Semitransparent monochromators for X-ray imaging based on highly oriented pyrolytic graphite (HOPG)

Alexander G. Touryanski[*a], Igor V. Pirshin[a], Alexander V. Vinogradov[a], Nelson Publicover[b], Victor Kantsyrev[b], Inna G. Grigorieva[c], Alexander A. Antonov[c]

[a]P.N. Lebedev Physical Institute, Moscow, Russia
[b]Biomedical Engineering Program, University of Nevada, Reno
[c]Optigraph Ltd, Moscow, Russia

ABSTRACT

Semitransparent HOPG monochromators are investigated with emphasis on their possible use in X-ray imaging. HOPG plates and films 5 - 100 μm thick up to 14 cm^2 in area were prepared for testing. Scanning technique has been used to measure distribution of local reflectivity, transparency, and thickness. A record parameter of mosaic spread $\Delta\omega$ down to 3.6' is received. HOPG film areas with average $\Delta\omega$=5' and 6' were equal respectively to 4.5 and 10 cm^2. The echelon-monochromator has been tested. This arrangement gives sharp increase in reflectivity and the possibility of primary beam sweeping. Obtained results are very promising for future applications in such important fields as medical diagnostics and surgical treatment monitored by X-ray digital camera.

Keywords: Semitransparent monochromator, HOPG, X-Ray imaging

1. INTRODUCTION

Decreasing dose rates in biomedical investigations is one of the basic tasks in designing of penetrating radiation systems[1]. Receiving high contrast images with the use of near *K*-jump absorption[2] is another important purpose. It is especially desirable for angiography allowing to obtain crisp and clear images of vessels without shades created by other biological tissues. Both problems are directly bound up with development of effective systems capable to convert polychromatic radiation into quasimonochromatic with a bandwidth < 5 keV.

Highly oriented pyrolytic graphite (HOPG) is widely used for X-ray beam focusing and monochromatization in the broad energy range E >1 keV. This is determined by the high integral reflectivity R of pyrographite[3-6], the possibility to produce monochromators of specified profiles[3,7], and a simpler adjustment procedure due to a lower required angular adjustment accuracy than for *Si, Ge*, and quartz crystal monochromators. However, the angle of mosaic spread $\Delta\omega$ for thick (~1 mm) monolithic HOPG plates is typically in a range 20′-50′ [3,6]. In projection imaging techniques it leads to undesirable smearing effects on edges and poor space resolution of small details. In biological and especially medical diagnostics it is very desirable to obtain comparatively large view zones with area >100 cm^2. But synchrotrons and other powerful X-ray sources possess a point-like focus and produce polychromatic beams with small angle divergence. As a result, the problem of the beam sweeping in the view zone of interest arises.

Semitransparent monochromators (SM) prepared from thin HOPG plates are of special interest for the effective solution of the above mentioned tasks. X-ray measurement schemes based on SM provide simultaneous using a number of spectral lines. This feature proved to be especially advantageous in X-ray reflectometry and other small angle methods[8-10]. In this study we first present results on HOPG SM investigations with emphasis on their possible use in X-ray imaging for biomedical applications.

2. SAMPLES AND METHODS

HOPG plates and films 5 - 100 mm thick up to 14 cm^2 in area were prepared for testing. All samples have been manufactured by Optigraph (Moscow). Measurements have been carried out on the standard X-ray diffractometer with a

* contact: tour@mail1.lebedev.ru; phone +7 095 132-6268; P.N. Lebedev Physical Institute, Leninskii pr.53, Moscow, Russia, 119991

line. Angle divergence in the measuring plane was about 20″. The size of irradiated area at the Bragg angle for HOPG (002) reflection was 6.5x0.3 mm^2. To appreciate local parameters of HOPG in front of the sample a 50 μm diaphragm was placed. Linear scanning was made in the vertical plane perpendicular to the incident beam (y coordinate). Π-like sample holders have been prepared for measurements of transmission T and reflection R coefficients and HOPG thickness t determination. The sample holder could be shifted in a horizontal plane in parallel with the sample surface (x coordinate).

