A single W/B_4C transmission multilayer for polarization analysis of soft x-rays up to 1keV

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Abstract: A transmission W/B_4C multilayer has been designed and characterized which shows significant phase retardation up to a photon energy of 1 keV, when operated near the Bragg condition. This allows, for the first time, the full polarization vector of soft x-radiation to be measured up to 1 keV in a self-calibrating method. Quantitative polarimetry is now possible across the 2p edges of all the transition metals.

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Introduction

Soft X-ray (SXR) synchrotron radiation with variable polarization is a sophisticated probe of the properties of matter. Many of the most advanced experiments take advantage of the inherently high degree of linear and/or circular polarization of such a source which is, in general, an elliptical undulator. However the beamline optics may seriously modify the output of the undulator light source [1]. Of particular interest are experiments probing magnetic circular dichroism from the 2p absorption edges of transition metals. Thus there is increasing demand to characterize beamline output over the range 500 eV to 1 keV.

Polarimeters designed to deliver the four Stokes parameters of a source rely on a phase retarder followed by a (linear) polarization analyzer, both of which are capable of independent azimuthal rotation about the beam [2–6]. In the VUV range the preferred retarders and analyzers are reflection optics [2, 7–12], while in the SXR range they are transmission and reflection multilayers [3, 13–22]. In the hard x-ray region, various standard monochromator crystals in Laue or Bragg geometry have been used [23]. Recently theoretical calculations have been performed for a mica crystal acting as a phase retarder [24] and calculations and

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measurements have been performed for a mica crystal acting as a polarizer [25, 26] covering the range 700 eV to 1.1 keV. Calculated phase retardances of up to 90° were reported.

Because of the enhancement of the multilayer performance near absorption edges, most multilayers have been designed to be used near the 2p absorption edges of the constituting materials (Cr/C, Cr/Sc, Mo/Si, Ni/Ti, and Ni/V). At best, they can operate at two distinct energies (e.g., Sc at 397 eV and Cr at 550 eV) [13, 27, 28].

MacDonald *et al* [21] reported the use of a non-resonant (W/B_4C) transmission multilayer as a phase retarder, capable of a complete polarization analysis of a synchrotron radiation beamline over an extended and continuous range of energies. This was the first report of a full characterization of a beamline in the vicinity of the 2p absorption edges of Fe, Co and Ni. However, the phase shift of the multilayer was low (approx 8°), with the consequence that data analysis relied on modeling of the multilayer. The W/B_4C phase retarder gains its contrast from the broad W–N shell absorption (4s, 4p, 4d, 4f) between ~200 and ~500 eV. Since there are no strong resonances in the atomic scattering factors between ~500 and ~1800 eV in any of the constituents [29] it acts as a non-resonant phase retarder. Thus it may be used to probe the polarization state of a beamline on either side of a transition element 2p absorption edge.

Here we report on an improved W/B_4C multilayer that shows sufficient phase shift up to a photon energy of 1 keV to allow a full beamline characterization without recourse to modeling of the multilayer. The 2p absorption edge of Zinc, the last of the first row transition metals is at approximately 1020 eV.

Experimental

Previously MacDonald *et al* [21] reported a multilayer that exhibited a phase retardance of approximately 8° at 850 eV photon energy. The co-deposited, transmission and reflection multilayers were designed to operate near the Brewster angle at the Fe (2p) absorption edge. The limit on performance is due to an estimated interface width of 0.275 nm.

Kimura *et al* [20] have shown that, while, for an ideal case (zero interface width) the phase retardation as a function of grazing angle (and hence, d) of a transmission multilayer is optimized at 45°, for a case with a finite roughness/interdiffusion the optimum occurs at more grazing angles. In this way, the ratio of roughness to d spacing of the new transmission multilayer (and hence its performance) was improved by choosing to design the multilayer to operate at more grazing angles. In this case the design criterion was to operate at 25° grazing angle at 850 eV. The present transmission multilayer used was manufactured by the X-ray Company, Russia and was found to be W/B_4C (d = 1.743 nm, Γ = 0.382, N = 350) as described below. It is an unsupported multilayer on a 15 x 15 x 0.5 mm^3 Si frame with a central 8 mm diameter unsupported region and is the main focus of this paper.

