A multiwire proportional counter for XAS fluorescence experiments

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A multiwire proportional counter was used in fluorescence X-ray absorption measurements and a comparison to a Si(Li) and NaI(Tl) detectors was done. The main features of the mutiwire proportional counter are its high counting rate capability (10^7 counts•s^{-1}) and large active area (6 x 6 cm^2). It was shown that the MWPC is suitable for fluorescence absorption. Although the maximum capability was not reached in the present experiments, it was found that as the counting rate increase the MWPC performance became better than Si(Li) detectors and shows a similar response to the scintillator counter at medium counting rates (up to 10^5 counts•s^{-1}).

Keywords: multiwire proportional counters, x-ray detectors, diluted samples, fluorescence EXAFS

1. Introduction

The X-ray absorption spectrum from diluted samples is usually obtained by detecting the fluorescence radiation of the selected (dilute) atom. The fluorescence emission is characteristic of the atom of interest and by tuning the detection window to this characteristic energy it is possible to collect only the selected photons and to eliminate almost all background contributions coming from other scattering and undesirable emission lines.

Scintillator and solid state detectors are commonly used for fluorescence absorption experiments. Scintillator detectors have high quantum efficiency but poor energy resolution. They are usually used for intermediate-concentration samples where the background contributions are considerably smaller than the selected emission line. The maximum counting rate that can be achieved with these detectors is of the order of 10^5 counts•s^{-1}. Solid state detectors, such as Si(Li) or HPGe, are used when high energy resolution is required (170 eV @ 5.9 keV) to get rid of strong background contributions. However, they typically have small detecting area (30-50 mm^2), low counting rate capability (10^4 counts•s^{-1}) and work at liquid nitrogen temperature, which limits their applications.

A multiwire gas proportional counter (MWPC) was recently developed by collaboration between LNLS and CBPF groups (Barbosa *et al.*, 1998). Its main features are the high counting rate capability (10^7 counts•s^{-1}) and large active area (6 x 6 cm^2).

The proposal of the present work was to use the MWPC as a fluorescence detector for XAS experiments and compare its performance to Si(Li) and scintillator detectors in terms of noise to signal ratio.

2. Materials and methods

The MWPC detector is composed by 30 resistive wires mounted parallel in a sealed chamber filled with P-10 mixture gas. Each wire work in the avalanche regime with an applied voltage of 1.9 kV (Barbosa *et al.*, 1998). The whole data acquisition system for the MWPC detector includes a pre-amplifier, a discriminator and a counter for each active wire. A control unit is also provided for assembling the information of the whole detector and storing data in memory. The analog part is composed of preamplifier and discriminator channels implemented in NIM modules (30 channels per module). In the digital parts a FPGA (Field Programmable Gate Array) logic is used to allow packing up to 30 counters per NIM module. The control unit communicates with each module through a private bus and with an IBM-PC compatible microcomputer that processes and displays data.

The samples used in the characterisation of the MWPC detector were: a) A solution of CuSO$_4$ diluted in distilled water with different mass concentrations of Cu (1%, 0.5%, 0.1%, 0.05% and 0.01%). These samples were prepared at the LNLS; b) Synthetic hydroxyapatite (HAP) samples with adsorbed Zn with different mass concentrations (20ppm, 50ppm, 100ppm, 150 ppm of Zn) (Barrea *et al.*, 2000).

Several XANES spectra at the Cu K and Zn K edges were taken in fluorescence mode using the Si(Li) solid state and the MWPC detectors. A fast NaI(Tl) scintillator counter was also used in the case of Zn K edge. The measurements were performed at the XAS beam line of the LNLS light source (Tolentino *et al.*, 1998).

The noise to signal ratio (ΔN/N) of a detector with energy discrimination is simply given by the relation

$$\frac{\Delta N}{N} = \frac{\sqrt{I_S}}{I_S} \qquad (1)$$

where I_s represents the number of the selected fluorescence photons registered by the detector.

For a detector without any energy discrimination, that relation becomes

$$\frac{\Delta N}{N} = \frac{\sqrt{I_T}}{I_S} \qquad (2)$$

where I_T is the total amount of photons and I_s represents the edge jump around the selected edge energy, i.e. the fluorescence coming from the emission lines of the selected atom.

