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R. Barchewitz, R. Marmoret. X-UV multilayer reflectivity tests using windowless soft X-rays tube and synchrotron source. *Revue de Physique Appliquée*, 1988, 23 (10), pp.1661-1674. 10.1051/rphysap:0198800230100166100 . jpa-00245995

HAL Id: jpa-00245995

<https://hal.science/jpa-00245995>

Submitted on 1 Jan 1988

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Classification
Physics Abstracts
61.10F

X-UV multilayer reflectivity tests using windowless soft X-rays tube and synchrotron source

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(Reçu le 5 février 1988, révisé le 10 mai 1988, accepté le 2 juin 1988)

Résumé. — La mesure absolue de la réflectivité d'un miroir multicouche dans une condition voisine de son utilisation future constitue l'un des moyens de prévoir ses performances. Le profil de la courbe de diffraction permet d'évaluer l'influence relative des différents défauts (rugosités interfaciales intrinsèques ou induites par le substrat et défauts d'empilement). Ces mesures nécessitent l'utilisation d'un rayonnement monochromatique polarisé perpendiculairement au plan d'incidence. Ceci est toujours possible avec le rayonnement synchrotron qui présente une forte polarisation localisée dans le plan de l'orbite. Dans le cas de l'utilisation de raies caractéristiques émises par un tube à rayons X, il est nécessaire de polariser le rayonnement à l'aide d'une ou deux multicouches inclinées sous l'angle de Brewster (peu différent de 45°). Nous présentons dans cet article les techniques utilisées pour mesurer les réponses spectrales de multicouches à l'aide de tubes à rayons X sans fenêtre et de sources synchrotron.

Abstract. — The absolute reflectivity measurement of a multilayer mirror under conditions similar to its future use is one of the means to estimate its performances. The diffraction pattern profile permits to evaluate the relative influence of the different defects (interfacial roughness either intrinsic or induced by the substrates and stacking defects). For these measurements it is necessary to use a monochromatic radiation polarized perpendicularly to the incident plane. That is always possible with the synchrotron radiation which presents a high polarization localized in the orbital plane. In the case of characteristic lines emitted by X-ray tubes, it is necessary to polarize the radiation with the help of one or two multilayers oriented under Brewster angle (about 45°). We present in this article the techniques used for measuring the spectral responses of multilayers with a windowless soft X-ray tubes and synchrotron sources.

Recent progress in thin film deposition technique has motivated a renewed interest in artificially prepared microstructures. The multilayers can be used as Bragg reflectors at angles from near-grazing to near-normal incidence. This material with 2d spacing up to 40 Å, bridges the gap between long wavelength reflectors and Bragg diffractors at short wavelengths. In this paper, we present the experimental measurements of diffraction by means of characteristic X-ray lines and continuous synchrotron X-ray beams.

The first laboratory test of reflectivity as a function of the Bragg angle was made with goniometers designed to study the X-rays specular reflectivity with hard X-ray window closed tubes at 1.54 Å or 0.71 Å. A typical arrangement for this method is

sketched in figure 1 [1]. A recently modified apparatus used at 1.54 Å is presented in a companion paper in this review.

Other experimental measurements were carried out using X-ray tube-excited secondary targets as the source, in a commercial apparatus with a single-crystal spectrometer [2]. A scheme of a similar apparatus is presented in figure 2. In flat crystal spectrometers, the resolution is usually limited by a collimator. For this reason the spectral distribution of the fluorescence incident beam (Voigt function) is the result of a convolution of a Gaussian collimation function with a Lorentzian emission line profile. The multilayer angular width (rocking curve) is obtained by deconvolution [3]. At first we illustrate the resolution of the multilayer by comparison with a

Fig. 1. — Experimental arrangement for the X-ray total reflection method of a smooth solid surface.

Fig. 2. — Scheme of spectrogoniometer Philips PW 1520.

long spacing crystal (an acide phthalate). Likewise the reflectivity measurements are compared with those obtained by means of Langmuir-Blodgett layers under the same conditions [4]. Very soon, we felt the need to build specific instruments to realize absolute reflectivity measurements.