Additionally two reflection multilayers were used as polarization analyzers in the polarimetry data sets. For the 720eV complete polarization data set a W/B_4C multilayer (d=1.2 nm, Γ=0.5, N=300) was used [30]. For all other such data sets, a reflection W/B_4C multilayer (d=1.03 nm, Γ=0.5, N=150), was used [30]. The reflection multilayers were deposited on silicon wafers. These multilayers were chosen because, amongst those available, they operated nearest to the Brewster angle. They are required as (linear) polarization analyzers when measuring full polarimetry data sets.

The measurements were carried out using the BESSY 6 axes polarimeter [3] on the helical undulator beamline UE56/2 PGM2 at BESSY [31–33]. The polarimeter houses two optical elements on two azimuthal rotation stages - α (for the transmission multilayer polarizer) and β (for the reflecting multilayer analyzer). The angle of incidence θ_p and θ_a of these elements can be independently set to match the Bragg angle of the structure in question. There is also a $2\theta_a$ arm on the reflection stage to hold and position the GaAsP-photodiode detector in two dimensions.

The results described below can be divided into four groups. First the transmission of the multilayer is recorded at normal incidence between 500 eV and 1100 eV. This is to determine the proportion of W in the transmission multilayer. Second, the absolute reflectivity of the

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transmission multilayer is recorded across the Bragg condition at several photon energies, for both s- and p- polarized light. (This involves recording a θ_a-$2\theta_a$ scan with the transmission multilayer placed in the analyzer position.) This is to provide data on the Bragg spacing, and interfacial roughness/interdiffusion of the transmission multilayer allowing it to be modeled. Third, since two reflection multilayers are required for the full polarimetry scans, the angle of their maximum Bragg reflectivity and the polarizance of that reflectivity are measured at several photon energies. This is because it useful to know these values for the full polarimetry scans. Fourth, using both the transmission and reflection multilayer (as a phase retarder and linear analyzer, respectively) full polarization measurements were taken by independent azimuthal rotation, about the beam, of the multilayers. These measurements were performed at photon energies of 720 eV, 820 eV, 920 eV and 1020 eV with the undulator set to give elliptically polarized light. This gives the full Stokes parameters for the beamline.

Minor misalignments between the azimuthal rotation axes, α and β, and the optical axis were corrected during the collection of the full polarization as described in Gaupp *et al* [34]. Detector intensities were normalized to the drain current from a gold mesh inside the polarimeter. Typical collection times for a complete polarization data set were approximately one hour and a typical beam lifetime was greater than ten hours.

Results

A normal incidence transmission curve was recorded for the multilayer between 550 eV and 1100 eV, with no reflection multilayer present and is shown as Fig. 1. From the known W and B_4C absorption, it is then easy to determine the ratio of the W layer thickness to total bilayer thickness (Γ), assuming that there are 350 bilayers in the sample and that the d spacing is given by the Bragg reflection measurements described below. Using the published optical constants from CXRO [35], Γ was found to be 0.382.

