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Two-beam photoelectric vacuum reflectometer for the vacuum ultraviolet range with automatic data recording*

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This paper gives a description of the design and operating principles of a two-beam photoelectric reflectometer for the vacuum ultraviolet range incorporating automatic data recording. This set-up, adapted for the measurements in the range from liquid nitrogen to room temperatures, features high sensitivity and high measuring accuracy better than 0.1%.

Investigations of the electronic structure of solids by optical methods, such as absorption or reflectivity measurements, have been carried out [n numerous projects both in metals [1, 2] and in semiconducting materials t3–7]. Reflectivity or absorption measurements remain an up-to-date technique, as they still are one of the few sources of information about the electronic properties of substances. Much information about this structure is obtained from the measurements in the 1–6 eV range of incident light energies, i.e. from the infrared to near ultraviolet. The investigations in this energy range are not hampered by any particular technical problems. On the other hand, examination of materials featuring, say, a large energy gap, and acquisition of data on the structure of the higher energy bands require the knowledge about the optical properties of these materials for incident light energies exceeding 6 eV (i.e. in the range of the vacuum ultraviolet) and the use of sophisticated measuring techniques. Owing to strong absorption in air, the measurements have to be made with special vacuum measuring arrangements, special light sources and appropriate radiation detectors and grating monochromators fit for work in the vacuum ultraviolet range [8, 9].

This paper gives a description of the design and operation principles of a two-beam photoelectric reflectometer for the vacuum ultraviolet range incorporating automatic data recording, which was built in the laboratories of the Institute of Physics of the Jagiellonian University. This set-up, adapted for measurements in the range from liquid nitrogen to room temperatures, features high sensitivity and high measuring accuracy better than 0.1%.

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The arrangement for measuring reflectivity and transmissivity consists of a two-beam photoelectric vacuum reflectometer, a Hilger and Watts E766 vacuum monochromator with a meter-long concave diffraction grating having 600 lines per mm and linear dispersion 4 Å/mm mounted on a Rowland circle, and a Hinteregger lamp [10, 11] manufactured by McPherson. The block diagram of the complete measuring set-up is shown in fig. 1. The path of the light rays in the two-beam reflectometer, designed according to the scheme published in [12], is illustrated in fig. 2.

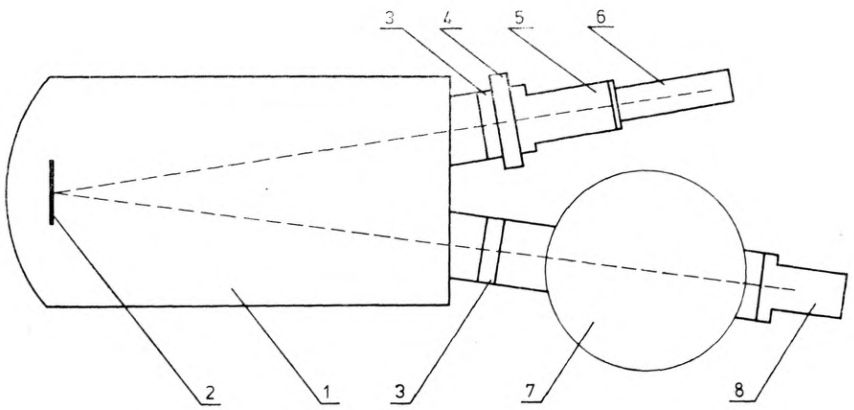

Fig. 1. Block scheme of the two-beam spectrometer:
1 – vacuum monochromator, *2* – concave one meter grating, *3* – LiF window, *4* – chopper, *5* – light source, *6* – He–Ne laser, *7* – reflectometer's chamber, *8* – photomultiplier