3. EXPERIMENTAL RESULTS

The mosaic spread variation with linear coordinate x along the 5 μm HOPG film is presented in Fig. 1. Values of $\Delta\omega$ are taken from ω-scans at fixed angle positions of the detector (Fig. 2). Data received for local illuminated areas on HOPG samples S=2 mm^2. Angle shifts of maximums on $I^R(\omega)$ curves can be attributed in part to experimental error due repeated steps of linear movements and fixing sample in a holder.

It is obvious, however, that, if the sample supporting area is much smaller than the sample itself, the process of its transfer and fixing in a holder may lead to increasing the effective value of $\Delta\omega$ because of irregular strains along the sample perimeter and accidental mechanical disturbances of HOPG surface. It is confirmed by results of linear scanning with a 50 μm diaphragm of the 46 μm HOPG plate (Fig. 3). In this case the detector and sample are positioned at the fixed angles θ_B and $2\theta_B$, where θ_B is the Bragg angle for (002) reflection. The observed shifts of diffraction maximums are attributed only local angle deviations of the HOPG reflecting surface (Fig.4). This considerably influences the reflective HOPG properties. The averaged value of R is equal to 0.21. However, a local value of R measured with a diaphragm reaches 0.33 at the point A. Note, that if the scanned sample is tuned at any chosen point A-D (see Fig. 3,4), then the peak values of R are in the rather narrow range from 0.27 to 0.33, as compared to 0.19 and 0.33 in Fig. 3.

In Fig. 5 ω-scans for a 68 μm plate are presented for two detector positions: $2\theta=0$ (transmission mode) and $2\theta=2\theta_B$ (reflection mode). According to predictions of kinematic theory of X-ray diffraction[11] for rather thin mosaic plates the relation between coefficients transmission $T(\omega)$ and reflection $R(\omega)$ can be written in a form

$$[R(\omega) + T(\omega)] \exp[\mu t/\sin(\omega)] = 1, \qquad (1)$$

where μ – linear coefficient of absorption in the plate, t - plate thickness. Summing experimental functions $T(\omega)$ and $R(\omega)$ gives exactly the calculated angle dependence of absorption $T^A(\omega)$ (see Fig. 5). It means that for increasing the monochromator reflectivity it is necessary to decrease the path of an X-ray beam in reflecting material. This can be achieved by means of the echelon SM arrangement proposed[6].

4. ECHELON-MONOCHROMATOP

It is well known that asymmetrically cut and properly oriented monocrystals provide broadening of the primary beam. For imaging applications it can be considered as sweeping of the beam. HOPG is very sensitive to mechanical procession that badly deteriorates reflective properties of high quality material. Alternative methods of obtaining asymmetric HOPG cuts are not sufficiently developed at present. Nonetheless, symmetric HOPG offers an effective solution of the sweeping problem, if a number of thin HOPG plates are placed in a row along the incident beam.

Fig. 6 shows an experimental setup for testing the HOPG echelon-monochromator. Radiation from X-ray tube 1 with a copper anode was monochromated by asymmetrically cut Si (111) single crystal 2 rotatable about the axis O_1. The characteristic line $CuK_{\alpha 1}$ was selected from the diffracted beam by a vertical slit 3. The beam was additionally bounded and centered in the vertical plane by a 3-mm-wide horizontal slit 4. The first 5 and third 10 HOPG plates (along the X-ray beam propagation direction) were mounted on rotating shafts 5 and 7 attached to heads for orienting single crystals. The heads provided translations of samples along the beam, perpendicular to it, and their rotations in the plane perpendicular to the beam. Plates 8 and 10 were independently adjusted for the angle of diffraction by rotating the shafts about axes O_2 and O_4 aligned with the X-ray beam plane during adjustment. The second plate 9 (along the beam propagation direction) was set in a standard holder 6 for flat samples aligned with the principal axis O_3 of a horizontal type goniometer. The distance between axis O_3 and the slit 11 was 190 mm, and the distances O_2-O_3 and O_3-O_4 were 35 mm. As echelon elements were chosen HOPG plates 46-76 mm thick with peak reflectivity in a range 0.22-0,29. All three SMs were tuned to the Bragg angle.