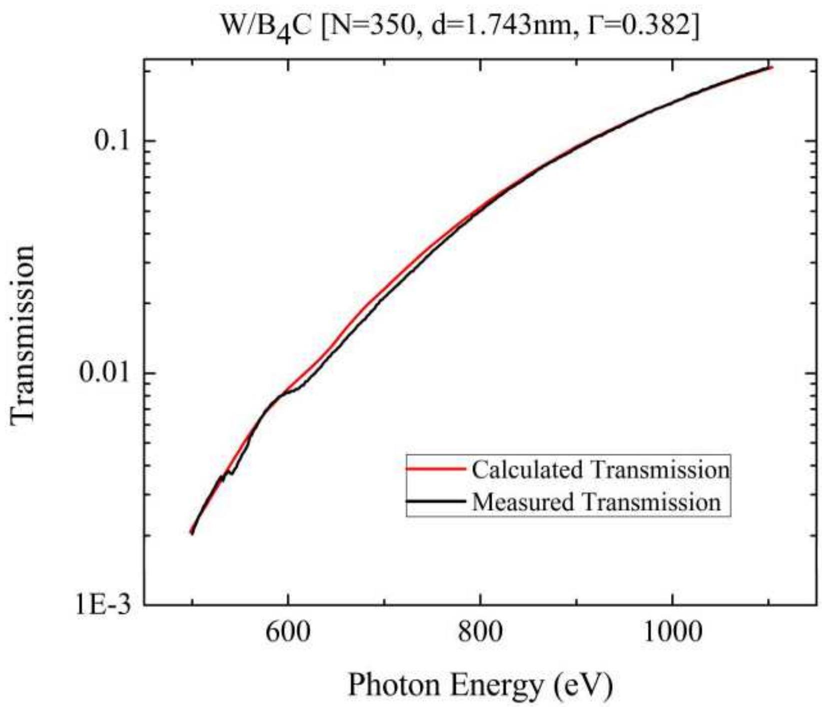

Fig. 1. Normal incidence transmission of the W/B_4C multilayer recorded between photon energies of 500 eV and 1100 eV. The black line shows the measured data and the red line shows and calculation

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The d layer spacing of the transmission multilayer and interface width were determined by measuring the absolute reflectivity of the transmission multilayer. This was done by placing the transmission multilayer on the reflection stage of the BESSY polarimeter and recording s- and p- polarized reflectivity curves, assuming that when operated in its linear mode the undulator and beamline give nearly perfectly polarized output (this assumption is confirmed later). Reflectivity plots (Fig. 2) were recorded at 720 eV, 820 eV, 920 eV and 1020 eV. The data were modeled using the program IMD [36] with the d spacing and interface width as free parameters. (Adjusting the d-spacing of the multilayer fixes the position of the Bragg peak and adjusting the roughness/interdiffusion fixes the absolute reflectivity of the Bragg peak). A known 300 μRadian error in θ_a, on azimuthal rotation of the analyzer has been corrected [34]. This defines the d-spacing of the multilayer as 1.743 nm and the roughness as 0.292 nm scaled as the error function using IMD.

