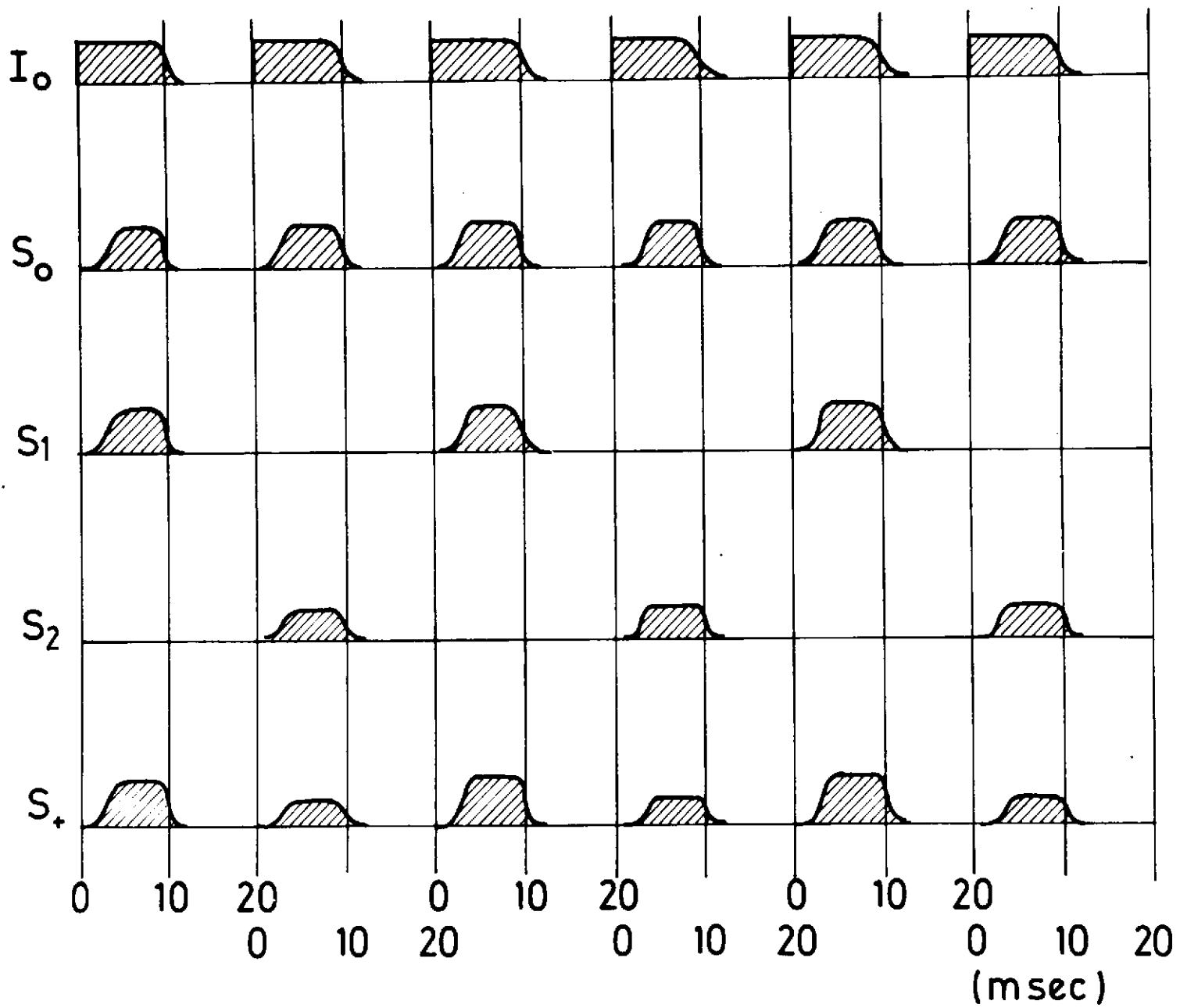