Synchrotron radiation is an ideal source to test the multilayers at any wavelength under conditions close to real use. But this needs sophisticated apparatus. The users perform preliminary tests by means of characteristic lines produced by windowless tubes. X and X-UV emissions extend up to hydrogen $\text{Ly}\alpha$ at about $1\,215.7\text{ \AA}$. The characteristic $\text{K}\alpha$ and $\text{L}\alpha$ lines are the more intense. Practically, they can be used with Bragg reflectors from $2.28\text{ \AA}$ ($\text{Cr K}\alpha$) to $113\text{ \AA}$ ($\text{Be K}\alpha$).

The increment can be reduced and the analysis range slightly extended by using the less intense $\text{M}\zeta$ lines. The use of a Finley source [5] permits to extend this domain up to $228\text{ \AA}$.

We give first some theoretical results to understand the type of test to be realized in order to obtain the parameters of multilayers, their resolution and reflectivity. Next, we shall consider successively the experimental apparatus used with laboratory X-ray tubes and with a synchrotron radiation source.

1. Theoretical results.

The X-ray dispersion behaviour of multilayers can be modeled from two viewpoints. One corresponds to the approach taken in the analysis of X-rays diffraction from crystal-scattering theory [6]. The other follows directly from optical dispersion theory as applied to various types of interference structures [7].

In the optical dispersion theory, Underwood and Barbee generalized Parratt's theoretical modeling based on a treatment of X-ray interference by a few contaminant layers on the surface of a glancing incidence X-ray mirror [1]. Figure 3 shows a typical theoretical reflection-diffraction curve obtained by the optical dispersion theory at $44.7\text{ \AA}$.

After the total reflection zone observed at small angles, a first series of fringes appear in the region where the reflectivity decreases rapidly. These fringes — Kiessig fringes — are sensitive to the space between the different strates and to the interfacial roughness. The main peaks are separated by $N - 2$ secondary peaks where N is the number of periods in the multilayer. Their regularity shows one of the bilayers good reflectivity. The successive position of Bragg peaks in different orders allows to deduce the

Fig. 3. — Theoretical reflectivity curve *versus* grazing angle at 44.7 Å.

Fig. 4. — Theoretical reflectivity curve *versus* the division parameter γ . Minima of reflectivity appears when γ is an integer multiple of $1/n$ (n : reflectivity order).

mean periodicity of bilayers ; their relative intensity gives informations concerning the thickness of the two elements of each bilayer (Fig. 4) [8].

Minima of reflectivity appear when the $(C - W)$ multilayer division parameter γ $\left(\gamma = \frac{d_w}{d_w + d_c}\right)$ is an integer multiple of $1/n$ (n : reflectivity order).

The difference between calculated and experimental reflectivity at the Bragg peak informs on the thickness defects and interfacial roughness [9] (Fig. 5).

2. Laboratory measurements.

We consider the characteristic X-ray lines produced by a windowless tube with an interchangeable anode.

The beam is generally collimated by a soller slit calculated to reduce the angular divergence to at least one tenth of the angular width of the expected

Bragg peak. It is then monochromatized by a crystal and analyzed by the multilayer.

The reflected intensity is generally analyzed below about $100 \text{ \AA}$ by means of a gas together with a quenching gas. For example, the geometry used at the Los Alamos National Laboratory is shown in figure 6 [10]. In this experimental equipment, using essentially the $\text{Al K}\alpha_1$ line, the electron energy is maintained at 2.6 keV and a $16 \text{ }\mu\text{m}$ Al filter is added to the primary beam to reduce Bremsstrahlung radiation. With these precautions the primary photon beam consists of 96% $\text{Al K}\alpha$ radiation and 4% of the photons are spread over a continuum from 700 eV to 1560 eV . A wavelength selective KAP crystal cut to diffract from the (001) planes has been included to further monochromatize the X-ray beam. In this type of measurements, the incident X-ray beam is not polarized. The main difficulty arises from the gradual polarization of the beam, which increases with the Bragg angle and reaches its

Fig. 5a. — Reflectivity *versus* the number of layers of the stack for various roughness at $44.7 \text{ \AA}$.

Fig. 5b. — Bandwidth of the mirror *versus* the number of layers for various roughness.

Fig. 5c. — Reflectivity *versus* the number of layers for various thickness drifts at $44.7 \text{ \AA}$.