Angle dependence of the transmitted and reflected by echelon intensity is presented in Fig. 7. As expected, the sum of peak R received after calibration is equal to 0.47. This value is much higher than corresponding parameters of the individual HOPG SM. For imaging applications it clearly demonstrates the possibility of the primary beam sweeping. Forexample, continuous distribution of X-ray intensity can be achieved if proper values of R and spacing between individual echelon elements are chosen.

5. CONCLUSIONS

Large area semitransparent HOPG films with a record parameter of mosaic spread $\Delta\omega_{min}$ down to 3.6' are received. Film areas with average $\Delta\omega$ 5' and 6' were equal to 4.5 and 10 cm^2. Differential reflectivity $R'=dR/dt$ up to 9.5 10^{-3} mm is achieved. Note, that if the thickness d of investigated object is 3 cm (mammography case) then in the registration plane located immediately behind the object for a given $\Delta\omega_{min}$ the image of a point detail will have the size $s\approx 8$ μm. This value is considerably less than the space resolution l of 2D X-ray detectors currently used in medical applications. In the case of angiography the value of d should be an order of magnitude higher. If the field of view is about 20x20 cm^2 and an imaging matrix consists of 2000x2000 pixels, then, again respective value of $s \leq l$. It means that obtained results are very promising in future applications in such important fields as medical diagnostics and surgical treatment monitored by X-ray camera. The main tasks are to increase the peak reflectivity and homogeneity of HOPG preserving the received value of $\Delta\omega_{min}$. It has been demonstrated that the effective X-ray beam sweeping could be provided by the echelon type arrangement of thin HOPG plates.

ACKNOWLEDGMENTS

The work was supported by CRDF grant # RP2 - 2105.

REFERENCES

1. E.G. Chikirdin, "Development of digital equipment for X-ray diagnostics" (in Russian), Medical Equipment (Russia), **3,** pp. 36-39, 1998.
2. CERN Currier. p. 8, April 2001.
3. I.G. Grigorieva and A.A. Antonov, "HOPG as powerful X-ray optics", *Proceedings of the Europian Conference on Energy Dispersive X-Ray Spectrometry 1998*, EDXRS-98, Bologna, Italy, 7-12 June, 1998, Edited by J.E.Fernandez and A.Tartari; pp.121-124, 1999.
4. M. Sanchez del Rio, M. Gambaccini, G. Pareshi, A.Taibi, A. Tuffanelli and A. Freund, "Focusing properties of mosaic crystals", *Proc. SPIE*, **3447**, pp. 246-255, 1998.
5. M. Ohler, M. Sanchez del Rio, A Tuffanelli, M. Gambaccini, A.Taibi, A. Fantini and G. Pareshi, "X-ray topographic investigation of the granular in a graphite mosaic crystal: a three-dimentional reconstruction", *J. Appl. Cryst*, **33**, pp. 1023-1030, 2000.
6. A.G. Tour'yanskii and I.V. Pirshin, "Pyrolitic Graphite X-Ray Echelon- Monochromator", *Instrum. Experim. Techn.* **41**, pp. 118-122, 1998.
7. B.Beckhoff, B.Kanngieber, J.Scheer et al, "Toroidaly Shaped HOPGcrystals as strongly focusing Bragg reflectors of characteristic X-ray tube radiation for EDXRF analysis", *Advances in X-Ray Analysis*, **37**, pp. 523-533, 1994.
8. A.G. Tour'yanskii, A.V. Vinogradov and I.V. Pirshin, "A Two-Wave X-Ray Reflectometer", *Instrum. Experim. Techn.* **42**, pp. 94-99, 1999.
9. R.M. Feshchenko, I.V. Pirshin, A.G. Touryanski and A.V. Vinogradov, "New Methods of X-Ray Reflectometry of Solids and Thin Solid Films", *Journ. Russian Laser Res.* **20**, pp. 136-151, 1999.
10. A.G.Touryanski, A.V.Vinogradov and I.V.Pirshin, "Two-channel X-ray reflectometer", Nucl. Instr. Meth. Phys. Res. **A448**, pp.184-187, 2000.
11. A.A. Rusakov, *Roentgenography of metals*, pp. 144-146, Atomizdat, Moscow, 1977.