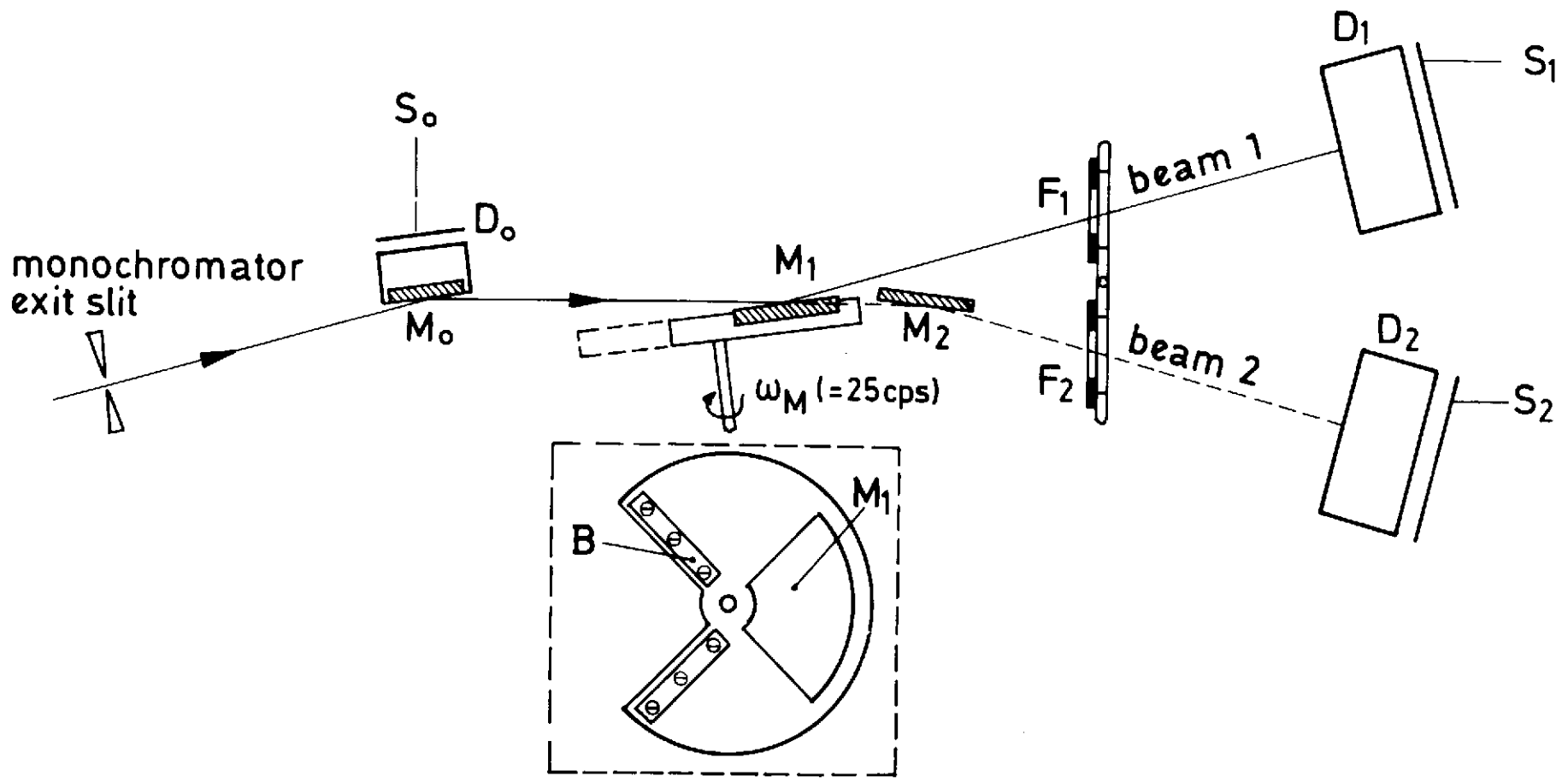


Fig. 2