Fig. 5d. — Bandwidth *versus* the number of layers for various thickness drifts.

Fig. 6. — The experimental geometry used for the reflectometer measurements with an X-ray tube at Los Alamos.

maximum at the Brewster angle ($i \approx 45^\circ$) [11], see figure 7.

We shall particularly consider an experimental apparatus which enables to suppress this effect. It was conceived in the Chimie Physique Laboratory in Paris [12]. It enables to insert a double multilayer system at 45° between the open X-ray tube and the multilayer being tested. The schematic drawing of the experiment is shown in figure 8. The goniometer D consists of two rotating plates mounted coaxially. A detailed description has been presented elsewhere [13]. The premonochromator C was equipped with two multilayers in the (+1, -1) mode orientated under the Brewster angle ($i_B \approx 45^\circ$). This equipment can not be used at wavelengths smaller than O_K radiation ($23.55 \text{ \AA}$) because the thickness of a bilayer cannot be lower than $15 \text{ \AA}$. For shorter wavelengths, natural crystals can be used, but their reflectivity is generally too low.

- The premonochromator has three functions :
- to isolate the characteristic line used
 - to suppress (or reduce) the intensity which may be reflected in higher orders (cf. Fig. 4)
 - to polarize the beam, so that the P-component be practically eliminated. The reflected beam includes only the S-component which is not sensitive to the polarization effect introduced by a multilayer.

The especially manufactured mirrors were made of up to 30 W-C bilayers ($2 d = 71 \text{ \AA}$, $\gamma = 0.43$) for the $CK\alpha$ line ($\lambda = 44.79 \text{ \AA}$) and 30 Ni-C bilayers

Fig. 7. — The calculated R_s , R_p total reflectivities and polarization rate of this Hf/Si multilayer, used at $304 \text{ \AA}$, show that quite good X-UV polarizers can be obtained with adapted multilayer mirrors ; this indicates also that reflectivity measurements outside grazing or normal incidence angles must take into account the polarization of the incident light, if absolute reflectivity coefficients are necessary.

($2 d = 102 \text{ \AA}$, $\gamma = 0.45$) for the $BK\alpha$ line ($\lambda = 67 \text{ \AA}$). The computed S-polarized reflectivity (perpendicular component) after double reflection is 4.4% for the $K\alpha$ carbon line and 4.8% for the $K\alpha$ boron line while the P-polarized reflectivity (parallel component) is negligible. The second harmonic is practically eliminated by appropriate choice of the division parameter γ . Consequently, the oxygen K emission, due to the oxidation of the anode which could appear in second order Bragg reflection ($\lambda = 23.55 \text{ \AA}$), is eliminated and does not perturb the measurements at $44.79 \text{ \AA}$. At $67.8 \text{ \AA}$, the third order is generally faint and is negligible in our arrangement (double reflection).

The studied multilayer rocking-curve is obtained by deconvolution of experimental curve by the spectral distribution of the polarized monochromatic incident beam (see p. 1661).

Figure 9, shows the Bragg peak bandwidth of

Fig. 8. — Principle of absolute reflectivity measurements with an X-ray tube.

Fig. 9. — Multilayer bandwidth *versus* Bragg angle at 44.7 Å.

Multilayer FWHM *versus* Bragg angle in the first order. The polarizer multilayers in the premonochromator present at the first order and optical spectral resolution further than 40 degree.

If the Bragg angle of the analyzed multilayer is small, the bandwidth of the monochromatic incident beam slightly contributes to the experimental rocking curve. In this case, a deconvolution is not necessary.

Theoretical Ni/C and Ni/B multilayers used in the premonochromator are respectively suitable for 44.7 Å and 67.8 Å.

Figure 10 [14], shows the measured and the calculated reflectivity curves of three Ni-C multilayers at 44.79 Å in the regions of the first and second order Bragg peaks. The parameters of these multilayers are indicated in the figures. In the case of figure 10c the extinction of the second order is due to the γ value ($\gamma = 0.5$) as predicted by the calculation (see Fig. 4). The theoretical reflectivity has been calculated by using a recursive procedure [1], [15] assuming an ideal periodic structure with a sharp interface, zero interfacial roughness and no random thickness errors in the periodicity.