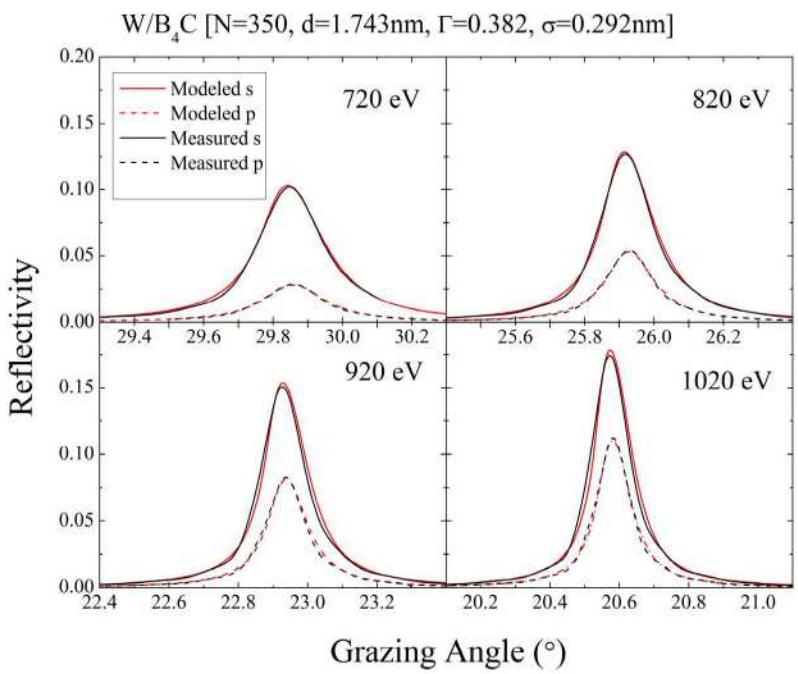

Fig. 2. Absolute reflectivity of the transmission multilayer measured at a) 720 eV, b) 820 eV, c) 920 eV and d) 1020 eV using both s- and p- polarized light. The black lines show measured data and the red lines show modeled results. Solid lines are for s- polarization and dashed lines are for p- polarization. The reflectivity scale for each graph runs from 0 to 0.2, while the angular range of each graph is 1.0°.

Because they are used in the polarimetry analysis, the ratios of the p- to s- polarized reflectivities at the maximum of the Bragg peak of the reflection multilayer were measured separately at the energies 720 eV, 820 eV, 920 eV and 1020 eV and were found to be 0.00415, 0.0095, 0.023 and 0.0955 respectively. As described above the reflection multilayer with d = 1.03 nm was used at 720 eV, and the reflection multilayer with d = 1.2nm was used at the other energies. Thus despite not being at the Brewster angle, the polarizance is sufficiently large for polarimetry purposes over the whole energy range. The procedure was to set the undulator to produce linearly polarized light and to measure the maximum reflected intensities at β=0° and β=90°. This assumes perfectly linearly polarized light (S_1=1). If this is not the case the value measured is an upper limit. These data are used as a starting point in the fitting procedure.

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The polarization of the incident, elliptical radiation was measured at the same four energies: 720 eV, 820 eV, 920 eV, and 1020 eV. These data sets consist of α and β scans at several closely spaced θ_p around the relevant polarizer Bragg angle. In these measurements, the polarizer stage holds the transmission multilayer and produces a phase retardance dependent upon the polarizer azimuthal angle, α. The analyzer stage holds one of the reflection multilayers and, by rotating β, gives the linear polarization of the soft x-radiation, following transmission through the polarizer, with the polarizance measured above. The angle, θ_a was kept fixed at the relevant analyzer Bragg maximum.

The polarizer azimuthal angle, α, was recorded over the range 0° to 360° in 10° steps, while the analyzer azimuthal angle, β, was recorded from 0° to 315° in 45° steps. In the fitting of the data, it is implicitly assumed that the angles of α and β are correctly aligned to each other as was checked by visual inspection to within ±0.5°. It is important that the background (dark) level of the detector is consistently checked and properly subtracted. At the end of each day a complete scan was taken with the beamline shutters closed to act as a dark current reference. There was no visible structure in this background and an average value of this scan was subtracted from each point in the relevant polarization scans.

The fitting procedure simultaneously determines the Stokes parameters and (S_0, S_1, S_2, S_3) and the optical properties of the multilayers: the phase retardance ($\Delta = \delta_p - \delta_s$) and transmission (intensity) ratio (T_p:T_s) = $|t_p|^2/|t_s|^2$ of the polarization stage and the reflection (intensity) ratio (R_p:R_s) = $|r_p|^2/|r_s|^2$ of the analyzer stage [37,2,4]. The data sets taken at various θ_p around the Bragg angle all must have the consistent Stokes parameters and reflection ratios. Data were taken with elliptically polarized light from the undulator across the whole range of θ_p probed and with linearly polarized light at one θ_p to act as a calibration point.