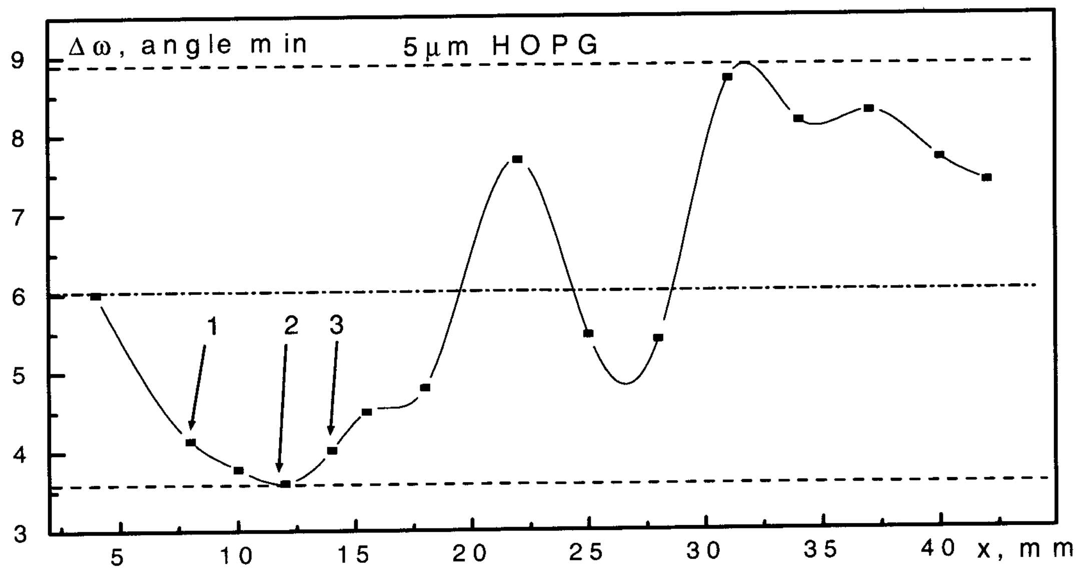

Fig. 1. Mosaic spread $\Delta\omega$ variation with linear coordinate x along the HOPG film. Local illuminated area S=2 mm^2.