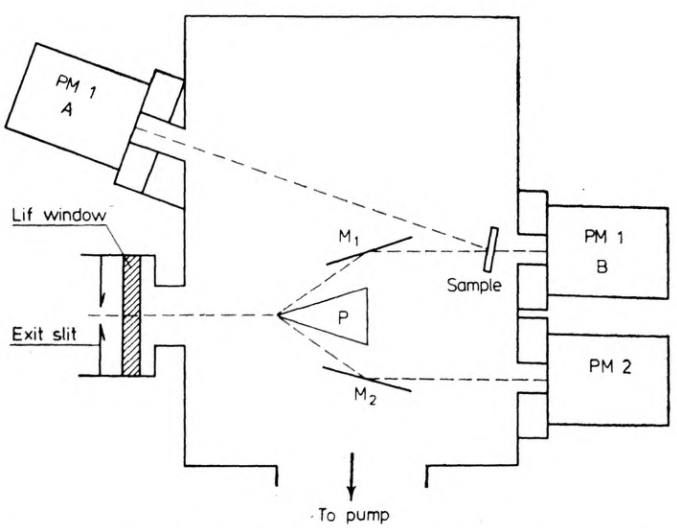

Fig. 2. Block optical scheme of the two-beam reflectometer:
P – splitting prism, M_1 and M_2 – mirrors

The light beam from the monochromator is divided in the reflectometer by the 30° metal-coated prism P into two rays which fall on mirrors M_1 and M_2. The upper ray hits the sample and after being reflected off it falls onto the photomultiplier PM1 at the position A or — when the reflectometer's instrumental function or transmissivity is being measured — onto the photomultiplier $PM1$ at the point B. The lower beam, acting as a reference beam, leaves the mirror M_2 directly to the photomultiplier $PM2$. In the photoelectric reflectometer arrangement the output voltage of the photomultiplier in the channel in which sample is mounted can be expressed in general terms as

$$U(E) = a_i \cdot I_0(E) \cdot Y(E) \cdot R_m(E) \cdot (E), \tag{1}$$

where: $I_0(E)$ is the intensity of the light beam entering the reflectometer, a_i is a geometrical factor, $Y(E)$ is the quantum yield of the photomultiplier, $R_m(E)$ is the overall reflectivity of the mirrors in the optical system, and $R(E)$ is the reflectivity of the sample.

In order to perform proper reflectivity or transmissivity measurements it is necessary to know the so-called instrumental function $Z_0(E)$ defined as the quotient of the voltage from the photomultiplier $PM1$ at the po, sition B, U_{1B}, and the photomultiplier $PM2$, U_2. Its form is

$$Z_0(E) = \frac{U_{1B}}{U_2} \frac{a_1 \cdot I_0(E, t) \cdot Y_1(E) \cdot R_{m1}(E)}{a_2 \cdot I_0(E, t) \cdot Y_2(E) \cdot R_{m2}(E)}, \tag{2}$$

where: $I_0(E, t)$ is the intensity of the light beam entering the reflectometer depending on time due to short-time fluctuations of intensity of light source and a_1, a_2, $Y_1(E)$, $Y_2(E)$, $R_{m1}(E)$, and $R_{m2}(E)$ are the respective geometrical factors, quantum yields of photomultipliers $PM1$ and $PM2$, and overall reflectivities of the mirrors in the optical system for the beams 1 and 2.

When the reflectivity $R(E)$ of the sample is being measured, the photomultiplier $PM1$ is placed in the position A, then the quotient of the output voltages of photomultipliers $PM1$ and $PM2$ takes the form

$$\begin{aligned} Z_R(E) = \frac{U_{1A}}{U_2} &= \frac{a_3 \cdot I_0(E, t') \cdot Y_1(E) \cdot R_{m1}(E) \cdot R(E)}{a_2 \cdot I_0(E, t') \cdot Y_2(E) \cdot R_{m2}(E)} \\ &= \frac{a_3 Y_1(E) \cdot R_{m1}(E) \cdot R(E)}{a_2 Y_2(E) \cdot R_{m2}(E)}, \end{aligned} \tag{3}$$

where: a_3 is the geometrical factor of the arrangement in the measuring position A, and index t' in term $I_0(E, t')$ means that the function $Z_R(E)$ is determined in another moment than the function $Z_0(E)$. After dividing by sides expressions (3) and (2) we get