Figure 11 shows an example of reflectivity measurements realized at 13.33 Å and 44.79 Å on a

Fabry-Perot multilayer. It consists of a "normal" multilayer coating in which extra periods of carbon have been incorporated. The contour represents the reflectivity which would give a multilayer with the same period as that of two multilayers situated on each side of the spacer. The narrowest fringes show the modulation effect induced by the spacer. Thicker the spacer is and more numerous are the fringes. More regular and reflexive is the multilayer and narrower are the fringes. A typical presentation of this study is published in a companion paper in this review (M. P. Bruijn).

3. Synchrotron measurements.

The synchrotron light sources emit a very intense continuous radiation which is elliptically polarized. It is well suited for the multilayers reflectivity tests in the UV to the X range. The degree of polarization of the radiation depends on its energy and on its direction with respect to the orbital plane. So, the S component is preponderant in the plane of the orbit. For the realization of these tests, it is necessary to use an isolated narrow spectral band of polarized radiation which is slightly divergent.

Fig. 10 (a, b, c). — Experimental and theoretical rocking curves of S polarized CK ($\lambda = 44.79 \text{ \AA}$) diffracted by Ni-C multilayers (first and second order Bragg reflection).

With this kind of light source, the reflectivity test of multilayers can be made either at fixed energy *versus* grazing angle or at fixed grazing angle *versus* energy. In accordance with the analyzed energy range, the radiation is monochromatized either with crystal or with grating.

3.1 CRYSTAL MONOCHROMATORS. — The radiation used is generally monochromatized by a double plane parallel crystal spectrometer. Over 500 eV the X-ray spectroscopy analysis requires either natural crystals (quartz, gypsum, mica, prochlorite, beryl, ...) or artificial crystals (RbAp, KAp, TlAp). In the 100-500 eV range Langmuir-Blodgett films can be used, but these materials are rapidly damaged by the destructive effect of synchrotron radiation. After monochromatization the radiation is used for the multilayer analysis in the $\theta - 2\theta$ goniometer [16].

Figure 12 shows a typical experimental arrangement for the reflectivity measurements of multilayers

realized at Stanford synchrotron facility (Chess) [17]. Slits S_1 and S_2 provided a beam to the multilayer with less than 2 arc second divergence. Another horizontal slit (not shown) limited the beam to a 1 mm horizontal width. With these vertical slits, the (220) silicon channel cut monochromator had an energy resolution of less than 1 eV at 8 keV and could be scanned from 7 to 40 keV.

A counter in position (2) could count the direct beam or could be positionned at (1) to receive the reflected beam. The ratio of the two measurements gave directly the experimental reflectivity.

The incident beam in fact was measured only on every fifth to tenth data point. Compton scattering from a 25 μm thick mylar sheet was monitored ahead of S_2 for the intervening point. A single channel analyzer then removed any contribution from harmonics and reduced pulse pileup.

Recently, apparatus especially built for X-ray optical tests have been realized [18], [19].

Fig. 11. — Reflectivity measurements *versus* Bragg angle realized at 13.33 Å and 44.79 Å on a Fabry-Perot multilayer.

Fig. 12. — Synchrotron radiation facility for multilayer tests at CHESS (Stanford). Synchrotron radiation was made monochromatic by a silicon (220) monolith collimated by S_1 and S_2 . The beam intensity ahead of S_2 was monitored by measuring the Compton scattering from a mylar sheet.

For such realizations two main features have been considered in order to :

- eliminate the radiation which can be reflected in higher orders
- fix the position and the direction of the monochromatic beam all over the spectral range analyzed.

Figure 13 shows the experimental set up used at Orsay storage ring (ACO, LURE) and realized by the Commissariat à l'Energie Atomique (Centre d'Etudes de Bruyères-le-Châtel) and the University P. et M. Curie (Laboratoire de Chimie Physique).

It consists of an energy low pass filter intended to cut off higher orders, a double plane parallel (-1 ; $+1$) monochromator in order to maintain a fixed exit beam, a $\theta - 2\theta$ goniometer associated to a flow gas proportional counter. These equipments are set in a vacuum chamber (10^{-6} Torr) connected to the radiation source by means of a differential pumping system through a symmetrical adjustable slit.