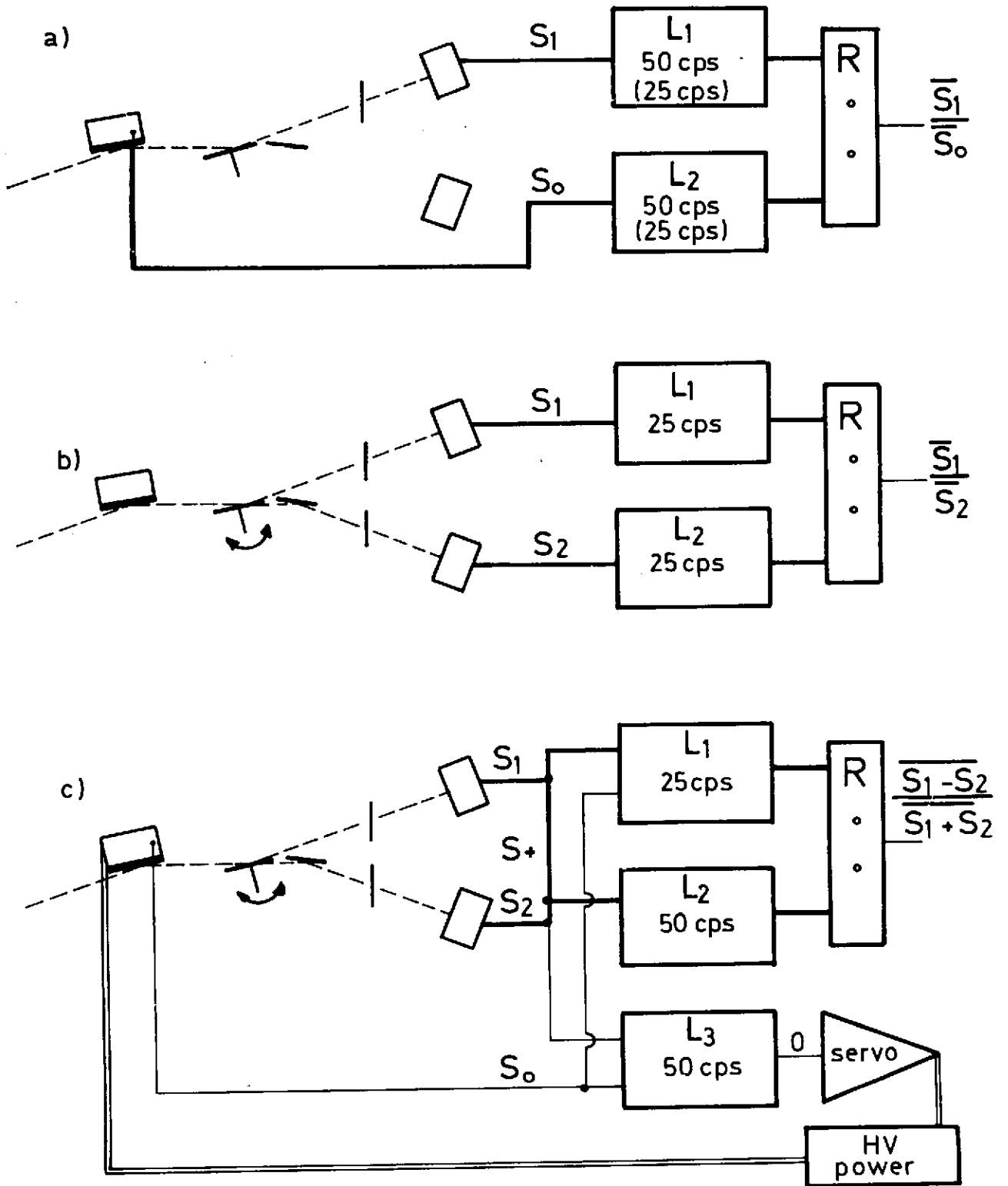


Fig. 3