The fitting equations of Gaupp and Mast [2] and of Koide *et al* [4] are essentially the same, except that the definitions of α and β differ by 90°. Gaupp and Mast arrange the terms in like functions of α and β, whereas Koide *et al* arrange the terms in groups corresponding to the Stokes parameters. Here the Koide et al formalism is reproduced, except using the definitions of α and β and the Stokes parameters corresponding to Gaupp and Mast.

$$
\begin{aligned}
&I(S_0, S_1, S_2, S_3, \psi_1, \psi_2, \Delta, \alpha, \beta) = \\
&\left(\frac{S_0}{S_1}\right)\left[1+\cos 2\psi_1 \cos 2\psi_2 \cos(2\alpha - 2\beta)\right] \\
&+\left(\frac{S_1}{S_0}\right)\left[\cos 2\psi_1 \cos 2\alpha + \tfrac{1}{2}\left(1+\sin 2\psi_1 \cos\Delta\right)\cos 2\psi_2 \cos 2\beta + \tfrac{1}{2}\left(1-\sin 2\psi_1 \cos\Delta\right)\cos 2\psi_2 \cos\left(4\alpha - 2\beta\right)\right] \\
&+\left(\frac{S_2}{S_0}\right)\left[\cos 2\psi_1 \cos 2\alpha + \tfrac{1}{2}\left(1+\sin 2\psi_1 \cos\Delta\right)\cos 2\psi_2 \sin 2\beta + \tfrac{1}{2}\left(1-\sin 2\psi_1 \cos\Delta\right)\cos 2\psi_2 \sin\left(4\alpha - 2\beta\right)\right] \\
&+\left(\frac{S_3}{S_0}\right)\left[\sin 2\psi_1 \cos 2\psi_2 \sin\Delta \sin\left(2\alpha - 2\beta\right)\right]
\end{aligned}
$$

Where S_0, S_1, S_2 and S_3 are the Stokes parameters, α and β are the azimuthal rotations of the polarizer and analyzer and Δ is the relative phase retardance between the p- and s-polarization components ($\delta_p - \delta_s$). ψ_1 and ψ_2 are functions of the ratio of the transmitted (or reflected) amplitudes of the p- and s- components on passing through the polarizer (1) or analyzer (2). $|t_p|/|t_s| = \tan\psi_1$ and $|t_p|/|t_s| = \tan\psi_2$.

The definitions of α and β and the Stokes parameters are such that α=β=0° and α=β=180° imply reflections in the vertical plane, where, α=β=0° implies deflection down to the ground and α=β=180° implies deflection towards the sky. α=β=270° implies deflection towards the right, α=β=90° implies deflection to the left with respect to the light direction. All motors rotate with the right hand rule with respect to the direction of light. S_1 = + 1 implies horizontally polarized light. S_1 = −1 implies vertically polarized light, S_3 = + 1 implies left

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hand circularly polarized light (LCP or σ^+), $S_3 = -1$ implies right hand circularly polarized light (RCP or σ^-). This follows the "customary" definition of circular polarization [1] where the polarization is left handed when, to an observer looking in the direction from which the light is coming, the end point of the electric vector appears to describe the ellipse in a counter-clockwise sense.

The 720 eV data set

All the data for 720 eV energy are fit simultaneously, keeping the Stokes parameters and the analyzer parameters the same for all θ_p but allowing the phase retarder parameters (Δ and $T_p{:}T_s$) to vary. The analyzer parameter $R_p{:}R_s$ was fixed at the value independently measured (0.00415). The data were fit with equal weighting. The results are shown in Table 1 and Figs.3(a) and 4(a).

The determination of Δ and S_3 comes from two types of term in the fitting equation. The unpolarized fraction of light is independent of Δ. The circularly polarized fraction (S_3) contains a term whose modulation, as a function of the azimuthal rotations α and β, scales as $S_3 \sin\Delta$. Thus for a beam that does not contain a significant fraction of linearly polarized light it is impossible to determine S_3 and Δ independently. It is the linear component in the beam that allows Δ to be determined uniquely, as it contains terms that modulate as ($1 \pm \sin 2\psi_1 \cos\Delta$). The fit is ill determined for small values of Δ as $\cos\Delta \approx 1$. (Although the linearly polarized component can determine the magnitude of Δ, because cosine is an even function, it cannot determine the sign of Δ, which requires the circularly polarized component.)