The detector performances were compared using the noise to signal ratio calculated for each detector. By defining the partial fluorescence yield (Y) as the ratio of the fluorescence coming from the element of interest (I_s) by the total count photons ($Y=I_s/I_T$), one can calculated the theoretical signal to noise ratio (ΔN/N) for each detector and compare their performance. Considering the maximum counting rate for each detector, i.e., 10^7 for the MWPC, 10^5 for the scintillator and 10^4 for the Si(Li), one obtains the behaviour

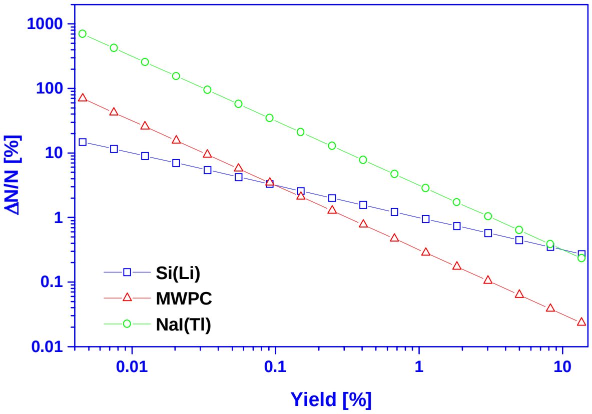

Figure 1
Theoretical noise to signal ratio as a function of the partial fluorescence yield.

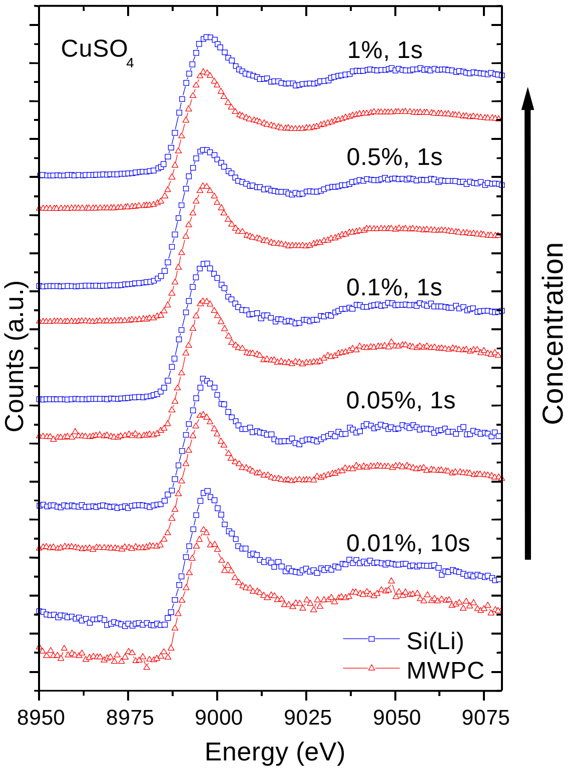

Figure 2
Normalized Cu K edge XANES spectra of $CuSO_4$ solution of different Cu concentrations. The counting time is indicated for each pair of spectra.

shown in figure 1. Both detectors without energy discrimination have essentially the same dependence on the partial yield Y, but the MWPC can stand much more photons, thence can attain a better noise to signal ratio. The solid state detector is better for smaller Y values or very diluted samples. This is because, even if it cannot stand high counting rates, energy discrimination eliminates the background contribution that is introducing more and more noise as the sample is becoming diluted.

The theoretical performance of the MWPC detector is better than the scintillator and Si(Li) detectors as soon as the partial fluorescence yield Y is greater than 0.1% and ~10^7 counts•s^{-1} can be achieved. This performance depends, of course, on the number of photons reaching the MWPC. It can be shown that, in situations when only low counting rates are achieved, the MWPC presents an equivalent behaviour to the scintillator NaI detector.

3. Experimental Results

Figure 2 shows the XANES spectra measured at the Cu K edge of $CuSO_4$ samples with the Si(Li) and MWPC detectors collecting as much photons as possible. It can be observed that the MWPC is noisier than the Si(Li) detector for the lowest Cu concentration (100 ppm). This relation is inverted for higher Cu concentrations being the Si(Li) noise to signal ratio higher than the ratio of the MWPC detector. The noise to signal ratio of these spectra, calculated in the energy region from 9035 eV to 9070 eV, defines two different domains of performance (figure 3): below 0.05% (500 ppm) the Si(Li) has a better performance and above this value the MWPC becomes better.