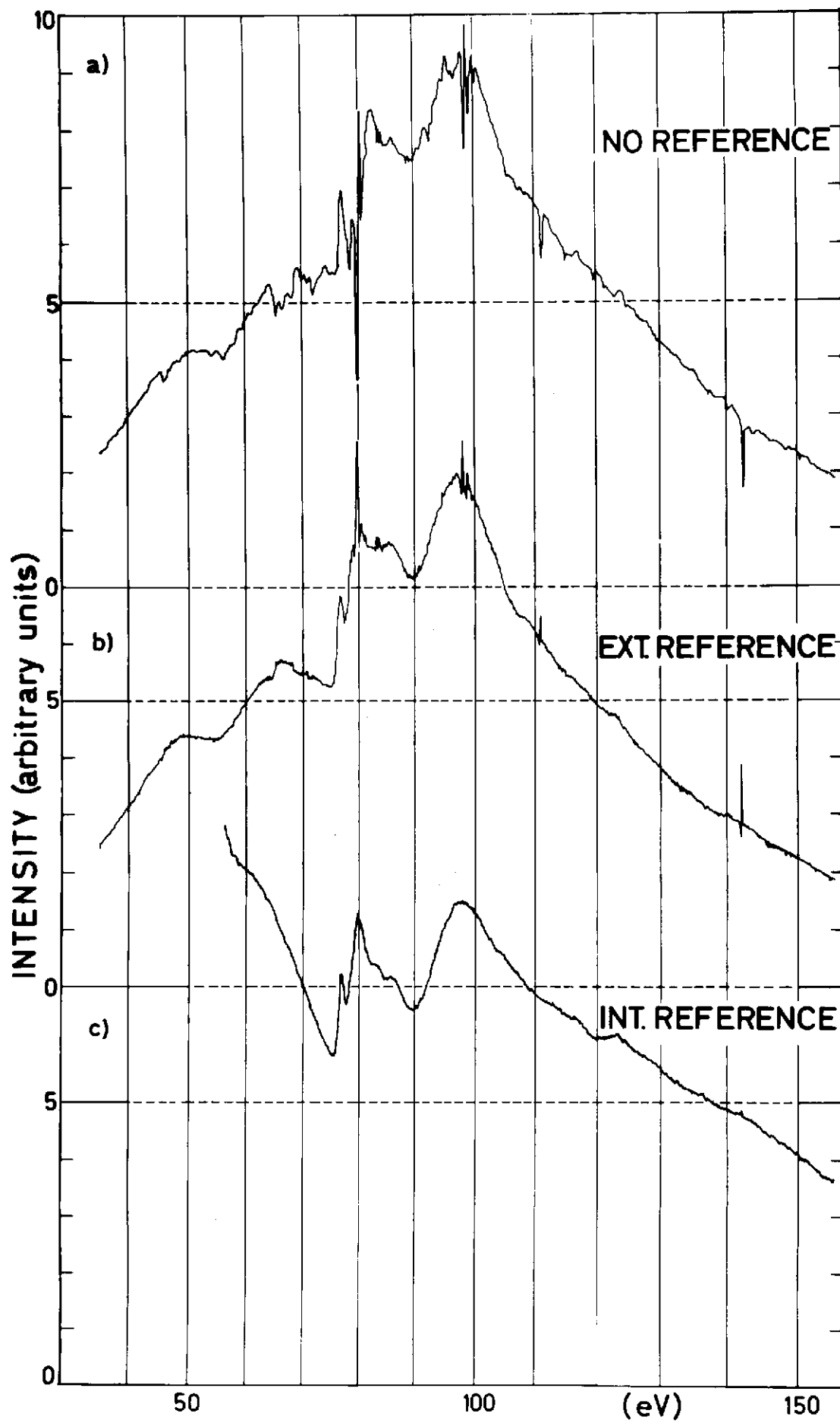


Fig. 4