$$\frac{Z_R(E)}{Z_0(E)} = \frac{a_3}{a_1} R(E), \tag{4}$$

whence the reflectivity is

$$R(E) = \alpha' \frac{Z_R(E)}{Z_0(E)}. \tag{5}$$

Here, $Z_R(E)$ and $Z_0(E)$, determined experimentally in successive measurements, are functions of the energy of the incident light independent of fluctuations of the incident beam intensity, $\alpha' = a_1/a_3$ is a constant geometrical factor independent of the incident light energy which may be determined from the sizes of the light spots on the photomultipliers. This task is not simple, however, especially when the beams are deformed by the optical system. For the new reflectometer the geometrical factor α' was found by comparing in the 5–6 eV range the reflectivity spectra acquired by means of the photoelectric vacuum reflectometer with results obtained with a two-beam photoelectric reflectometer adapted for absolute measurements in the visible and ultraviolet range [13].

When the transmissivity $T(E)$ of materials is being determined, i.e. when the light intensity is measured by the photomultiplier $PM1$ at position B, the quotient of voltages at the photomultipliers $PM1$ and $PM2$ is expressed as

$$Z_T(E) = \frac{a_1 \cdot I_0(E, t') \cdot Y_1(E) \cdot R_{m1}(E) \cdot T(E)}{a_2 \cdot I_0(E, t') \cdot Y_2(E) \cdot R_{m2}(E)}, \tag{6}$$

whence after division by sides of eq. (6) by eq. (2) we have

$$T(E) = \frac{Z_T(E)}{Z_0(E)}. \tag{7}$$

It follows from the formulae (5) and (7) that in order to determine reflectivity $R(E)$ or transmissivity $T(E)$ it is necessary to find the values of $Z_R(E)$ or $Z_T(E)$ and $Z_0(E)$, which may be determined straightforwardly from the output voltages of photomultipliers $PM1$ and $PM2$.

Systems for direct recording of reflectivity applied in many studies [4–7] have a common feature, namely, the division of the signal from the sample by the reference signal is accomplished in the analog mode. With this kind of signal processing the relative measuring error of R or T does not exceed one or two per cent. In the reflectometer now being described the photomultiplier output voltages are recorded digitally following the scheme described in [14]. Therefore, they are divided and processed further on a Mera 305 computer. The digital procedure now applied improves the measuring accuracy ten times at least ten times. Figure 3 presents the block diagram of the detecting system and automatic data recording scheme for the two-beam photoelectric reflectometer. The system possesses two independent, but identical detection paths consisting of a photomultiplier, a preamplifier, a homodyne nanovoltmeter and a digital voltmeter.

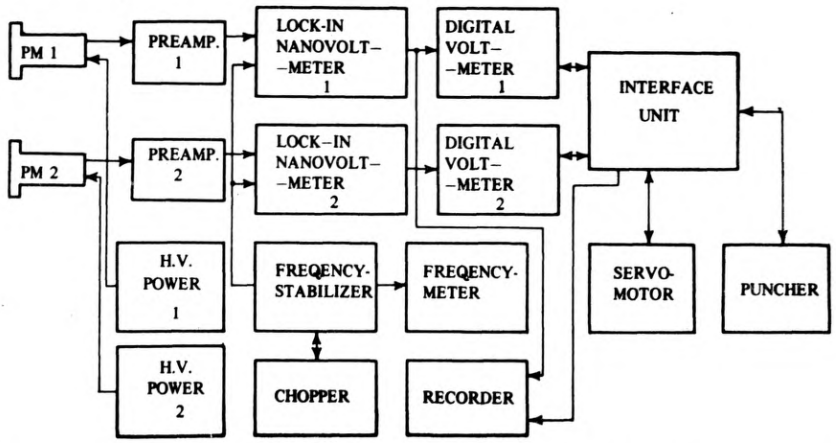

Fig. 3. Electronic block of the two-beam spectrophotometer with automatic data recording system

It also incorporates electronic circuitry common for both the paths which enables the signals to be read simultaneously from both the paths and voltages from the digital voltmeters to be punched in a pre-defined sequence on paper tape. Moreover, this system counts the measurement points and controls the operation of the motor driving the monochromator grating.

