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#### Short Communication

# Proportional counter for simultaneous conversion X-ray and transmission Mössbauer spectroscopy

I. Bibicu and M. S. Rogalski

Institute of Atomic Physics, P.O. Box MG-06 Bucharest, Romania

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**Abstract.** — The characteristics of an Ar-CH<sub>4</sub> gas flow proportional counter, which is designed to record, simultaneously, both conversion X-ray and transmitted  $\gamma$ -ray Mössbauer spectra, and operates at room temperature, are reported. The performances of the detector are demonstrated by measurements on a stainless steel sample.

#### 1. Introduction.

Mössbauer spectroscopy can be performed in the transmission mode (TMS), which records the transmitted  $\gamma$ -rays and in the backscattering mode, using either conversion electrons (CEMS), conversion X-rays (CXMS) or reemitted  $\gamma$ -rays which originate in the surface layer. Backscattering geometry is particularly useful for surface measurements of technical interest, since not only it is non-destructive but also it requires no sample preparation, as recently demonstrated by studies on laser treated [1, 2] and radiofrequency treated [3] surfaces.

The purpose of this communication is to report a new flow proportional counter for simultaneous CXMS and TMS modes, operating at room temperature. The performances of the device for an improved detection have been determined by measurements on a stainless steel sample.

#### 2. Experimental.

The cross section of the toroidal proportional detector is shown in figure 1. The body of the detector, made from aluminium, consists of only two parts, in order to lower the leak rate. We have taken into account the absorption of 6.4 keV Fe-X-rays and 14.4 keV Fe- $\gamma$ -rays by the gas mixture for the design of dimensions which minimize the 14.4 keV  $\gamma$ -rays detection in the central part of the detection area. The sample is effectively exposed to  $\gamma$ -rays.



Fig. 1. — Cross section of the toroidal detector body.

The counting wire, a golden tungsten wire, is guided by eight rods of teflon which are mounted on the back side of the detector body. The anode is shifted by about 10% of the diameter of the toroidal cross section towards the counter axis, so that the electric field gradient immediately around the anode wire is made more uniform [4].

The detector runs with a constant flow of a gas mixture which consists of 90% Ar + 10% CH<sub>4</sub>. The flow is controlled by a flowmeter at a pressure of about 0.1 MPa. The gas access was designed to avoid both the anode vibration and the appearance of a gas concentration gradient inside the detector, due to the flow rate.

The front side of the CXMS detector is closed by a thin window of aluminized mylar, which enables the 14.4 keV  $\gamma$ -rays to pass through and hit the sample. On the rear side of the detector is mounted a removable sample holder equipped with a thin aluminized mylar window. Therefore the incident 14.4 keV  $\gamma$ -rays from the Mössbauer source S (see Fig. 1) may permeate the sample A and are detected by a conventional proportional counter located behind the CXMS detector. The holder allows an easy manipulation of the sample, outside the detector and can always be repositioned in a reproducible manner with respect to the detector body.

We have used an economical shielding which consists of a combination of lead and steel disks. To destroy the characteristic radiation, the lead and steel disks are mounted in alternance. In order to absorb the unfavourable Fe KX-rays coming from the source, an aluminium filter (F in Fig. 1) was placed in front of the shielding. It is noteworthy that the side parts of the detector are completely insensitive to the  $\gamma$ -rays coming directly from the source.



Fig. 2. — Pulse height spectra of photons scattered from a sample of natural stainless steel, recorded with and without filters.



Fig. 3. — The resonance effects for various values of the filter thickness.

Fig. 4. — The resonance effects for various values of the anode voltage.

Measurements have been carried out by inserting the detector into an Elscint AME-50 driving system with a compatible Promeda 01 unit for data acquisition. The spectrometric chain has been supplemented with additional modules, which allow simultaneous recording of two spectra. We have used a 5 mCi <sup>57</sup>Co diffused in Pd Mössbauer source obtained from the Russian company "Izotop". The parameters of Mössbauer spectra have been calculated using a computer fitting program which assumes Lorentzian line shape.



Fig. 5. — Simultaneously recorded Mössbauer spectra of a natural stainless steel sample at room temperature: a) conversion X-rays Mössbauer spectrum (CXMS); b)  $\gamma$ -rays transmission spectrum (TMS).

#### 3. Results.

The Mössbauer test measurements of the detection system have been carried out at room temperature, on a 25  $\mu$ m thick natural stainless steel sample, obtained from Amersham International, Plc., Co.

The detector displays the best performance in the voltage range from 1100 V to 1300 V. A special attention was paid to the filter thickness and the voltage range applied to the anode. In figure 2 the energy spectra of backscattered photons from the sample, obtained with and without filters, at 1100 V, are plotted. The thickness of the filters was 15 and 20  $\mu$ m respectively. The amplitude spectrum obtained without filter has an energy resolution of 19% for 6.4 keV Fe KX-rays so that the 14.4 keV  $\gamma$ -rays are not practically detected. Thus, by an adequate design of its dimensions, the detector was made insensitive to the  $\gamma$ -rays coming directly from the source. As explained in previous works [5, 6], it was not necessary to use an additional polarized anode. The use of the filter reduces drastically the low energy background signals. The importance of the filter is best reflected in the dependence of the resonance effects on the

filter thickness. This dependence is reproduced in figure 3, and was obtained for an anode voltage of 1100 V. There is a significant increase in the resonance effects up to a filter thickness of 20  $\mu$ m. In an effective measurement, the thickness filter can be determined according to the semiempirical criteria for optimizing Mössbauer measurements [7]. For the aim of finding the optimum anode voltage we have measured, for the same filter, the dependence of the resonance effects on the applied voltage. This dependence is given in figure 4. The effect slightly decreases with the increase in the voltage applied to the anode. This tendency can be explained by a stronger electric field, which leads to a higher background. In the argon filled counters the main source of background is Compton electrons produced by the 122 keV photons, when scattering inside the detector volume. Background signals of about 6 keV are mainly due to the scattered  $\gamma$ -rays produced at the sample by the 122 keV  $\gamma$ -rays of <sup>57</sup>Fe.

Figure 5 shows the conversion X-rays and  $\gamma$ -rays transmission Mössbauer spectra, recorded simultaneously. The conversion X-rays spectrum is obtained by using a 15  $\mu$ m thick filter. The computer fitting gives a line width of  $0.37 \pm 0.02$  mm/s for the conversion X-ray spectrum and  $0.47 \pm 0.02$  mm/s for the corresponding transmission peak. A smaller line width is expected indeed in the backscattering geometry, due in first place to a lack of saturation broadening.

#### 4. Conclusions.

A proportional detector of toroidal shape for simultaneous CXMS and TMS is reported, with a lower efficiency for the 14.4 keV  $\gamma$ -rays. The resonance effect can be optimized by a proper choice of the anode voltage and the filter. The design is simpler then those previously reported for similar devices [5, 6]. The detection system can be successfully used for Mössbauer studies of surface and bulk phenomena.

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