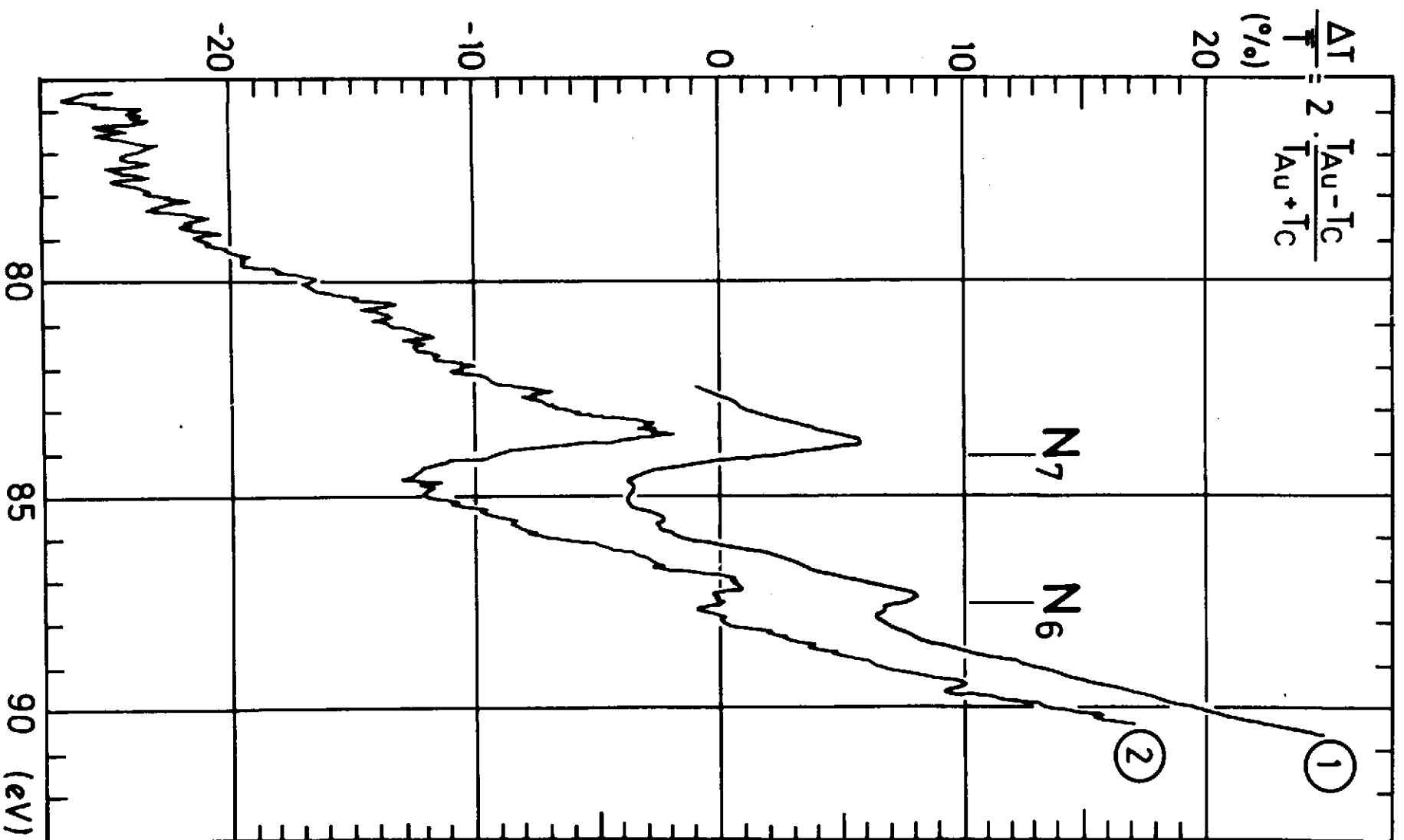


Fig. 5