Because of the phase-sensitive detection applied, the light ray was modulated by a chopper placed in front of the entrance slit of the monochromator. Its frequency is variable within a range of $f = 80$ to 120 Hz. The chopper is driven by a d.c.-motor with precision electronic control and rps stabilization (with an accuracy of ± 0.5 Hz).

The choice of the proper working frequency and rps stabilization of the chopper is particularly important when light sources based on spark discharges are used, for modulation frequencies near the discharge frequency should be avoided because of the elevated sensitivity of the homodyne nanovoltmeter to electromagnetic disturbances at frequencies falling within the detection band. Secondly, it is necessary to select a modulation frequency ensuring the maximum level of the output signal which depends on the number of spark discharge cycles occurring within a cycle of the reference signal of the homodyne nanovoltmeter.

In order to have the smallest possible losses in light intensity in the vacuum ultraviolet range, the mirrors M_1 and M_2 and the splitting prism P (fig. 2) are coated by evaporation in vacuum with a layer of aluminium and then a layer of MgF_2 of a thickness of about 250Å [15].

The autonomous pumping system of the E766 monochromator ensured a working vacuum of $10.64 \cdot 10^{-4}$ Pa (8×10^{-6} Torr). The working pressure in the reflectometer of $1.33 \cdot 10^{-4}$ Pa (1×10^{-6} Torr) was achieved within

a short period of time (about 15 min.) by means of a standard pumping system connected directly to the reflectometer chamber. The entrance and exit slits of the monochromator were separated from the other parts of the system by LiF windows. This allows measurements to be made in the reflectometer to an energy of about 11 eV. The measuring range can be extended to higher energies without making any structural alterations in the arrangement, but simply by exchanging some of the optical elements (the grating, the coating of the mirrors and prism, the detectors) and removing the LiF windows. A small He–Ne laser with internal mirrors was installed permanently in the measuring arrangement to facilitate the alignment of the system and visual positioning of the sample, which is rendered difficult by the relatively low intensity of light emitted by the source in the visible range. The laser beam, passing radially through the lamp capillary, precisely determines the optical axis of the entire measuring set-up. This design allows the system to be quickly, easily and accurately aligned for measurements. In the aforementioned Hintereggerr discharge lamp the active gases are usually hydrogen and noble gases, depending on the range in which measurements are to be made. In the newly constructed reflectometer the testing was accomplished with the use of hydrogen and xenon, which produce a continuous spectrum of sufficient intensity to about 9 eV. Optimum conditions of excitation were achieved at hydrogen pressure of 12 mm Hg gas flow rate of 10 cc/min. and discharge current of 40 mA, the spark discharge frequency