Fitting each polarization data set individually, including those with small Δ, gives uncertainty in the S_3 and the total polarization of ~0.15, as determined by the spread of results obtained. The global fit of all the data sets improves this with an estimated uncertainty in S_3 of ~0.02. Each individual polarization data set took about one hour to collect, so the total time required to characterize the beamline at 720 eV was about one day.

The 820 eV, 920 eV and 1020 eV data sets

A different approach was taken to collecting and analyzing the data at 820 eV, 920 eV and 1020 eV. In these cases a polarization data set was taken with the undulator set to give predominantly linear light. This was fit and used to determine the polarizer parameters and Stokes parameters at a specific polarizer Bragg angle (θ_p). The analyzer polarizance ($R_p{:}R_s$) was constrained, in the fit, to be less than or equal to the values measured above. A second data set was recorded at precisely the same θ_p, and photon energy (monochromator settings), but with elliptically polarized light – the polarizer parameters previously determined (Δ, $T_p{:}T_s$ and $R_p{:}R_s$) were used in the fit of this data set and the Stokes parameters were thus determined. Using this technique the uncertainty in S_3 and the total polarization is estimated to be ~0.02. However the requirement is only to record two data sets, so the total time required is approximately two hours. It is not necessary or assumed that the linear polarized data set is perfectly polarized. The results of this analysis are shown in Table 1 and Figs. 3(b)-(d) and 4(b)-(d). The circled points in Figs. 3 and 4 show where the linear polarization data sets were recorded.

Additional polarization data sets were recorded at closely spaced θ_p. These were analyzed using the Stokes vector and the analyzer polarizance ($R_p{:}R_s$) determined above as fixed parameters in the fit and the phase retarder parameters (Δ and $T_p{:}T_s$) as variable terms in the fit. These results are also plotted in Figs. 3(b)–(d) and 4(b)–(d) along with the modeled results. It should be emphasized that no calibration of the multilayer based on the IMD model was used.

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Table 1. The output Stokes parameters of Beamline UE56/2 PGM2 at BESSY, with the undulator set to produce elliptical or linear light. The total polarization $P=\sqrt{(S_1^2+S_2^2+S_3^2)}$ is also shown. The estimated uncertainty in each Stokes parameter is ±0.02. The values given are from the results of the fits described above and are not constrained to be less than or equal to 1. The data are rounded to two decimal places, with P being calculated from the more precise values before rounding.

Photon Energy	Elliptical Polarization				Linear Polarization			
	S_1	S_2	S_3	P	S_1	S_2	S_3	P
720 eV	0.42	0.01	0.91	1.00				
820 eV	0.46	0.01	0.87	0.99	1.01	−0.01	0.01	1.01
920 eV	0.49	−0.01	0.86	0.99	0.99	−0.01	0.01	0.99
1020 eV	0.48	0.03	0.84	0.97	1.02	0.00	0.03	1.02

There is ambiguity in reference [21], as the geometry of the analyzer and Stokes parameters are not defined. If using this geometry and the definitions in this paper the values for S_1 in reference [21] should be multiplied by −1. These are the standard definitions used at BESSY.