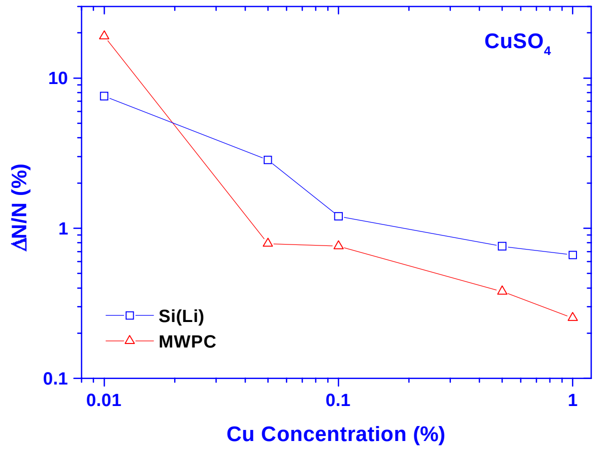

Figure 3
Noise to signal ratio of the MWPC and the Si(Li) detector as a function of Cu concentration in the $CuSO_4$ sample. The values for the lowest concentration were multiplied by $\sqrt{10}$ to account for the integration time of 10 sec.

Figure 4 shows the XANES spectra measured at the Zn K edge of HAP samples. It can be seen that the MWPC is better than the Si(Li) detector for the whole range of Zn concentrations. This happens because most background was coming from the Ca fluorescence photons, which were absorbed by the detector window and the air, putting the partial fluorescence yield at higher values than the dilution. In the most diluted case, the maximum counting rate of the MWPC was ~10^5 counts•s^{-1}, due to the small illuminated area (1x1 mm^2) of the sample. This maximum counting rate was only one order of magnitude greater than for the Si(Li) detector and essentially the same as for the scintillator detector. For all concentrations, the NaI(Tl) scintillator shows a similar performance as the MWPC. It must be pointed out that the advantage of the MWPC detector is for those situations where high counting rates are achievable, which was not the case here.

4. Conclusions

The MWPC was used in fluorescence X-ray absorption measurements and a comparison to a Si(Li) and NaI(Tl) detectors was done. It was shown that the MWPC is suitable for fluorescence absorption. The comparison of the MWPC response with Si(Li) and scintillator detectors depends on the fluorescence yield of the sample and the counting rate achieved in the experiment. Although the maximum capability was not reached in the present experiments, it was found that as the counting rate increase the MWPC performance became better than Si(Li) detectors. The comparison between the MWPC and scintillator detectors shows that the responses are similar at medium counting rates (up to 10^5 counts•s^{-1}), but as soon as the counting rate overpasses this value, the MWPC becomes better.

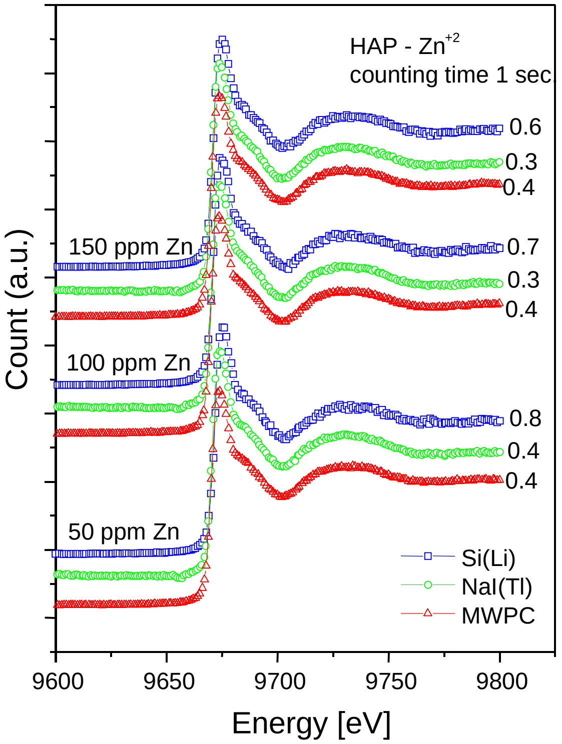

Figure 4
Normalised Zn K edge XANES spectra of HAP+ adsorbed Zn of different Zn concentrations. The counting time was 1 sec. for all the measured spectra. The noise to signal ratio is indicated for each detector type and spectrum.

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