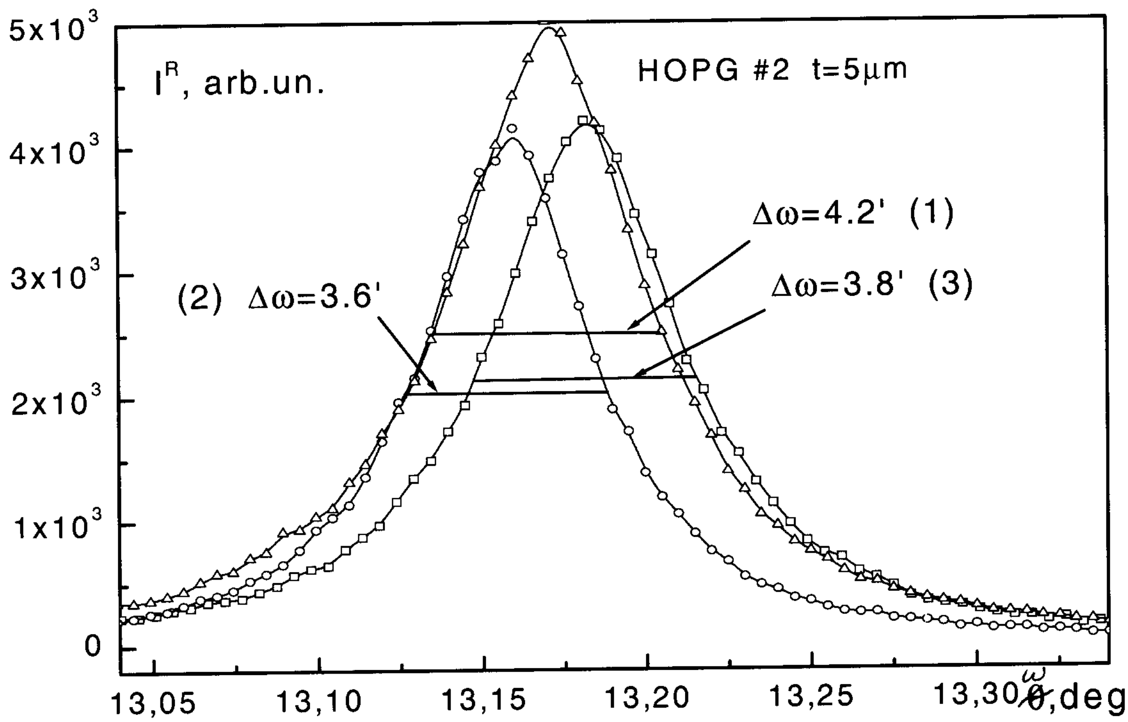

Fig. 2. Angle dependence of reflected intensity for 5 μm HOPG film at fixed detector position (ω-scans) corresponding to dots (1-3) in Fig. 1.

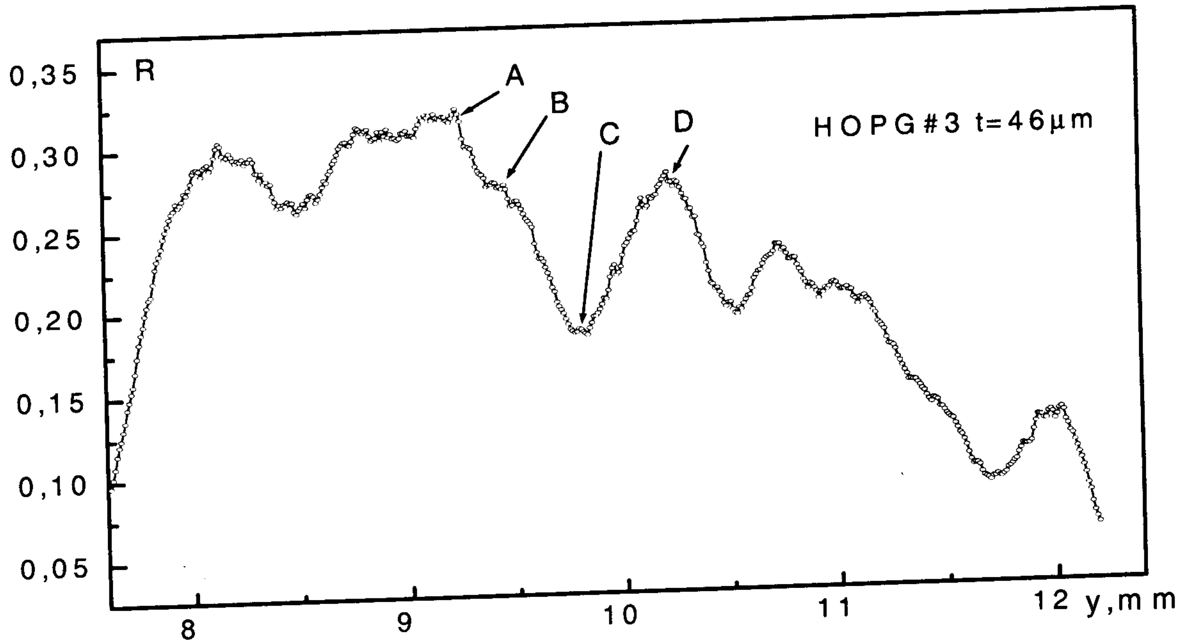

Fig. 3. Reflectivity vs y coordinate (perpendicular to the primary beam measured by scanning 50 µm diaphragm.) for the 46 µm HOPG plate.