A rotating sample holder with 12 positions is installed beyond the collimator slit, it supports thin films as filters or samples for the photoabsorption experiments.

The low pass filter consists of two plane parallel mirrors placed in the path of the beam so that the axis of rotation of the first mirror is in the plane of the average orbit and perpendicular to the beam direction. Thus, the beam is always intercepted symmetrically whatever the glancing angle. The second mirror is situated so that its edge lies on line with the center of the first mirror. Each mirror can be independently translated and rotated, the glancing angle u can be adjusted with 0.02 mrad accuracy and the translation is made by $2\mu\text{m}$ steps. The mirrors have to be easily removable, because they undergo rapid alteration under a direct synchrotron beam. On the other hand lower wavelengths which could be selectively reflected in higher orders by the monochromator are practically suppressed.

Scheme of experimental set up.

A: Autocollimator telescope, B: N₂ liquid trap, C: Slit, D: Turbomolecular pump, E: Sample holder, F: Low pass filter, G: Double crystal monochromator, H: Goniometer, I: Proportional counter, J: Localization counter, K: Cryogenic pump.

Fig. 13. — Experimental arrangement for the reflectivity measurements of multilayers at Orsay storage ring (ACO-LURE).

The installation of mirrors on the synchrotron beam path provides a mean of absorbing a considerable quantity of radiation energy provided by a direct line and consequently prevents the dispersive device from rapid thermal damage.

The design of the two crystal monochromator has two independent rotating movements associated to a translation one, as shown in figure 14. The crystal rotates with 0.02 mrad steps and translates with 0.1 μm steps. This monochromator can be used for Bragg angles between 5 and 85 degrees according to the scheme of figure 14. The translation of the first crystal is given by the following relations :

$$T = h / \tan 2\theta \quad (\theta \leq 45^\circ)$$

$$T = h / \tan 2(\pi - \theta) \quad (\theta \geq 45^\circ)$$

where h represents the vertical deviation of the emergent beam.

For the whole angular range, the incident beam has to stay parallel to the translation direction. For this reason, the orientation of the second mirror of the low pass filter can be adjusted. In order to respect the symmetry of the setup it is essential that the rotation axis of the crystals be contained in the medium plane of the beam. In such conditions, the geometry of the setup is preserved. This configuration has been chosen on the one hand to obtain a fixed exit beam and on the other hand to use the same part of the crystal surface all over the spectral range. This particularity eliminates the influence of the crystal structural anomalies on the spectral distribution of the reflected radiation.

Our spectrometer has been equipped with two beryl crystals [3 (BeO), Al_2O_3 6 (SiO_2)] whose 2d spacing between the 10 $\bar{1}0$ planes is 15.956 Å. The independent rotation movements of the two crystals do allow the verification of the rocking curve between the two extremes of the spectral range.

The goniometer consists of two rotating plates ; one drives the dispersive device through a θ rotation, the other drives the photon detector through a 2θ rotation. The 2θ amplitude extends over 180 degrees. Each movement is executed with 0.02 mrad

accuracy using the stepping motor. One particularity of the experimental device is to work in horizontal and vertical positions in order to analyse both polarizations of radiation.

The photon detector can be of various types. At present, above about 100 eV we use a gas flow ionization counter filled with a 90-10 Ar-CH₄ mixture. The pressure of the gas and the bias voltage can be adjusted to be matched to the energy of the radiation of interest. The anode is a 50 μm -diam. tungsten-gold wire. The window of the counter has a 15 $\times$ 2 mm useful aperture. It is made of either a 2 μm -thick makrofol film or a 0.4 μm -thick polypropylene foil. A channel electron multiplier (CEM) possibly associated with an alkali halide or gold coated photocathode could be employed, which is favorable for long wavelengths.

In order to achieve the setting of our optical elements in the beam we use a localization counter with a thin entrance window.

The data collection is automatically controlled by a Hewlett-Packard 85B microcomputer through the standard IEEE 488 bus. A dispatching unit can pilote independently the different elements of the setup. An event counter manages the breakings of the bus controller when a data acquisition is realized.

Figure 15 shows typical results obtained with this apparatus.

The Bragg peak observed at the first selective reflection order splits into two peaks denoted a and b.

