DOUBLE MULTILAYER MONOCHROMATOR
WITH FIXED EXIT GEOMETRY

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Abstract

To use the power of Total Reflection X-ray Fluorescence Analysis (TXRF) as an analytical method for very low detection limits on surfaces it is necessary to have a monoenergetic, energy adjustable x-ray beam with an intensity as high as possible. Therefore a wide band pass monochromator is desired because it increases the flux of photons impinging on the sample and an energy width of up to 200 eV does not affect the choice of exciting or suppressing a particular element. The importance of the high flux of photons within the energy band chosen will result in improved sensitivity and finally in better detection limits. Multilayers (ML) produced at PSI have been tested and successfully used for that purpose. Alignment of the TXRF set-up for a given energy is time consuming and therefore a stable beam geometry is required for the energies adjusted. So in case of only one ML in the beam path this can not be fulfilled and requires a realignment of the TXRF set-up for each energy. A double ML monochromator unit allows the design of a fixed exit geometry for a wide range of energies. The technical solution, the design and construction for such an arrangement is described. The experience is discussed and results from experimental series with x-ray tubes, as well as at the HASYLAB Hamburg, Germany, with the newly designed equipment are presented. Finally an inherent advantage of double ML systems is the possibility to suppress higher.

Introduction

TXRF is an accepted method to determine small amounts of chemical elements on surfaces with extremely good detection limits [1,2]. The geometrical arrangement provides a couple of substantial advantages. Efficient excitation by the effect of total reflection. Both primary and reflected beam add to fluorescence radiation with nearly double intensity. As the incident beam scarcely penetrates into the material, scatter radiation is reduced. Additionally the scatter contribution shows a minimum for 90 degree between incident and scattered radiation. Both imply reduced spectral background and limited counting rates for the scatter peaks, an important aspect for energy dispersive detection systems where high count throughput is a problem. Another consequence of the small incidence angle is the possibility to mount the detector very close to the sample. This results in large detection solid angles.

To exploit the advantages of TXRF and to improve the detection limits several pre-conditions have to be created. TXRF needs a spatially very stable beam because small angle variations cause tremendous changes in excitation conditions. Slight vertical variations as well cause large
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horizontal shifts whereby the sample is not illuminated properly. The beam may also show a spatial profile that leads to changes in the excitation conditions if shifts occur. If single elements are of interest it is important to use monoenergetic radiation with an energy just above the absorption edge of the element. Therefore energy tuning by means of the monochromator is necessary. This monochromator should provide a spectral distribution with a FWHM value smaller than the difference of the absorption edge energies of neighboring elements if multiple elements are of concern. It also has to be able to resist very intense beams. Particularly if the excellent properties of synchrotron radiation should be used it is important that the monochromators withstand the high energy densities transferred. The increase of the intensity of the photon flux leads to an increase of the sensitivity and therefore to achieve better detection limits.

Experimental

Within the experiment for monochromatization multilayers were used. Beside the wide band pass, another advantage of multilayers compared to single crystal monochromators is the possibility to vary their parameters over a wide range. With appropriate chosen parameters (d-spacing, division ratio \(\Gamma\), layerpair number \(N\), roughness) high intensity transmission and wide energy tuning can be achieved that match the experimental demands. To provide an energy adjustable monochromator with 'Fixed Exit' it is necessary to arrange two monochromators, in case of two equivalent multilayers, in \(+n/-n\) geometry [3-6]. Batterman and Bilderback suggest a right angle geometry where the axis of rotation coincides with the point where the normal of the first multilayer intersects the elongation of the surface of the second multilayer [7]. The disadvantage of this method is that the line where the beam impinges on the first multilayer shifts if the energy changes. This can be rectified if the axis of rotation is identical to the intersection line of the incident beam on the surface of the first monochromator (Figure 1). Due to the limited length of the multilayers the second multilayer must be positioned on a translator parallel to its surface.