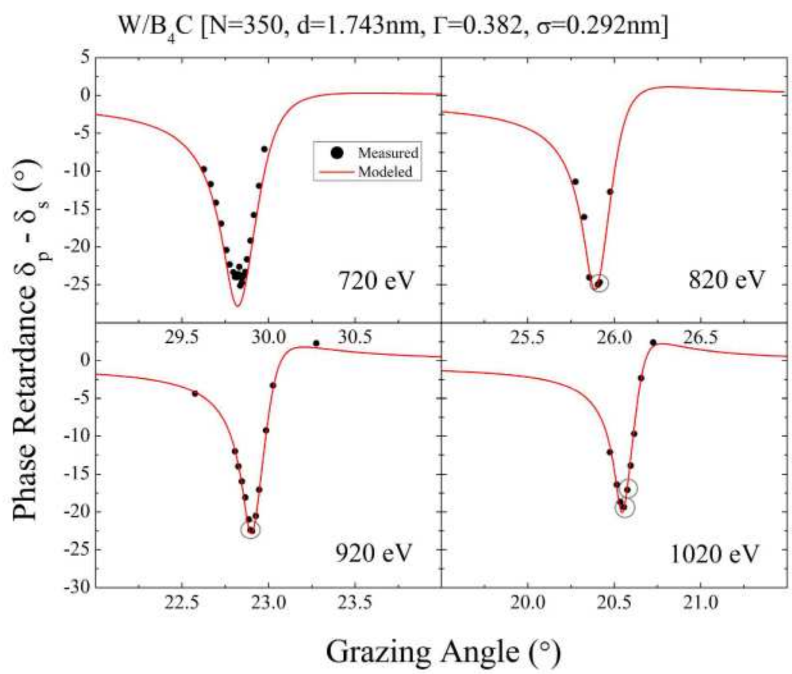

Fig. 3. The phase retardance (δp - δs) of the transmission multilayer polarizer as modeled and as measured at a) 720 eV, b) 820 eV, c) 920 eV and d) 1020 eV photon energies. The points marked in circles are where the data was cross-calibrated with linear polarized radiation. The phase retardance scale on each frame runs from 30° to +5°, while the angular range of each graph is 2°.

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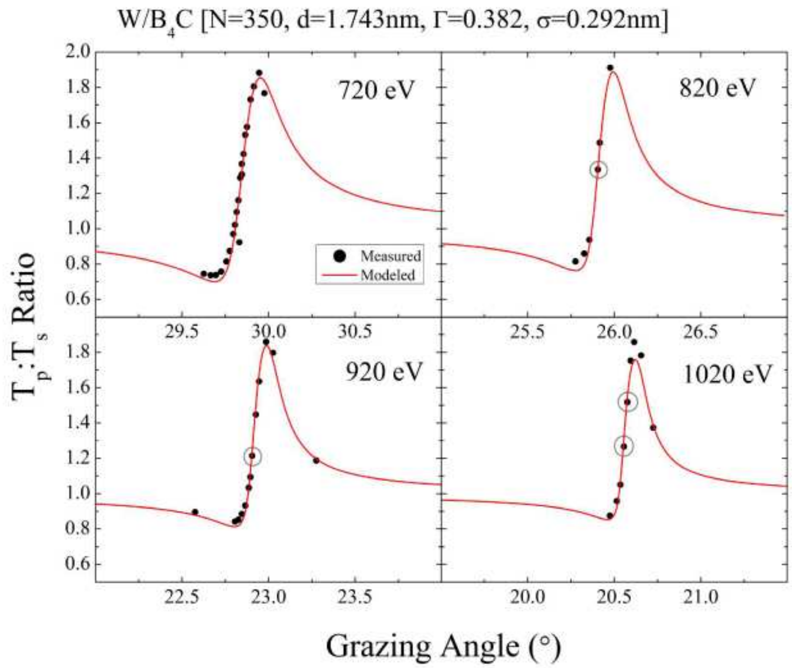

Fig. 4. The polarizance (Tp/Ts) of the transmission multilayer as modeled and as measured at a) 720 eV, b) 820 eV, c) 920 eV and d) 1020 eV photon energies. The points marked in circles are where the data was cross-calibrated with linear polarized radiation. The vertical scale on each frame is identical while the angular range of each frame is 2°.

Summary and outlook

A non-resonant multilayer phase retarder on the basis of a freestanding W/B_4C multilayer used in transmission has been designed and characterized. This single optical element was used to measure the full polarization vector of a beamline at photon energies between 700 eV and 1000 eV. The multilayer has sufficient phase shift to be self calibrating, unlike a previous multilayer [21], which required calibration using a modeled system.

The non-resonant nature of the phase retardance means that data can be taken at any energy required. Hence, quantitative polarimetry is possible now at the 2p edges of the magnetic substances Fe, Co, and Ni, for the benefit of the magnetic circular dicroism-spectroscopy work done there. The Cu and Zn edges can also be covered.

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