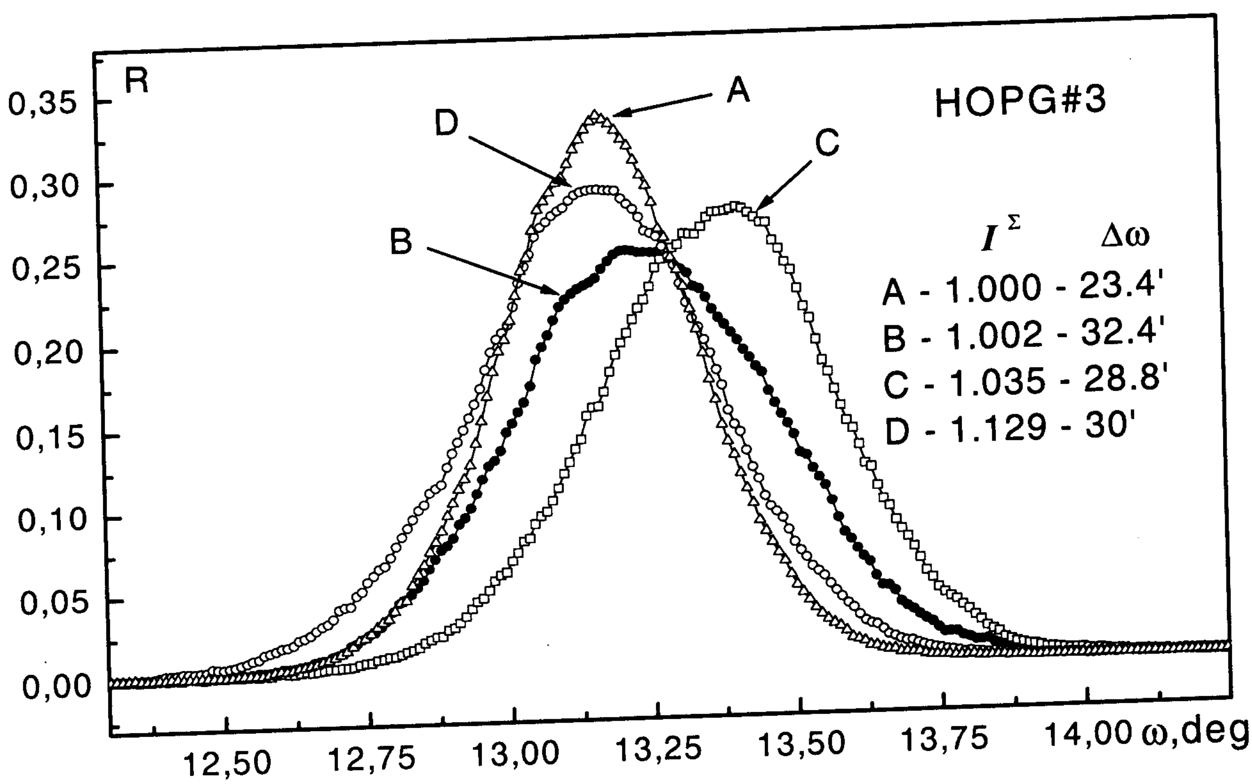

Fig. 4. ω-Scans for the 46 µm HOPG plate (see corresponding dots A-D in Fig. 3). I^{Σ} normalized integrals under reflection curves.