**Figure 1:** Fixed Exit Geometry: To select different energy adjustments the whole system has to be rotated around the axis tangent to the surface of the first multilayer. If the single monochromatized beam does not hit the second multilayer the multilayer has to be translated parallel to its surface.
For changing the energy the complete system must be rotated so that the first multilayer matches Bragg's law for the according energy. Then the second multilayer must be translated to the point of intersection of the centre of the surface with the monochromatized beam in order to reflect the beam parallel to the incident beam. In fact the so called 'Fixed Exit' is not really perfectly fixed. But in case of hard x-rays, which also means small angles of incidence, the height shift can be neglected. For the reason of the primary adjustment it is necessary that each multilayer is positioned on a separate rotator and vertical translator. Figure 2 gives a schematic overview of the mechanical arrangement.

Figure 2: Mechanical Arrangement: Each multilayer is positioned on a separate translator and rotator for the first adjustment. The second multilayer is additionally mounted on a big translator that compensates the limited spatial dimension of the multilayer. All together is rotateable around the axis being tangent to the surface of the first multilayer.

Results

The experimental results with synchrotron radiation are shown in Figure 3. If the double multilayer monochromator is adjusted once, the energy tuning can be performed in a very simple way. The graphs show two different adjustments at 10 and 15 keV respectively. For higher energies a broadening of the peak of the monochromatized spectrum can be observed. To find an explanation for the high background in the spectra one has to consider some effects that
are not shown in Figure 1. When the white beam hits the first multilayer only radiation of a certain energy is reflected according to Bragg's Law. The remaining photons of the beam are partly absorbed, especially the high energetic ones are scattered and also transmitted through the multilayer. Due to the small vertical distance between the primary and the double monochromatized beam the cone of the scattered radiation and the monochromatized beam intersect at the point of detection.

Figure 3: Two different energy adjustments for monochromatization of synchrotron radiation with double multilayer monochromator. The relatively high background is caused by the primary radiation penetrating the first multilayer.

The problems caused by scattered radiation are not so critical when using a beam of an x-ray tube (W-anode) because the intensity of the high energy radiation is relatively small. As energy, the characteristic Lβ-line of the anode was selected (Figure 4). On the left graph the spectrum of the beam reflected by the first multilayer is shown. For the harmonics of higher order the peaks become broader and the intensity decreases. Of course one has to take into consideration that the primary spectrum is not white. On the right graph the spectrum of the double monochromatized beam shows really monochromatic properties. Due to inherent slight de-adjustments of the second multilayer the higher harmonics are suppressed and the energy resolution of the first harmonic is determined by the detector properties.

Figure 4: Monochromatized x-ray tube spectrum at W-Lβ: The left graph shows the spectrum of the single monochromatized beam with the harmonics up to the fourth order. The right one shows the spectrum of the double monochromatized beam. Due to inherent de-adjustment of the second multilayer the higher harmonics are suppressed.
Figure 5 shows the analogue graphs at a Bragg angle that reflects radiation of the continuous spectrum. For the selected energy of about 28 keV the spectrum of the single monochromatized beam has a quite broad peak. But with slight de-adjustments of the second multilayer the width of the peak can be reduced.

![Graphs showing the effect of de-adjustments on the width of the peak.](image)

**Figure 5**: Monochromatized x-ray tube spectrum at 28keV: The single monochromatized beam shows a quite broad peak at this energy (left graph). By de-adjusting the second multilayer the width of the peak can be reduced (right graph).

**Discussion of the results**

When using synchrotron radiation it is necessary to put a beam stopper behind the first multilayer that absorbs the transmitted and scattered primary radiation to achieve a good background. If the monochromator system is operated at higher energies it might be useful to mount a beam stopper behind the second multilayer. The beam stoppers can also be combined with an extended shielding to absorb the background radiation caused multiple scattered radiation inside the hutch.

Another problem represents the damages of the multilayers. If the surrounding gas is air the highly-energetic, intense synchrotron radiation produces radicals that interact with the multilayers. This can be avoided by filling the volume surrounding the multilayers with an inert gas or evacuation. Additionally the heat damages due to energy deposition have to be taken into account. The layers can be destructed permanently if the heat load is too high. Cooling units at the rear side take remedial action.

**References**