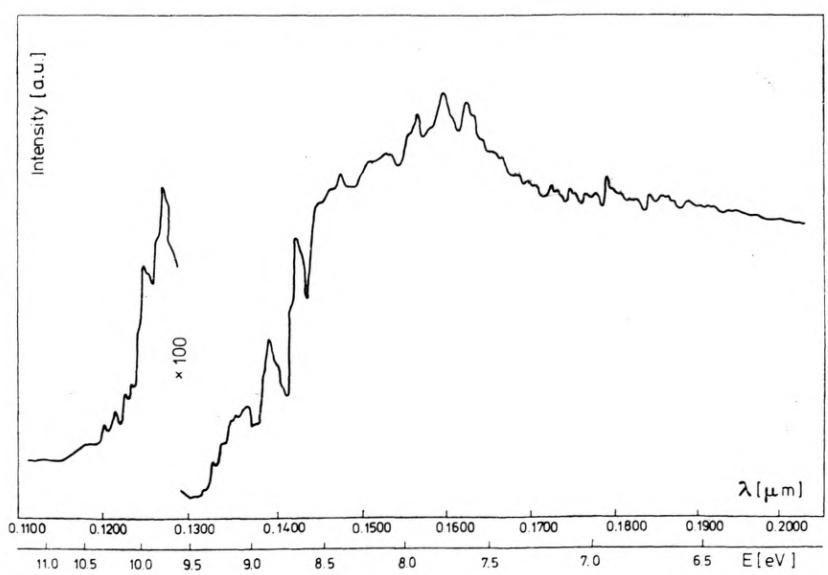

Fig. 4. Hydrogen spectrum registrated directly on Hilger–Watts monochromator slit by natrium silicilate coated EMI 9789 Q.B. photomultiplier for chosen discharge conditions $C = 0.0032\ \mu F$, $L = 0$

being defined by the capacitance $C = 0.0032$ μF and inductance $L = 0$, and dynamic resistance of the lamp (no ballast resistors were used). With the use of other gases it is necessary to find experimentally the optimum conditions for exciting the continuous spectrum. Figure 4 shows the continuous spectrum of the lamp filled with hydrogen operating under the described conditions. Test measurements of the system were performed in the energy range of 3.0–8.55 eV (4100–1450 Å) for a quartz plate (Spectrosil, diameter 18 mm, thickness 2 mm), and the transmissivity and reflectivity spectra as well (fig. 5). Analysis shows that the

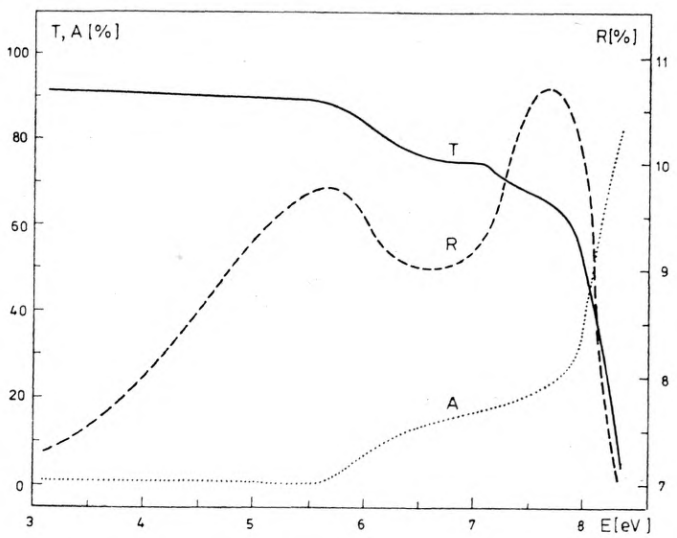

Fig. 5. Reflectivity and transmission spectra of the quartz plate (Spectrosil) in 3.0–8.55 eV energy range

sum of reflectivity and transmissivity in the region of total plate transparency is equal to unity with an accuracy of one per cent. If the contribution of coherent and incoherent light scattering in measured reflectivity and/or transmissivity coefficients resulting from roughness of the surface and internal stresses of the sample is neglected, the estimated accuracy of the absolute reflectivity, owing to objective difficulties in determining the geometrical factors, is not better than one or two per cent; but relative accuracy of the reflectivity is better than 0.1 per cent in the entire measuring range. The influence of factors mentioned above i snowa separate problem just now being analysed.

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Двупучковый вакуумный фотоэлектрический рефлектомер для ультрафиолета с автоматической регистрацией данных

В настоящей работе описано устройство и действие двупучкового фотоэлектрического рефлектомера с автоматической регистрацией данных на область вукуумной ультрафиолетовой части спектра. Измерительная схема приспособлена к измерению коэффициента отражения R и передачи T в пределах энергии 5,5–8,55 эВ, при температурах от жидкого азота до комнатной. Применение цифровой регистрации и обработки результатов измерений даёт возможность достижения значительной относительной точности измерений, лучшей, чем 0,1%.