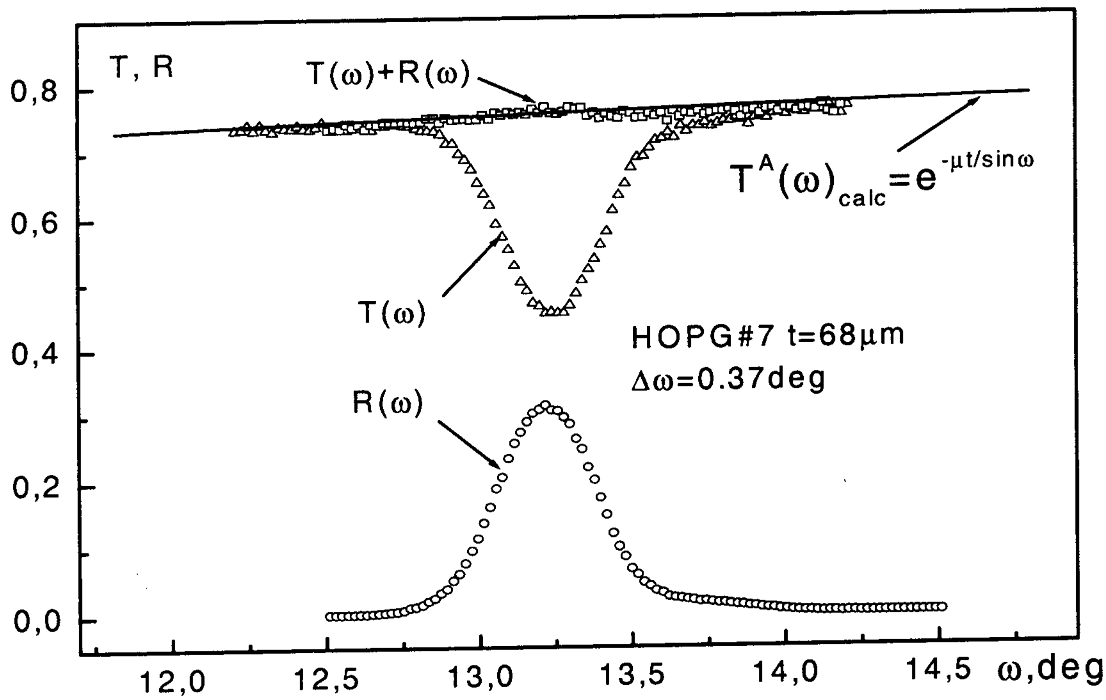

Fig. 5. Corresponding angle dependencies of reflection and transmission (ω-scans) for the 68 μm HOPG plate (illuminated area S=2 mm^2).

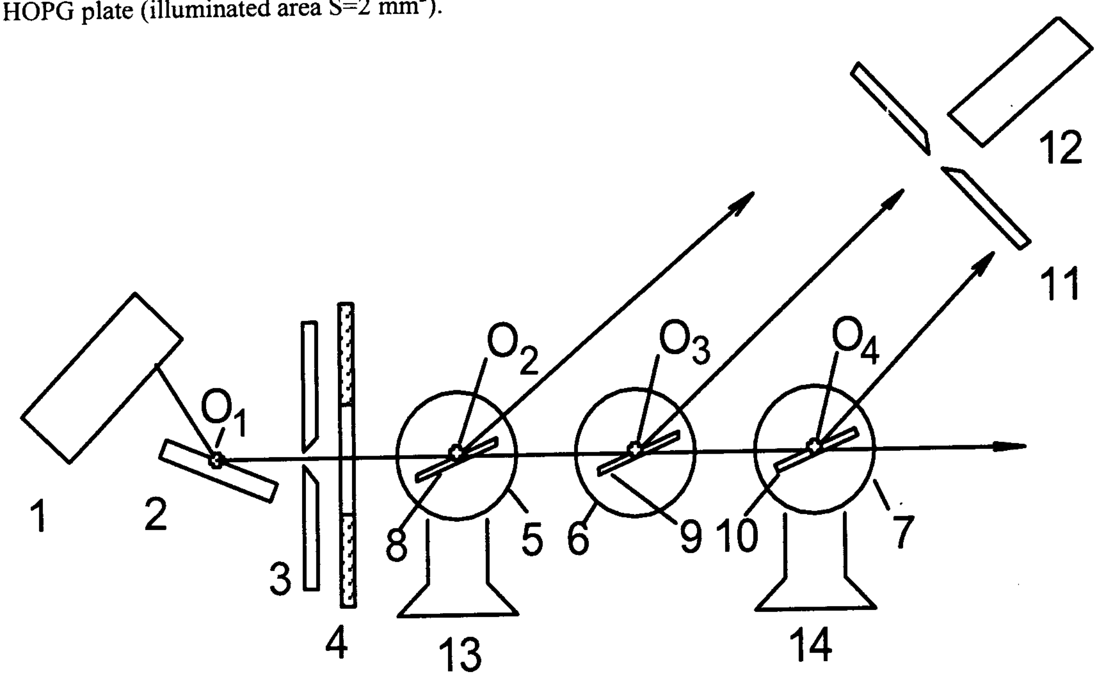

Fig. 6. Experimental setup of the echelon-monochromator: 1 - X-ray tube; 2 - Si crystal-monochromator; 3,11 - vertical slits; 4 - horizontal slit; 5,7 - rotating shafts; 6 - standard holder; 8-10 - HOPG plates; 12 detector; 13,14 - heads for single-crystal orientation.