These two peaks have also been observed in the second selective reflection order. The stacks of bilayers are 51 Å and 48.5 Å apart respectively. Another peak, denoted by c appears in the second selective reflection order. This is probably due to a stack of bilayers separated by 49.75 Å located near the substrate. We can explain this by saying that the penetration depth of the radiation for the second reflection order is more important than that of the first order. The third stack can be seen by the incoming radiation. These defects indicate a structural disagreement between the effective and the expected structure. The thickness measurements of

Fig. 14. — Depiction of the scanning of the monochromator to achieve a constant deviation device.

Fig. 15 (a, b). — Reflectivity curve *versus* Bragg angle for the first and second orders.

layers carried on the electronic photomicrograph are in good agreement with these results. They display three groups of stacks having 26 bilayers ($d \approx 48 \text{ \AA}$), 11 bilayers ($d \approx 51 \text{ \AA}$) and 3 bilayers located near the substrate ($d \approx 50 \text{ \AA}$) respectively. Our experimental reflectance spectra observed

versus energy for the first reflection order confirm these results.

3.2 GRATING MONOCHROMATORS. — The LSM's samples were characterized generally on the grashoper monochromator with the incident beam pho-

Fig. 15c. — Reflectivity curve *versus* energy for the first order.

ton energy varying continuously from 40 to 500 eV [20], [21].

Some reflectivity tests with both polarization directions have disclosed the possibility to make X-UV polarizers, which are very useful for the characterization of synchrotron radiation sources [11].

The experimental geometry used for the diffraction profile measurements at Stanford synchrotron radiation Laboratory is show schematically in figure 16 [10].

The output of the monochromator was refocused by a bendable cylindrical mirror and the beam diameter defined by a set of apertures located between the refocussing mirror and the goniometer.

The goniometer provided synchronous rotation of the mirror and detector in the whole range of angle (θ to 2θ).

A channel electron multiplier (C.E.M.) with Aluminium or CsI photocathode was employed as a detector of the diffracted beam.

When the sample was removed from the photon beam the incident intensity could also be measured across the same energy region. The beam energy width and divergence were chosen such that they did not contribute significantly to the energy width of the diffracted beam. The LSM reflectivity was measured directly by dividing the diffracted beam intensity by the incident beam intensity at the same

Fig. 16. — The experimental geometry used for the diffraction profile measurements at SSRL.

energy. Correction to the data were applied for background counts in the C.E.M. and to normalize the effect of decaying ring currents.

Conclusion.

This article shows the present methods which permit to measure the multilayer rocking curve.

The P component of the radiation reflected by multilayers becomes very small at incidence angles near 45° , because the real part of the refractive index is very close to unity for X-rays [22]. Polarization effect by the multilayers has been shown with an experimental set up based on the Nörrenberg apparatus principle [23/24].

So it is necessary to cut off the P component in order to avoid the disadvantage resulting from the intensity variation as a function of the Bragg angle. The use of the S component permits to make absolute reflectivity measurements of multilayers out of the grazing incidence. The synchrotron radiation is perfectly adapted because it is strongly polarized in the orbit plane. From the natural light emitted by an X-ray tube the P component can be cut off by a first reflection on one or two multilayers oriented along the Brewster angle [12].

The present theories do not permit to clearly separate the role of the roughness due to nucleation from the role of the interface scattering. Reflection measurement using long wavelengths can give information on thickness errors, substrate waviness and roughness of the interfaces [9, 25, 26].

Reflectivity tests realized at different wavelengths must permit to solve this problem, because they

Fig. 17. — X-ray beam splitter realized by multilayer mirrors on thin (transparent) substrates (or, ideally, free standing multilayers).

make variations of the ratio between the defects size and the diffracted wavelength.

The normal incidence tests are particularly interesting for the realization of X-ray telescopes [28].

An new test consists in using multilayer mirrors on thin (transparent) substrates. X-ray beam splitters [27] can be tested, as shown in figure 17. The first experimental results of multilayer beam splitter used in the X-rays Laser cavity have been obtained at $130 \text{ \AA}$ and $208 \text{ \AA}$ [29], [30]. Recently a Carbon Tungsten beam splitter has been tested at $12.4 \text{ \AA}$ [31].

Acknowledgments.

The authors are grateful to Professor Y. Cauchois for valuable advice.

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